

Synthesis, crystal structures and catalytic properties of threedimensional nickel(II) and manganese(II) coordination polymers with dicarboxylate and semi-rigid bis(imidazole) ligands

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Abstract Two coordination polymers (CPs), namely $[Ni(L)(chdc)]_n$ (1) and $[Mn(L)(ndc)(H_2O)]_n$ (2) (L = 4,4'bis(imidazol-1-ylmethyl)biphenyl, H₂chdc = 1,2-cyclohexanedicarboxylic acid, H₂ndc = 2,6-naphthalenedicarboxylic acid), have been hydrothermally synthesized and characterized by physicochemical and spectroscopic methods and also by single-crystal diffraction. Both CPs feature 3D-diamond-like networks with point symbol of 6⁶; CP-1 displays a 2-fold interpenetrated net, while CP-2 presents a non-interpenetrated framework. The thermal stabilities, solid-state luminescence properties and catalytic activities of both CPs for degradation of methyl orange in a Fenton-like process were investigated.

Introduction

The current interest in coordination polymers (CPs) is stimulated not only by their intriguing topological structures, but also by their potential utilization in areas such as molecular recognition, magnetism, gas adsorption and

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catalysis [1–4]. Among the numerous strategies for construction of CPs, the self-assembly of transition metal salts with dicarboxylic acids and neutral N-donor co-ligands under hydrothermal or solvothermal conditions has become an effective approach [5–12]. 4,4'-Bis(imidazol-1-ylmethyl)biphenyl, (L) as a typical semi-rigid N-donor ligand can bend or rotate to adopt appropriate conformations when coordinated with metal ions in target CPs [13–21]. As with heterocyclic 4,4'-biphenyl-based ligands, the donor N atoms are sufficiently well separated to allow for the construction of interpenetrating motifs. However, to the best of our knowledge, 4,4'-biphenyl-based CPs rarely display interpenetrating frameworks [14]. In continuation of our ongoing research into bis(imidazole)-based CPs, we selected the semi-rigid bis(imidazole) L together with 4,4'biphenyl-based spacers as ligands. This approach afforded two CPs, $[Ni(L)(chdc)]_n$ (1) and $[Mn(L)(ndc)(H_2O)]_n$ (2) $(H_2chdc = 1, 2$ -cyclohexanedicarboxylic acid, $H_2ndc =$ 2,6-naphthalenedicarboxylic acid, Scheme 1). The crystal structures of these CPs are reported here. In addition, their thermal stabilities, luminescence properties and catalytic performances for the degradation of methyl orange in a Fenton-like process using sodium persulfate as the primary oxidant have been investigated.

Experimental

Materials and methods

The free ligand L was prepared according to the reported literature [22]. All other reagents and solvents were commercial products and used without further purification. Elemental analyses for C, H and N were obtained on a Perkin-Elmer 240C automatic analyzer. IR spectra were



Sch. 1 Structural formulas of all ligands

obtained with an Avatar 360 (Nicolet) spectrophotometer in the 4000–400 cm⁻¹ region using KBr pellets. Thermogravimetric analyses (TGA) were carried out on a NETZSCH TG 209 thermal analyzer from room temperature to 800 °C under N₂ at a heating rate of 10 °C/min. Luminescence spectra for the powdered solid samples were investigated with a Hitachi F-7000 spectrophotometer. X-ray powder diffraction (XRPD) data were recorded on a Rigaku D/Max-2500 diffractometer at 40 kV, 100 mA for a Cu-target tube and a graphite monochromator. The absorptivity value of methyl orange was measured with a Puxi 1901 UV–visible spectrophotometer.

