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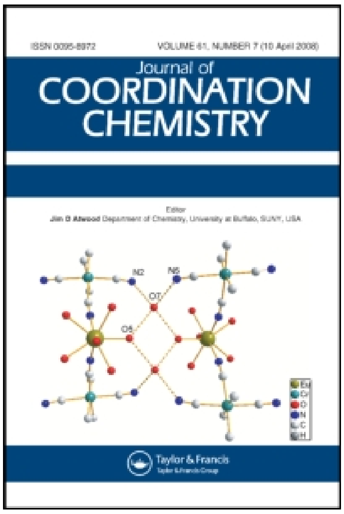

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Two manganese(II) complexes constructed from 2-(3-pyridyl)-imidazo[4,5-*f*] 1,10-phenanthroline and organic dicarboxylates: syntheses, crystal structures, and luminescence

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Two manganese(II) complexes constructed from 2-(3-pyridyl)-imidazo[4,5-*f*]1,10-phenanthroline and organic dicarboxylates: syntheses, crystal structures, and luminescence

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In this study, two new Mn(II) complexes consisting of a phenanthroline derivative and organic acid ligands, $[Mn(3\text{-}PIP)(1,3\text{-}bdc)]_n$ (**1**) and $[Mn(3\text{-}PIP)_2(1,4\text{-}bdc)]_n$ (**2**) (3-PIP = 2-(3-pyridyl)-imidazo[4,5-*f*]1,10-phenanthroline, 1,3-H_2bdc = benzene-1,3-dicarboxylic acid, 1,4-H_2bdc = benzene-1,4-dicarboxylic acid), have been synthesized *via* hydrothermal reaction and characterized by Fourier transform infrared (FT-IR) spectra, elemental analyses, and single-crystal X-ray diffraction. Complex **1** is a one-dimensional (1-D) twisted double chain bridged by 1,3-bdc. The 3-PIP ligands in a parallel fashion are alternately attached to both sides of the 1-D double chain. Complex **2** exhibits a 1-D zigzag chain, to which pairs of crossed 3-PIP ligands are alternately attached. The two complexes are further extended into three-dimensional (3-D) supramolecular structures by hydrogen-bonding and π–π stacking interactions. The N-donor ligands with an extended π-system play a crucial role in formation and stabilization of the final supramolecular frameworks. Thermal properties of **1** and **2** and fluorescence of **2** are investigated in the solid state.

Keywords: Hydrothermal synthesis; Crystal structures; Phenanthroline derivative; Manganese complexes; Organic dicarboxylate

1. Introduction

Self-assembly of metal-organic complexes has attracted interest due to their intriguing topological structures and applications as functional materials in catalysis, optics, magnetism, etc. [1–7]. The final structures of coordination polymers can be influenced by multiple factors, among which the most important are geometrical and electronic properties of metal ions and ligands [8–10]. Hydrogen-bonding, π–π stacking, and weak coordinative interactions may further link discrete subunits or low-dimensional entities into high-dimensional supramolecular networks [11–17].

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We see that 1,10-phenanthroline and its derivatives have been widely used to construct supramolecular architectures [18, 19]; derivatives, such as pyrazino[2,3-f][1,10]phenanthroline, 2-phenyl-1*H*-1,3,7,8-tetraaza-cyclopenta[*l*]-phenanthrene and dipyrido[3,2-a:2′,3′-c]phenazine, have attracted attention [20–23]. To our knowledge, 2-(3-pyridyl)imidazo[4,5*f*]1,10-phenanthroline (3-PIP), which possesses excellent coordinating ability (additional coordination sites), extended π-system, and potential hydrogen-bonding groups with more possibility to form high-dimensional supramolecular architectures, are less investigated [24, 25]. Therefore, 3-PIP has attracted our interest for construction of metal-organic supramolecular architectures. Aromatic dicarboxylates, such as benzenedicarboxylates, have been extensively employed to construct coordination polymers that exhibit a wide range of structural diversities and potential applications as functional materials [26–28].

In this article, we report the syntheses, structures, and properties of two new Mn(II) coordination polymers, $[Mn(3\text{-PIP})(1,3\text{-bdc})]_n$ (**1**) and $[Mn(3\text{-PIP})_2(1,4\text{-bdc})]_n$ (**2**). Both structures are extended into 3-D supramolecular frameworks by weak interactions.

