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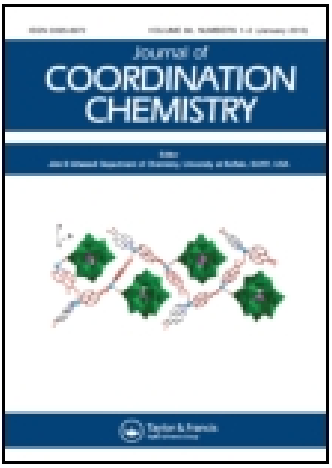

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Two new coordination polymers constructed by 1,4-bis(2-methylimidazol-3-ium-1-yl)biphenyl and dicarboxylate ligands: from a twofold interpenetrated pcu topological net to a 2-D+2-D→3-D polycatenated framework

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Two new coordination polymers constructed by 1,4-bis(2-methylimidazol-3-ium-1-yl)biphenyl and dicarboxylate ligands: from a twofold interpenetrated pcu topological net to a 2-D+2-D→3-D polycatenated framework

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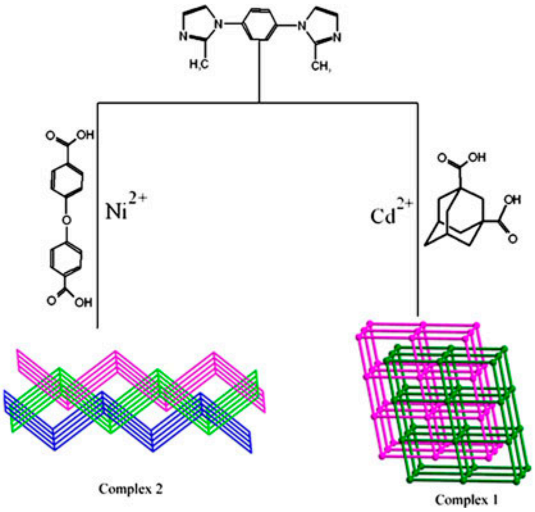

Two new coordination polymers, $[Cd(ADC)(bmib)0.5]_n$ (**1**) and $[Ni(obb)(bmib)]_n$ (**2**) [H_2ADC = 1,3-adamantanedicarboxylic acid, bmib = 1,4-bis(2-methylimidazol-3-ium-1-yl)biphenyl and H_2obb = 4,4′-oxybis(benzoic acid)], have been obtained from hydrothermal reactions. Their structures were determined by single-crystal X-ray diffraction analyses and further characterized by elemental analyses, IR spectroscopy, and powder X-ray diffraction. Complex **1** exhibits a 3-D twofold interpenetrated six-connected $(4^{12}\cdot6^3)$-pcu net based on dinuclear Cd(II) units. Complex **2** exhibits a 2-D+2-D→3-D polycatenated framework based on 2-D $(4^4\cdot6^2)$-*sql* net. Luminescence of **1** is investigated in the solid state at room temperature and the magnetic properties of **2** are investigated.

Keywords: Coordination polymers; Crystal structures; Cd(II)/Ni(II)

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1. Introduction

Synthesis of coordination polymers (CPs) has current interest in crystal engineering, for promising applications in gas storage, separations, magnetism, luminescence and catalysis, and for fascinating architectures and topologies [1–7]. One of the most frequent ligand choices is an aromatic dianionic dicarboxylate, which can bridge metal cations while providing the structural integrity and the required charge balance for formation of a stable neutral crystalline framework [8–14]. Diverse structural topologies are observed in these systems, depending on the coordination environment preferences of the metal ions and the geometric orientation of the carboxylate groups on the aromatic ring [15–17]. The two imidazole groups of 1,4-bis(2-methylimidazol-3-ium-1-yl)biphenyl would freely rotate around the central phenyl ring, giving different possible conformations. Thus, it may be possible to yield interpenetrated coordination networks. As indicated by Cambridge Structure Database survey with the help of ConQuest version 1.3, few CPs based on 1,4-bis (2-methylimidazol-3-ium-1-yl)biphenyl were reported [18–20].

We synthesized and characterized two new coordination polymers, $[\mathrm{Cd(ADC)(bmib)0.5}]_n$ (**1**) and $[\mathrm{Ni(obb)(bmib)}]_n$ (**2**), exhibiting twofold interpenetrated pcu topological net and 2-D+2-D→3-D polycatenated framework, respectively.

2. Experimental

2.1. *General materials and method*

All reagents and solvents were commercially available and used without purification. C, H, and N analyses were carried out with a Perkin-Elmer 240C elemental analyzer. FT-IR spectra were recorded with a Bruker Equinox 55 FT-IR spectrometer as dry KBr pellets from 400 to 4000 $\mathrm{cm^{-1}}$. Solid-state fluorescence spectra were recorded on a Hitachi F-4600 equipped with a xenon lamp and a quartz carrier at room temperature. X-ray powder diffraction (XRPD) measurements were performed on a Bruker D8 diffractometer operated at 40 kV and 40 mA using Cu-Kα radiation (λ = 0.15418 nm).