Synthesis of CP-1

A mixture of NiSO₄·6H₂O (52.6 mg, 0.2 mmol), L (31.4 mg, 0.1 mmol), H₂chdc (34.4 mg, 0.2 mmol), NaOH (16 mg, 0.4 mmol) and H₂O (10 mL) was sealed in a 25-mL Teflon-lined stainless-steel autoclave. The mixture was heated to 140 °C for 3 days and then cooled to room temperature at 10 °C/h. Green block-shaped crystals were collected by filtration. Yield: 41.2 % (based on ligand L). Calc(%). for C₂₈H₂₈N₄NiO₄ ($M_r = 543.25$): C 61.9, H 5.2, N 10.3. Found: C 61.7, H 5.1, N 10.2. IR (KBr pellet, cm⁻¹): 3117 w, 2906 w, 1543 s, 1517 s, 1415 m, 1400 w, 1090 w, 735 w.

Synthesis of CP-2

The preparation of CP-2 was similar to that of CP-1, except H₂chdc was substituted by H₂ndc (43.2 mg, 0.2 mmol) and NiSO₄·6H₂O was replaced by MnSO₄·2H₂O (33.8 mg, 0.2 mmol). Light yellow block-shaped crystals were collected by filtration. Yield: 50.1 % (based on ligand L). Anal. Calc (%). for C₃₂H₂₆N₄MnO₅ ($M_r = 601.52$): C 63.9, H 4.4, N 9.3 %. Found: C 63.7, H 4.2, N 9.1. IR (KBr pellet, cm⁻¹): 3405 w, 3120 w, 1683 s, 1602 m, 1570 m, 1402 w, 1262 m, 1190 w, 931 w, 789 w, 659w.

Catalytic activity test

The catalytic performances of the CPs were investigated for the degradation of methyl orange (MO) in a Fenton-like process as follows: 0.01 mmol of the solid catalyst (CP-1 or CP-2) and 0.02 mmol of sodium persulfate were mixed together with 150 mL of MO solution (10 mg/L, pH = 5.0) in a 250 mL round-bottom flask. The temperature was maintained at 30 ± 1 °C by water circulation. During the given time interval, about 3.0 mL of the suspension was taken and separated by centrifugation and then analyzed with a Puxi 1901 UV–visible spectrometer at 506 nm wavelength. This procedure was repeated with equal amounts of NiSO₄·6H₂O, MnSO₄·2H₂O or without any catalyst as control experiments under the same conditions. The degradation efficiency (%D) is defined as:

$$\%D = \frac{(C_0 - C_t)}{C_0} \times 100\%$$
(1)

where C_0 (mg/L) is the initial concentration of MO, and C_t (mg/L) is its concentration at reaction time *t* (min).

In order to study the stability of each catalyst, three cycles of catalytic degradation experiments were performed under the same conditions as mentioned above. After each cycle, the catalyst was removed by centrifugation, washed several times with distilled water, and dried under vacuum for 10 h.

X-ray crystallography

Single-crystal X-ray diffraction data for both CPs were collected at 293(2) K on a Bruker Smart CCD diffractometer using graphite-monochromated Mo-K α radiation ($\lambda = 0.71073$ Å) with ω scan mode. Semiempirical absorption corrections were applied using the SADABS program [23]. The structural data were refined by full-matrix least-squares methods on F^2 using the SHELXL-97 program [24]. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms of water molecules could be located from the difference Fourier maps, while the other hydrogen atoms were included in calculated positions and refined with isotropic thermal parameters riding on the corresponding parent atoms. Crystal data and refinement parameters for both CPs are given in Table 1. Selected bond lengths and angles are listed in Table 2.