2. Experimental

2.1. *General materials and methods*

Solvents and starting materials were commercially available and used as received. Also, 3-PIP was synthesized by the method of the literature [29] and characterized by Fourier transform infrared (FT-IR) spectra and ^{1}H-NMR. FT-IR spectra (KBr pellets) for **1** and **2** were taken on a Magna FT-IR 560 spectrometer. Thermogravimetric data were performed using a Pyris Diamond thermal analyzer. Elemental analyses (C, H, and N) were performed on a Perkin Elmer 240C analyzer. Fluorescence spectra were recorded at room temperature on a Hitachi F-4500 fluorescence/phosphorescence spectrophotometer.

2.2. *Syntheses*

2.2.1. $[Mn(3\text{-PIP})(1,3\text{-bdc})]_n$ (1). A mixture of $MnCl_2 \cdot 4H_2O$ (0.02 g, 0.1 mmol), 3-PIP (0.015 g, 0.05 mmol), 1,3-H_2bdc (0.0166 g, 0.1 mmol), NaOH (0.2 mmol), and H_2O (8 mL) was stirred for 20 min and sealed in a 25 mL Teflon-lined stainless-steel container. The container was heated to 170°C and held at this temperature for 4 days. It was then cooled to room temperature at a rate of 5°C h^{-1}. Yellow block crystals suitable for X-ray diffraction of **1** were isolated by mechanical separation from amorphous solid in 25% yield (based on Mn^{II} salt). Anal. Calcd for $C_{26}H_{15}MnN_5O_4$: C, 60.42; H, 2.90; and N, 13.56%. Found: C, 60.37; H, 2.93; and N, 13.60%. IR (KBr, cm^{-1}): 3062(w), 2856(w), 2360(m), 1568(s), 1458(s), 1386(s), 1070(m), 804(s), and 703(m).

2.2.2. $[Mn(3\text{-PIP})_2(1,4\text{-bdc})]_n$ (2). The synthetic procedure for **2** is the same as that for **1** except that 1,4-H_2bdc (0.0166 g, 0.1 mmol) was used instead of 1,3-H_2bdc. Yellow block crystals suitable for X-ray diffraction were isolated by mechanical separation from amorphous solid in 20% yield (based on Mn^{II} salt). Anal. Calcd for

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

	1	**2**
Empirical formula	$C_{26}H_{15}MnN_5O_4$	$C_{44}H_{26}MnN_{10}O_4$
Formula weight	516.37	813.69
Crystal system	Monoclinic	Monoclinic
Space group	*C*2/*c*	*C*2/*c*
Unit cell dimensions (Å, °)		
a	15.2062(9)	19.097(5)
b	15.8226(9)	9.994(5)
c	18.8195(11)	19.479(5)
α	90.000(5)	90.000(5)
β	109.9910(10)	96.3660(10)
γ	90.000(5)	90.000(5)
Volume (Å^3), *Z*	4255.2(4), 8	3654(2), 4
Calculated density (g cm^{-3})	1.612	1.479
Absorption coefficient (mm^{-1})	0.669	0.424
F(000)	2104	1668
Goodness-of-fit on F^2	1.047	1.158
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0344$, $wR_2 = 0.0888$	$R_1 = 0.0315$, $wR_2 = 0.1076$
R indices (all data)	$R_1 = 0.0454$, $wR_2 = 0.0955$	$R_1 = 0.0358$, $wR_2 = 0.1219$

[a] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; [b] $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$.

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$C_{44}H_{26}MnN_{10}O_4$: C, 64.89; H, 3.20; and N, 17.21%. Found: C, 64.82; H, 3.22; and N, 17.25%. IR (KBr, cm^{-1}): 3182(w), 3110(w), 3062 (w), 2974(w), 2366(w), 1612(s), 1539(s), 1444(s), 1380(s), 1307(w), 1188(w), 1076(m), 821(s), 715(m), and 636(w).

2.3. *X-ray crystallographic study*

A Bruker Apex CCD diffractometer (Mo-Kα radiation, graphite monochromator, $\lambda = 0.71073$ Å) was used to collect data. The structures were solved by direct methods with SHELXS-97 and Fourier techniques and refined by full-matrix least-squares on F^2 with SHELXL-97 [30, 31]. All non-hydrogen atoms were refined anisotropically, and hydrogens of the ligands were generated theoretically onto the specific atoms and refined isotropically with fixed thermal factors; hydrogens of water were located in difference Fourier synthesis maps. All the crystal data and structure refinement details for **1** and **2** are given in table 1. Relevant bond distances and angles are listed in table 2.