2.2 *Syntheses*

2.2.1. Preparation of $[\mathrm{Cd(ADC)(bmib)}_{0.5}]_n$ (1). Mixture of $\mathrm{Cd(NO_3)_2{\cdot}4H_2O}$ (0.5 mM, 0.145 g), $\mathrm{H_2ADC}$ (0.5 mM, 0.112 g), bmib (0.5 mM, 0.119 g), NaOH (1 mM, 0.04 g), and 12 mL of water were placed in a 25 mL Teflon reactor which was heated to 150 °C for 3 days and then cooled to room temperature; the pH changed from 9.5 to 6.8. Colorless crystals were obtained, selected, washed with distilled water, and dried in air (Yield: 45% based on Cd). Elem. Anal. Calcd (%) for $\mathrm{C_{19}H_{22}CdN_2O_4}$: C, 50.29; H, 4.66; N, 6.17. Found: C, 50.34; H, 4.65; N, 6.18. IR/$\mathrm{cm^{-1}}$ (KBr): 1616(s), 1513(s), 1308(m), 1245(m), 1190(m), 1031(m), 942(m), 801(m), 764(m).

2.2.2. Preparation of $[\mathrm{Ni(obb)(bmib)}]_n$ (2). Mixture of $\mathrm{Ni(NO_3)_2{\cdot}6H_2O}$ (0.5 mM, 0.145 g), $\mathrm{H_2obb}$ (0.5 mM, 0.1261 g), bimb (0.5 mM, 0.119 g), NaOH (1 mM, 0.04 g), and 12 mL of water were heated to 150 °C for 3 days, and then cooled to room temperature.

Table 1. Crystallographic data and structure refinement summary for **1** and **2**.

Empirical formula	$C_{19}H_{21}CdN_2O_4$	$C_{28}H_{22}N_4NiO_5$
Formula weight	453.78	553.21
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	$P\text{-}1$
Unit cell dimensions	a = 10.025(5) Å	a = 9.394(5) Å
	b = 13.258(5) Å	b = 11.591(5) Å
	c = 13.965(5) Å	c = 13.159(5) Å
		α = 65.394(5)°
	β = 98.513(5)°	β = 83.590(5)°
		γ = 83.983(5)°
Volume (Å^3)	1835.7(13)	1291.9(10)
Z	4	2
Calculated density (mg/m^3)	1.642	1.422
Independent reflections ($I > 2\sigma(I)$)	3951	5244
$F(0\,0\,0)$	916	572
θ range for data collection	2.345–27.496	1.71–27.54
Limiting indices	$-13 \le h \le 12$	$-12 \le h \le 11$
	$-13 \le k \le 17$	$-13 \le k \le 15$
	$-18 \le l \le 18$	$-17 \le l \le 17$
Goodness of fit on F^2	1.068	1.050
$R_1{}^a$, $wR_2{}^b$ [$I > 2\sigma(I)$]	R_1 = 0.0202, wR_2 = 0.0530	R_1 = 0.0315, wR_2 = 0.0806
$R_1{}^a$, $wR_2{}^b$ (all data)	R_1 = 0.0219, wR_2 = 0.0545	R_1 = 0.0373, wR_2 = 0.0.842
	−0.430 and 0.692	−0.202 and 0.387

${}^aR = \Sigma(||F_0| - |F_C||)/\Sigma|F_0|$. ${}^b wR = [\Sigma w(|F_0|^2 - |F_C|^2)^2/\Sigma w(F_0{}^2)]^{1/2}$.

The pH changed from 9.7 to 6.9. Green crystals were obtained in pure phase, washed with water and ethanol, and dried at room temperature (Yield: 33% based on Ni). Anal. Calcd for $C_{28}H_{22}NiN_4O_5$: C, 60.79; H, 4.01; N, 10.13. Found: C, 60.80; H, 4.00; N, 10.12. IR/cm^{-1} (KBr): 1608(s), 1533(s), 1471(s), 1305(m), 1243(m), 1108(m), 975(m), 836(m), 774(m), 693(m).

2.3. *X-ray crystallography*

Single-crystal X-ray diffraction analyses of **1** and **2** were carried out on a Bruker SMART APEX II CCD diffractometer equipped with graphite monochromated Mo-$K\alpha$ radiation (λ = 0.71073 Å) by using a ω-scan mode. Empirical absorption correction was applied using SADABS [21]. All the structures were solved by direct methods and refined by full matrix least squares on F^2 using SHEXL 97 [22]. All non-hydrogen atoms were refined anisotropically. Hydrogens were located by geometric calculations, and their positions and thermal parameters were fixed during the structure refinement. Crystallographic data and experimental details of structural analyses for coordination polymers are summarized in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. *Structure description*