Results and discussion

Crystal structure of CP-1

Single-crystal X-ray diffraction analyses reveals that CP-1 crystallizes in the orthorhombic space group *Aba2*. The

 Table 1 Crystal and refinement

 data for two CPs

Complex	pplex CP-1	
Empirical formula	C ₂₈ H ₂₈ N ₄ NiO ₄	C32H26MnN4O5
Formula weight	543.25	601.51
Crystal system	Orthorhombic	Monoclinic
Space group	Aba2	$P2_1/n$
<i>a</i> , Å	25.1320 (2)	18.1263 (12)
b, Å	16.2343 (13)	6.0575 (4)
<i>c</i> , Å	12.4725 (13)	25.9020 (17)
α, deg	90	90
β , deg	90	93.4410 (10)
γ, deg	90	90
<i>V</i> , Å ³	5088.7 (8)	2838.9 (3)
Ζ	8	4
Calculated density, g/cm ³	1.418	1.407
Absorption coefficient/mm ⁻¹	0.805	0.514
F (000)	2272	1244
Crystal size, mm	$0.22 \times 0.19 \times 0.18$	$0.19\times0.17\times0.15$
θ range, deg	1.62–27.49	2.25-27.52
Index range h, k, l	-32/30, -21/14, -16/15	-23/23, -7/7, -33/33
Reflections collected	15,243	25,037
Independent reflections (R_{int})	5669 (0.0605)	6512 (0.0578)
Data/restraint/parameters	5669/1/334	6512/0/379
GOF on F^2	0.945	1.014
Final R_1 , wR_2 $(I > 2\sigma (I))$	0.0447, 0.0791	0.0479, 0.1204
Largest diff. peak and hole/e $Å^3$	0.281, -0.333	0.837, -0.281

asymmetric unit contains one Ni(II) atom, one chdc^{2–} ligand and two halves of L ligands. Each Ni(II) center is surrounded by two nitrogen atoms (N1, N3) from two L ligands and four carboxylate oxygen atoms (O1A, O2A, O3, O4, symmetry code: A = -x + 1/2, y, z + 1/2) from two different chdc^{2–} ligands, generating a distorted octahedral coordination geometry (Fig. 1a). The Ni1–O bond lengths are in the range of 2.052(3) Å–2.219(3) Å, and the Ni1–N bond distances are 2.040(3) Å (Ni1–N1) and 2.043(3) Å (Ni1–N3). All the bond lengths and angles around Ni(II) center are in the normal ranges [25].

Two carboxylate groups of one *e*, *a-cis*-chdc^{2–} ligand display the same chelating coordination mode. The chdc^{2–} ligands are fully deprotonated and link neighboring Ni(II) centers to form a 1D zigzag chain, in which the Ni…Ni separation is 6.403(9) Å and Ni…Ni…Ni angle is 153.80 (11)° (Fig. 1b). There are two kinds of L ligands, both of which adopt *cis*-conformation with N_{donor}…N–C_{sp3}…C_{sp3} torsion angles of 21.36(3) and 24.09(3)°. The ligands L act as μ_2 -bridging linkers to connect adjacent Ni(II) into a 1D left-handed [Ni(L)]_n helical chain with a pitch of 16.234(1) Å (Fig. 1c). The interlacing of the two distinct 1D chains gives rise to a 3D framework via shared Ni(II) atoms (Fig. 1d). In order to understand the architecture of CP-1, the topological method has been used to analyze the 3D structure by using TOPOS 4.0 software [26]. The Ni atoms are defined as 4-connected nodes, and the L and chdc²⁻ ligands are regarded as linkers. Hence, the 3D network can be classified as a classical uninodal dia net with a point symbol 6⁶. CP-1 has a 3D network with quadrilateral channels formed by connecting four linear ligands to the Ni(II) centers. The diamond cage exhibits dimensions of $6.403 \times 12.787 \times 14.892$ Å, as measured by throughspace Ni1-Ni distances. Inspection of the structure of CP-1 shows that the potential voids are filled via identical networks, resulting in a 2-fold interpenetrated network (Fig. 1e). The two-interpenetrated nets are related by a single translational vector (Class Ia) [9], with $Z_t = 2$ and $Z_n = 1$, where Z_t represents the number of interpenetrated nets related by translation and Z_n denotes the number of interpenetrated nets related by crystallographic symmetry.