3. Results and discussion

3.1. *Description of crystal structures*

3.1.1. Structural analysis of [Mn(3-PIP)(1,3-bdc)]$_n$ (1). Single-crystal X-ray diffraction analysis reveals that **1** is a 3-D supramolecular network composed of a twisted double chain based on binuclear units. As illustrated in figure 1, each Mn(II) is six-coordinate in a distorted octahedral arrangement, being ligated by two nitrogens from a chelating 3-PIP with bond distances of 2.3175(15) Å [Mn1–N1] and 2.2781(15) Å [Mn1–N2], and four oxygens of three 1,3-bdc ligands (the bond lengths of Mn–O ranging from 2.1050(13) Å to 2.3362(13) Å).

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

1			
Mn(1)–O(3)	2.1050(13)	Mn(1)–O(4)	2.1217(12)
Mn(1)–O(1)	2.2443(13)	Mn(1)–N(2)	2.2781(15)
Mn(1)–N(1)	2.3175(15)	Mn(1)–O(2)	2.3362(13)
O(3)–Mn(1)–O(4)	99.34(5)	O(3)–Mn(1)–O(1)	85.22(5)
O(4)–Mn(1)–O(1)	109.79(5)	O(3)–Mn(1)–N(2)	85.57(6)
O(4)–Mn(1)–N(2)	159.97(5)	O(1)–Mn(1)–N(2)	89.89(5)
O(3)–Mn(1)–N(1)	121.84(5)	O(4)–Mn(1)–N(1)	89.75(5)
O(1)–Mn(1)–N(1)	144.30(5)	N(2)–Mn(1)–N(1)	71.40(5)
O(3)–Mn(1)–O(2)	142.07(5)	O(4)–Mn(1)–O(2)	91.73(5)
O(1)–Mn(1)–O(2)	56.96(4)	N(2)–Mn(1)–O(2)	96.23(5)
N(1)–Mn(1)–O(2)	94.16(5)		
2			
Mn(1)–O(1)	2.1273(14)	Mn(1)–O(1A)	2.1273(14)
Mn(1)–N(2A)	2.2627(18)	Mn(1)–N(2)	2.2627(18)
Mn(1)–N(1A)	2.3135(17)	Mn(1)–N(1)	2.3135(17)
O(1)–Mn(1)–O(1A)	95.01(8)	O(1)–Mn(1)–N(2A)	88.05(6)
O(1A)–Mn(1)–N(2A)	100.76(6)	O(1)–Mn(1)–N(2)	100.76(6)
O(1A)–Mn(1)–N(2)	88.05(6)	N(2A)–Mn(1)–N(2)	167.02(9)
O(1)–Mn(1)–N(1A)	87.22(6)	O(1A)–Mn(1)–N(1A)	172.63(5)
N(2A)–Mn(1)–N(1A)	72.26(6)	N(2)–Mn(1)–N(1A)	98.45(6)
O(1)–Mn(1)–N(1)	172.63(5)	O(1A)–Mn(1)–N(1)	87.22(6)
N(2A)–Mn(1)–N(1)	98.45(6)	N(2)–Mn(1)–N(1)	72.26(6)
N(1A)–Mn(1)–N(1)	91.45(8)		

Symmetry code: A: $-x$, y, $-z+1/2$.

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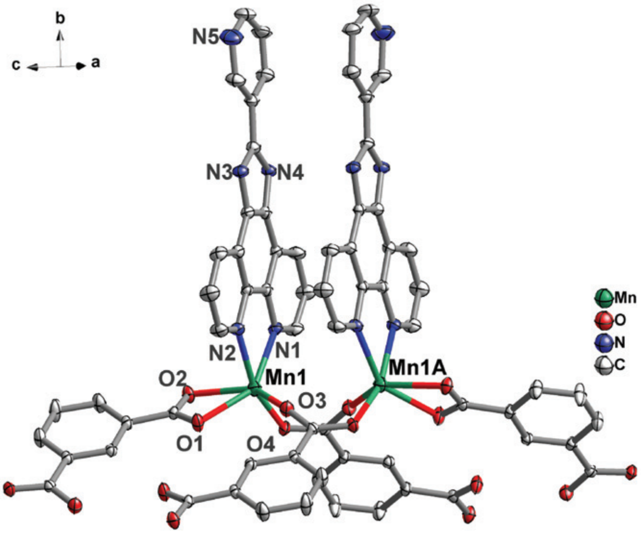

Figure 1. The coordination environment for Mn(II) in **1**.

Two carboxylates of 1,3-bdc adopt bridging bidentate and chelating bidentate coordination modes; thus the V-shaped 1,3-bdc serves as a μ_3 bridge. Mn1 and Mn1A [Mn(1)$\cdots$Mn(1A) = 4.0575(5) Å] are bridged by two bidentate carboxylates separately from two equivalent 1,3-bdc ligands to form a binuclear unit, in which 3-PIP ligands are parallel to each other. Each binuclear unit is connected to an adjacent binuclear unit

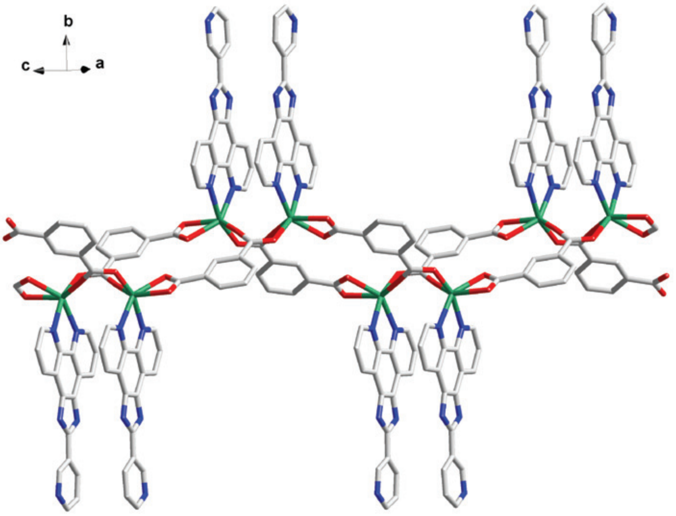

Figure 2. The 1-D twisted double chain in **1**.

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through four 1,3-bdc ligands, generating a 1-D twisted double chain, as shown in figure 2. The parallel 3-PIP ligands are alternately attached to both sides of the 1-D twisted double chain. A face-to-face distance of 3.6686(2) Å between a pair of parallel 3-PIP ligands coordinated to two Mn(II) is observed, showing significant intramolecular π–π interactions (figure 3). The twisted double chain structure may be reinforced by intramolecular π–π interactions. There are also intermolecular π–π stacking interactions [3.950(3) Å] between neighboring twisted double chains. The adjacent 1-D twisted double chains are further connected by intermolecular π–π stacking interactions and hydrogen-bonding interactions between nitrogen of imidazole and carboxylate [N(4)–H(4A)$\cdots$O(2) 2.8328(17) Å, 154(5)°] to form a 3-D supramolecular network (figure 3).

3.1.2. Structural analysis of [Mn(3-PIP)$_2$(1,4-bdc)]$_n$ (2). The structure of **2** is a 1-D zigzag chain. The asymmetric unit of **2** is composed of one Mn(II), two 3-PIP ligands, and one 1,4-bdc (figure 4). Each Mn(II) is distorted octahedral, coordinated by two oxygens from two 1,4-bdc ligands and four nitrogens from two chelating 3-PIP ligands. The Mn–O bond lengths are 2.1273(14) Å and the Mn–N bond lengths are of the range 2.2627(18)–2.3135(17) Å. Each Mn(II) is linked to adjacent Mn(II) through the bridging bis-monodentate 1,4-bdc, forming a unique 1-D zigzag chain structure (figure 5), where the Mn–Mn–Mn angle, defined by the orientation of the 1,4-bdc ligands in the chain, is 117.64°; the angle between two 3-PIP ligands coordinated to one Mn(II) is also approximately 117°. Thus, the crossed 3-PIP ligands in **2** are arranged at both sides of the chain in a parallel or linear fashion, respectively, leading to a structure suitable to form π–π stacking interactions. The 1-D zigzag chains are further extended into a 2-D supramolecular architecture by intermolecular π–π interactions (figure 5). The face-to-face distance between 3-PIP ligands is 3.904 Å. In addition, there exist hydrogen-bonding interactions between nitrogen of imidazole and carboxylate [N(3)–H(3B)$\cdots$O(2) 2.732 Å, 162°] in **2**. Thus, **2** is further assembled to a 3-D supramolecular