Crystal structure of CP-2

The CP-2 is a 3D network with the monoclinic space group $P2_1/n$. The asymmetric unit contains one Mn(II) atom, one L ligand, two halves of ndc^{2–} ligands and one coordinated water ligand. The Mn(II) center is coordinated by three

Table 2	Selected	bond	lengths	[Å]	and	angles	[°]	for	CPs	1-	-2
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Parameter	Value	Parameter	Value	
CP-1				
Ni1–N1	2.040 (3)	Ni1–N3	2.043 (3)	
Ni1–O1A	2.052 (3)	Ni1–O3	2.123 (3)	
Ni1-O4	2.146 (3)	Ni1–O2A	2.219 (3)	
N1-Ni1-N3	92.24 (12)	N1-Ni1-O1A	95.08 (11)	
N3-Ni1-O1A	94.77 (11)	N1-Ni1-O3	164.85 (12)	
N3-Ni1-O3	96.40 (12)	O1A-Ni1-O3	96.56 (11)	
N1-Ni1-O4	104.44 (11)	N3-Ni1-O4	101.65 (11)	
O1A-Ni1-O4	153.79 (10)	O3-Ni1-O4	61.65 (10)	
N1-Ni1-O2A	91.22 (11)	N3-Ni1-O2A	156.30 (11)	
O1A-Ni1-O2A	61.56 (10)	O3-Ni1-O2A	85.85 (10)	
O4-Ni1-O2A	100.16 (10)			
CP-2				
Mn1–O4	2.106 (2)	Mn1–O1 W	2.139 (2)	
Mn1–N4A	2.215 (2)	Mn1–O2	2.2371 (18)	
Mn1-N1	2.243 (2)	Mn1–O1	2.382 (2)	
O4–Mn1–O1 W	103.15 (9)	O4–Mn1–N4A	93.18 (9)	
O1 W-Mn1-N4A	91.77 (9)	O4–Mn1–O2	171.47 (9)	
O1 W-Mn1-O2	85.32 (7)	N4A-Mn1-O2	85.41 (8)	
O4–Mn1–N1	83.86 (9)	O1 W-Mn1-N1	91.69 (9)	
N4A-Mn1-N1	175.89 (9)	O2-Mn1-N1	97.09 (8)	
O4-Mn1-O1	115.08 (9)	O1 W-Mn1-O1	141.70 (7)	
N4A-Mn1-O1	88.54 (8)	O2-Mn1-O1	56.52 (7)	
N1-Mn1-O1	90.12 (8)			

Symmetry codes: for CP-1: A: -x + 1/2, y, z + 1/2; for CP-2: A: x-1/2, -y + 1/2, z-1/2

carboxylate oxygen atoms (O1, O2 and O4) from two ndc^{2-} ligands, plus one oxygen atom (O1 W) from the water ligand and two nitrogen atoms (N1, N3A, symmetry code: A = x-1/2, -y + 1/2, z-1/2) from two L ligands, forming a distorted octahedral geometry (Fig. 2a). The bond distance of Mn1–O ranges from 2.106(2) to 2.382(2) Å, and the Mn1–N bond lengths are 2.215(2) and 2.243(2) Å, which is in accordance with the values for other reported Mn(II) complexes [27].