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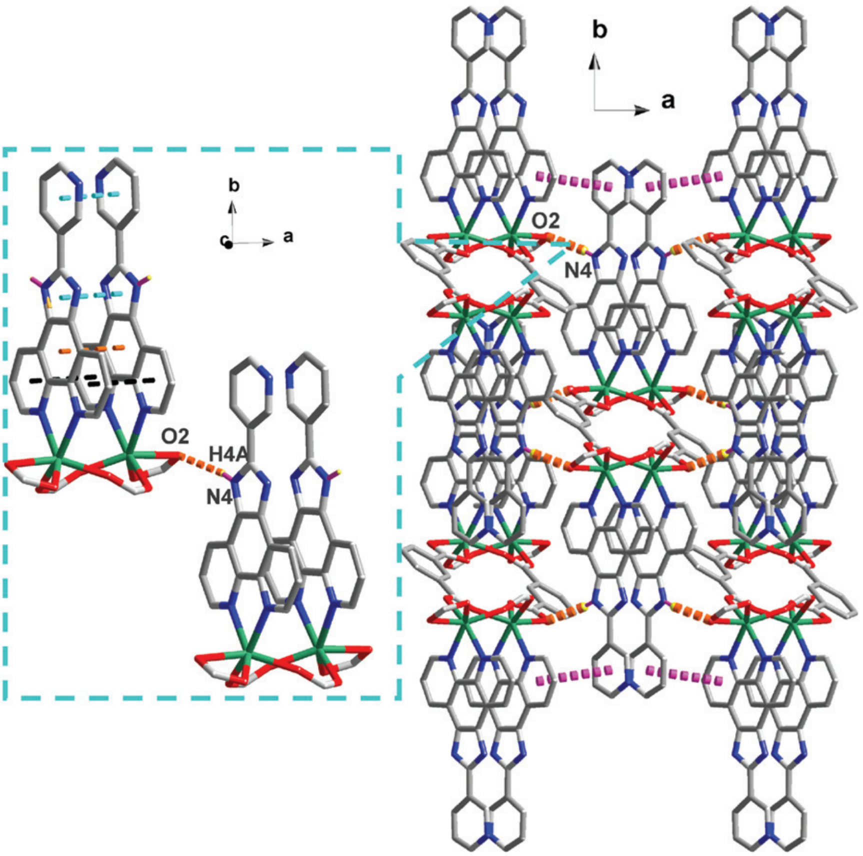

Figure 3. View of the 3-D supramolecular framework of **1** formed through weak interactions (pink broken lines represent π–π stacking interactions; orange broken lines represent H-bonding interactions).

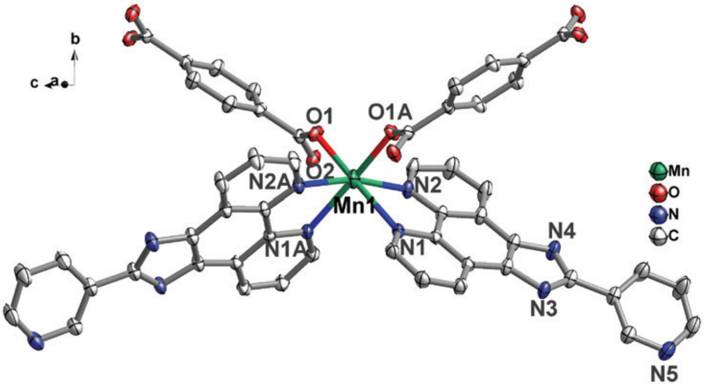

Figure 4. The coordination environment for Mn(II) in **2**.