In CP-2, the ndc²⁻ ligands connect neighboring Mn(II) atoms into a 1D zigzag-like [Mn(ndc)]_n chains with Mn1····Mn1 distances of 13.846(1) Å and 13.139(9) Å (Fig. 2b), in which the ndc²⁻ anions show two different coordination modes ($\mu_2 - \eta^1: \eta^1$ and $\mu_2 - \eta^2: \eta^2$). Moreover, the ligand L adopts *trans*-conformation with a N_{donor}···N-C_{sp3}···C_{sp3} torsion angle of 122.67(2)°. Adjacent Mn(II) centers are connected by μ_2 -bridging L ligands to form an infinite a *meso*-helical [Mn(L)]_n motif (Fig. 2c). The Mn1···Mn1 separations across the L ligands are 16.786(9) Å. Hinging at the metal atoms, the pitch of the *meso*-helix is 30.710(2) Å. The interlaced connections from different directions of these chains further give rise to a complicated 3D framework (Fig. 2d). From the topological point of view, the Mn1 atoms are simplified to 4-connected nodes, while the ndc^{2-} and L ligands are considered as linkers. Accordingly, the 3D framework of CP-2 can be simplified to a 4-connected dia topology with the topological point symbol of 6^6 (Fig. 2e). The total void volume of the framework (after removal of all solvent molecules) reaches 35 $Å^3$, as calculated by PLATON software [26], which is approximately 1.2 % of the unit cell volume(2838.9 \AA^3). Notably, quadrilateral channels are observed in this manganese organic framework with a size of ca. $13.140 \times 13.846 \times 16.786$ Å. The crystal structure is further stabilized by hydrogen-bonding interactions between the coordinated water and ndc^{2-} ligands, which refer to O1 W-H1WA...O1C (2.780(3) Å) and O1W-H1WB...O1C (2.592(3) Å) (symmetry code: A = x-1/2, -y + 1/2, z - 1/2; B = 1-x, 1-y, 2-z; C = x, 1 + y, z).

XRPD results and IR spectra analysis

X-ray powder diffraction (XRPD) patterns of both CPs were in reasonable accordance with those simulated from their single-crystal structure data, indicating the bulk phase purity of samples of CP-1 and CP-2 (Fig. S1). The infrared spectra of both CPs exhibit no absorption peaks around 1700 cm^{-1} , demonstrating that all carboxyl groups of the dicarboxylate ligands are deprotonated [28]. For CP-2, a strong broadband at about 3400 cm^{-1} corresponds to the O-H stretching vibrations of water molecules. Characteristic peaks at 1543 and 1415 cm^{-1} for CP-1, and at 1683, 1520 and 1402 cm⁻¹ for CP-2 may be attributed to the asymmetric and symmetric vibrations of carboxylate groups. The separations $[v_{as} (COO) - v_s (COO)]$ between these bands indicate the presence of monodentate (281 cm^{-1} for CP-2), and chelating (128 cm^{-1} for CP-1, 118 cm^{-1} for CP-2) carboxylate coordination modes. Strong bands at 1517 cm^{-1} for CP-1 and 1515 cm^{-1} for CP-2 are assigned to the $v_{C=N}$ stretching of the imidazole rings of the L ligands.

Thermal and luminescence properties

Thermogravimetric analysis was carried out for both CPs in order to investigate their thermal stabilities. The TGA curves show that the decomposition of CP-1 from 300 to 630 °C, and the residual weight matches well with the formation of NiO (obsd.: 14.4 %, calcd.: 13.8 %). For CP-2, a weight loss in the range of 20–180 °C is attributed to the loss of water (obsd 2.4 %, calcd 3.0 %). Decomposition of the organic framework occurs at ca. 320 °C with the

Fig. 1 a The coordination environment of Ni(II) center in CP-1 with ellipsoids at 30 % probability, all hydrogen atoms are omitted for clarity (symmetry codes: A: -x + 1/2, y, z + 1/2; B: -x + 1, -y + 2, z). b The 1D "V"-like chain. c The 1D left-handed [Ni(L)]_n helical chain. d View of the 3D supramolecular framework. e Twofold interpenetrated 3D dia topology in CP-1



final residue corresponding to MnO (obsd 12.5 %, calcd 11.8 %) (Fig. S2).