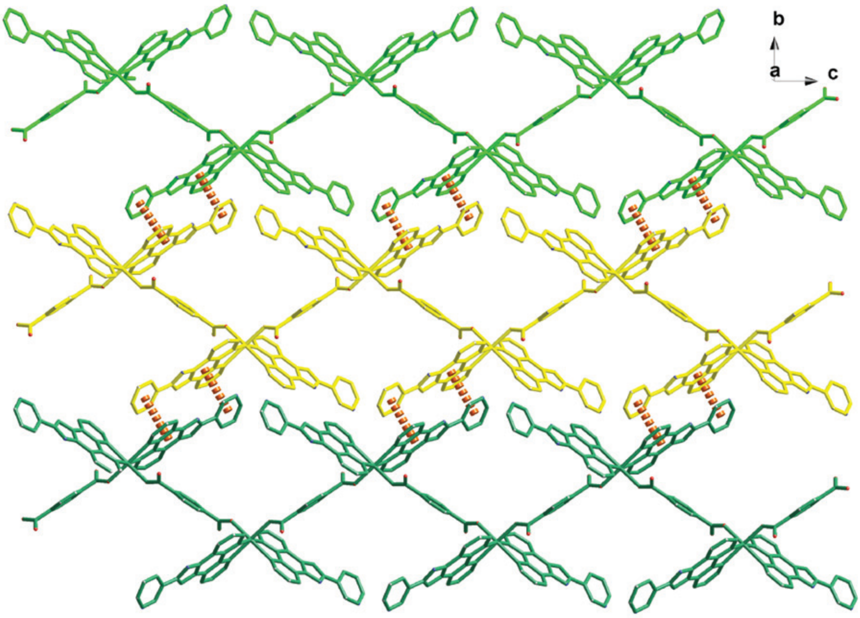

Figure 5. The 2-D supramolecular structure of **2** formed through π–π weak interactions between 1-D chains.

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framework *via* weak interactions (figure 6); the weak noncovalent interactions are important in the formation of the final supramolecular structure of **2**.

Many metal complexes based on N-donor ligands and dicarboxylates, such as 1,10-phenanthroline, 2,2′-bipyridine, isophthalate, benzene-1,4-dicarboxylate, and biphenyl-dicarboxylic acid, have been reported [32–35]. However, Mn(II) complexes constructed from 3-PIP ligand are limited. In the previously reported related complexes $[Fe(phen)(ipt)]_n$ (phen = 1,10-phenanthroline, ipt = isophthalate) and $[Mn(dpa)(2,2'\text{-bipy})]_n$ (H_2dpa = 3,4′-biphenyl-dicarboxylic acid, 2,2′-bipy = bipyridine) [32, 33], the carboxyl groups of dicarboxylates are at 1,3-position, which are convenient bridging units for linking adjacent metal centers, leading to formation of dimetal units $[Fe_2N_4O_8]$ and $[Mn_2N_4O_8]$. In this respect, **1** is similar to the reported analogous complexes [32, 33], dinuclear units $[Mn_2N_4O_8]$ have been obtained in **1**. However, **1** is ultimately extended to a 3-D supramolecular network due to 3-PIP, which is different from the above reported 2-D supramolecular networks. Further, compared with Mn-dicarboxylate complexes [34, 35], **2** is also a 3-D supramolecular framework. The results indicate that 3-PIP ligand with extended π-system and an imidazole ring as hydrogen bond donor plays an important role in the formation of 3-D supramolecular complexes.

3.2. *Effect of the organic carboxylate anions and N-donor ligand on the structures of the complexes*

The structural difference of **1** and **2** indicates that organic carboxylates play an important role in formation of such coordination architectures in the presence of 3-PIP. Contrasting **1** and **2**, the 1,3-H_2bdc and 1,4-H_2bdc possess different steric hindrance and

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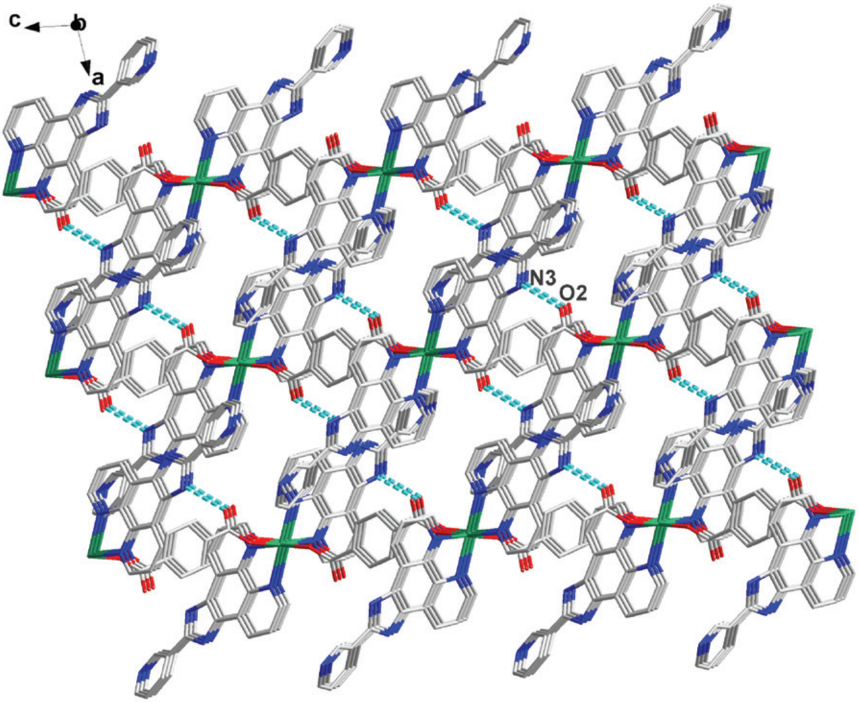

Figure 6. 3-D supramolecular framework of **2** formed through hydrogen bonding and π–π weak interactions.

the two carboxylate groups have 120° and 180° angles, respectively. The V-shaped 1,3-bdc adopts bridging bidentate and chelating bidentate coordination modes, serving as a μ_3 bridge, which results in a 1-D twisted double chain in **1**. Different from 1,3-bdc, the carboxyl groups of 1,4-bdc as a μ_2 bridge connect adjacent Mn(II) atoms to form a 1-D zigzag chain in **2**. In **1**, 3-PIP in a parallel fashion are alternately attached to both sides of the 1-D double chain. Thus, a kind of π–π stacking interaction is found in **1**. For **2**, pairs of crossed 3-PIP ligands are alternately attached to both sides of the 1-D zigzag chain. Complex **2** possesses two kinds of π–π stacking interactions. Similar hydrogen-bonding interactions involving N–H···O were found in **1** and **2**. The two complexes are further extended into 3-D supramolecular structures by hydrogen bonding and π–π stacking interactions. The N-donor ligands with extended π-system play a crucial role in the formation and stabilization of the final supramolecular frameworks.

3.3. *IR spectra of 1 and 2*

Both the complexes have similar IR spectra. The main features in the IR spectra of **1** and **2** concern the benzene dicarboxylates and 3-PIP ligands. IR spectra display the typical stretching bands of carboxylate groups between 1380 and 1606 $\mathrm{cm^{-1}}$. For **1**, the asymmetric and symmetric vibrations of carboxylate groups are at 1568, 1458, and 1386 $\mathrm{cm^{-1}}$, respectively (1612, 1539, and 1380 $\mathrm{cm^{-1}}$ for **2**). The Δ value, which

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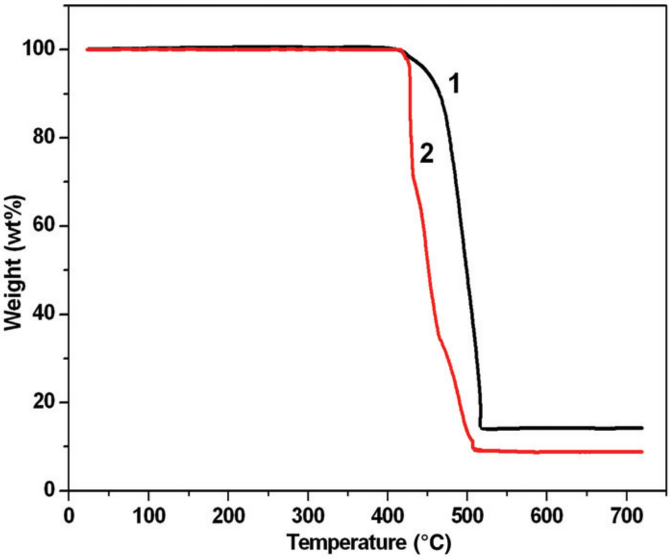

Figure 7. TG curves of **1** and **2**.

represents the separation between ν_{asym}(–COO) and ν_{sym}(–COO), is 182 and 72 cm^{-1} (232 and 159 cm^{-1} for **2**). These values are consistent with coordination modes of the title compounds. The bands at 735 cm^{-1} are attributed to $\nu_{C–N}$ of imidazole ring of 3-PIP, centered at 723 cm^{-1} for **1**, and 715 cm^{-1} for **2**. No strong absorption peaks around 1700 cm^{-1} for –COOH are observed, indicating that carboxyl groups in **1** and **2** are completely deprotonated [36].