The solid-state luminescence behaviors of both CPs and the free ligand L have been investigated at room temperature. As shown in Fig. 3, an emission for the free N-donor ligand is observed with a λ_{max} of 420 nm on excitation at 250 nm, which is probably attributable to $\pi^* \rightarrow \pi$ transitions [29]. A strong emission peak is observed at about 390 nm ($\lambda_{ex} = 250$ nm) for CP-1 and 400 nm ($\lambda_{ex} = 280$ nm) for CP-2. The emission bands for both CPs exhibit blue shifts of ca. 30 nm (for CP-1) and 20 nm (for CP-2) relative to the free L ligand, which is due to the fact that the coordinated ligands are not allowed to relax by torsional modes on photoexcitation [30]. The different shifts may result from the different crystal architectures and coordination environments of the metal centers.

Catalytic degradation behaviors

Due to their extensive industrial use, large amounts of dye are released into effluent streams [31, 32]. Given their chemical stability, removal of dyes from wastewater is an important ecological problem. Recently, advanced oxidation technology based on Fenton-like reactions has been developed for the degradation of azo dyes [33]. Fenton-like reactions involve the production of strongly oxidizing sulfate radicals (SO₄·⁻) from sodium persulfate in the presence of CPs as catalysts [4, 8, 10]. Sulfate radicals are highly active for the decomposition of organic pollutants, including dyes.

To study the catalytic properties of the present CPs, methyl orange (MO) was chosen as a model dye to assess the efficiency of its catalytic degradation. As shown in Fig. 4 and S3, little decolorization was observed in a blank experiment, such that the degradation of MO only reached 14.5 % after 100 min, indicating that MO cannot be effectively oxidized by $Na_2S_2O_8$. However, when CP-1 or CP-2 was introduced as a heterogeneous catalyst into the reaction system, after 100 min, the degradation efficiency of MO was increased considerably, to 86.9 or 92.7 % for CP-1 and CP-2, respectively. In the presence of equimolar amount of H₂chdc, H₂ndc, free ligand L, NiSO₄·6H₂O or MnSO₄·2H₂O as further control experiments, approximately 17.3, 14.9, 18.7, 48.3 and 44.3 % of MO were decomposed after 100 min, respectively. **Fig. 2** a The coordination environment of Mn(II) center in CP-2 with ellipsoids at 30 % probability, all hydrogen atoms are omitted for clarity (symmetry codes: A: x-1/2, -y + 1/2, z-1/2; B: -x + 1, -y + 1, -z + 2). b The 1D wave-like chain. c The 1D meso-helical [Mn(L)]_n motif. d View of the 3D network structure. e The 3D dia network in CP-2





Fig. 3 Emission spectra of two CPs and free ligand L in the solid state at room temperature

As shown in Table 3, after three successive cycles of degradation experiments under the same conditions, the activity of the recovered catalyst (CP-1 or CP-2) was



Fig. 4 Results of CPs, metal salts and no catalyst for the degradation of methyl orange

slightly decreased, due to leaching of metal ions. The recovered catalysts retained the same structure as the crystals used for X-ray powder diffraction (Fig. S1).

 Table 3 Repeated catalytic experiments results of methyl orange

Cycle runs	D (%)	Cycle runs	D (%)
CP-1		CP-2	
1st run	86.9	1st run	92.7
2nd run	82.5	2nd run	88.7
3rd run	75.4	3rd run	82.6

Therefore, both CPs exhibit not only good catalytic properties but also reasonable stabilities [34, 35].

Conclusion

In this work, we have synthesized and characterized Ni and Mn coordination complexes of N-donor ligands with two distinct dicarboxylate co-ligands. CPs 1 and 2 have 3D twofold interpenetrated and non-interpenetrated dia nets, respectively. CP-2 shows higher catalytic activity and stability than CP-1 in the Fenton-like decomposition of methyl orange.

Supplementary materials

X-ray crystallographic files in CIF format for CPs 1-2 have been deposited at the Cambridge Crystallographic Data Centre with CCDC No: 1472278–1472279. 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; fax: (+44) or e-mail: deposit@ccdc.cam.ac.uk.

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