3.4. *Thermogravimetric analysis*

Thermogravimetric analysis (TGA) gages the thermal stability of these complexes, as shown in figure 7. For **1**, there is only one-step weight loss beginning at 414°C. The weight loss of 86.0% between 414°C and 519°C corresponds to loss of coordinated 3-PIP and 1,3-bdc ligands (Calcd 86.3%). The remaining weight of 14.0% corresponds to the final product MnO (Calcd 13.7%). For **2**, there is also only one-step weight loss. The weight loss of 91.2% between 391°C and 513°C corresponds to loss of 3-PIP and 1,4-bdc (Calcd 91.3%). The remaining 8.8% corresponds to Mn and O components (Calcd 8.7%), indicating that the final product is MnO. The TGA curve of **2** indicates that the 1-D network remains stable until 391°C.

3.5. *Luminescent property of 2*

The solid-state luminescence properties of **2** and free neutral ligand 3-PIP were measured at room temperature (figure 8). Complex **2** exhibits luminescent emissions at $\lambda_{max} = 470$ nm upon excitation at $\lambda_{ex} = 330$ nm, while the free ligand 3-PIP shows luminescence at $\lambda_{max} = 424$ nm upon excitation at $\lambda_{ex} = 260$ nm in the solid state under

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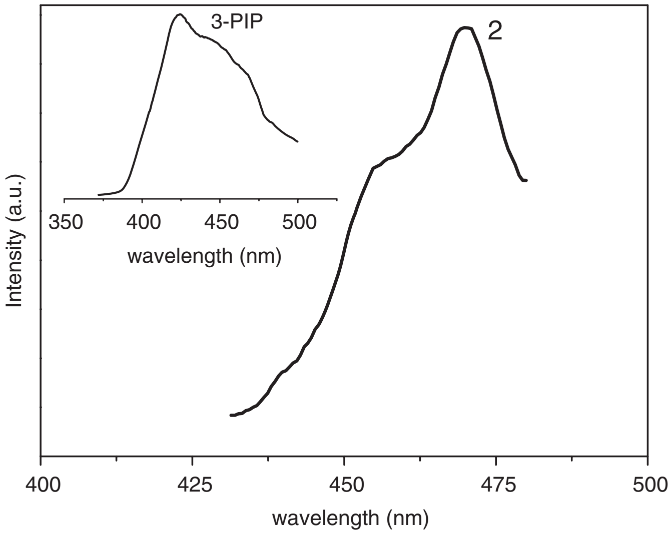

Figure 8. The emission spectra of ligand 3-PIP (inset) and **2** in the solid state at room temperature.

the same experimental conditions. Compared with free 3-PIP, the emission profile of **2** is red-shifted 46 nm, which may be due to increased conjugation upon metal coordination and ligand-to-metal charge-transfer (LMCT) [37]. Meanwhile, the emission profile of **1** is very weak compared with that of **2**, which may be attributed to the significant difference of their structures.

4. Conclusion

We synthesized two new coordination polymers, $[Mn(3\text{-PIP})(1,3\text{-bdc})]_n$ (**1**) and $[Mn(3\text{-PIP})_2(1,4\text{-bdc})]_n$ (**2**), by hydrothermal techniques. Complex **1** is a 1-D twisted double chain bridged by 1,3-bdc and **2** exhibits a 1-D zigzag chain; both are further extended into 3-D supramolecular frameworks by weak interactions. The key role of 3-PIP, an N-donor chelating ligand with extended π-systems, in the construction of metal-organic supramolecular architectures was demonstrated. More efforts will focus on the construction of coordination polymers based on phenanthroline derivatives and organic polycarboxylate ligands.

Supplementary material

Crystallographic data for the structures reported in this article have been deposited in the Cambridge Crystallographic Data Center with CCDC reference numbers 778380 and 778381 for **1** and **2**, respectively.

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