3.1.1. $[Cd(ADC)(bmib)0.5]_n$ (1). Single-crystal X-ray analysis shows that **1** has the 3-D pcu topological net. In the asymmetric unit of **1**, there are one crystallographically

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

Complex **1**			
Cd(1)–O(1)	2.3498(15)	Cd(1)–O(3)	2.4463(15)
Cd(1)–N(1)	2.2287(15)	Cd(1)–O(4a)^{i}	2.3730(15)
Cd(1)–O(2)	2.2835(18)	Cd(1)–O(4)	2.2780(15)
O(4a)^{i}–Cd(1)–O(3)	128.06(4)	O(4)–Cd(1)–O(2)	96.59(6)
O(4)–Cd(1)–O(3)	54.56(5)	O(1)–Cd(1)–O(3)	110.97(6)
O(4)–Cd(1)–O(4a)^{i}	73.73(5)	O(1)–Cd(1)–O(4a)^{i}	115.89(6)
O(4)–Cd(1)–O(1)	147.72(6)	N(1)–Cd(1)–O(3)	102.90(6)
N(1)–Cd(1)–O(2)	150.12(6)	N(1)–Cd(1)–O(4a)^{i}	90.03(6)
O(2)–Cd(1)–O(3)	102.37(7)	N(1)–Cd(1)–O(4)	111.01(6)
O(2)–Cd(1)–O(4)^{i}	86.83(7)	N(1)–Cd(1)–O(1)	100.07(6)
Complex **2**			
Ni(1)–O(2)	2.1107(13)	Ni(1)–N(3)	2.0395(16)
Ni(1)–O(1)	2.1119(13)	Ni(1)–N(1)	2.0599(14)
Ni(1)–O(4)^{i}	2.2399(15)	Ni(1)–O(5)^{i}	2.0708(14)
N(3)–Ni(1)–N(1)	91.64(6)	N(3)–Ni(1)–O(1)	91.62(6)
N(3)–Ni(1)–O5^{i}	100.01(5)	N1–Ni(1)–O(1)	167.30(5)
N(1)–Ni(1)–O5^{i}	93.58(6)	O5^{i}–Ni(1)–O(1)	97.93(5)
N(3)–Ni(1)–O(2)	96.34(6)	O(2)–Ni(1)–O(1)	62.46(5)
N(1)–Ni(1)–O(2)	104.97(6)	N(3)–Ni(1)–O(4)^{i}	160.72(5)
O(5)^{i}–Ni(1)–O(2)	154.85(5)	N(1)–Ni(1)–O(4)^{i}	93.35(6)

Note: Symmetry code for **1**: (i) $-x+1, -y+2, -z+1$; for **2**: (i) $x, y+1, z-1$.

independent Cd(II), one ADC^{2-} and half of bmib. Each Cd(II) is six-coordinate by five carboxylate oxygens from three ADC^{2-} ligands [Cd–O, varying from 2.2780(15) to 2.4463 (15) Å] and one nitrogen from one bmib [Cd(1)–N(1) = 2.2287(15) Å], showing a distorted octahedral geometry (figure 1). The carboxylate ligands act as μ_3-linkers connecting cadmium(II) to a 2-D layer structure (figure 2). The carboxylate groups adopt μ_1-$\eta^1:\eta^1$ and μ_2-$\eta^1:\eta^2$ coordination modes. Carboxylate groups of μ_2-$\eta 1:\eta^2$ coordination connect two cadmium(II) ions to form a dinuclear cadmium(II) unit, with Cd···Cd distance of 3.721 Å (figure 3). The bmib ligands as pillars link the 2-D layer structure to a 3-D framework (figure 4). If the dinuclear cadmium(II) units as a node and the organic ligands are simplified as linkers, the 3-D framework can be viewed as a pcu topological net with the 11.322 × 15.940 Å rhombic window. Due to the absence of guest molecules, twofold interpenetrating pcu nets are formed (figure 5).

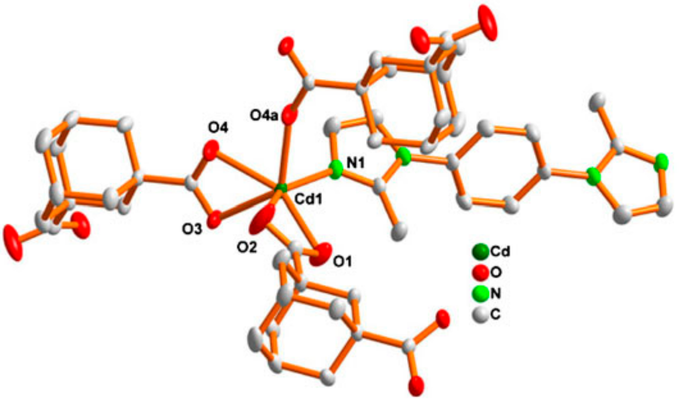

Figure 1. The coordination environment of Cd(II) in **1**. Hydrogens have been omitted for clarity.

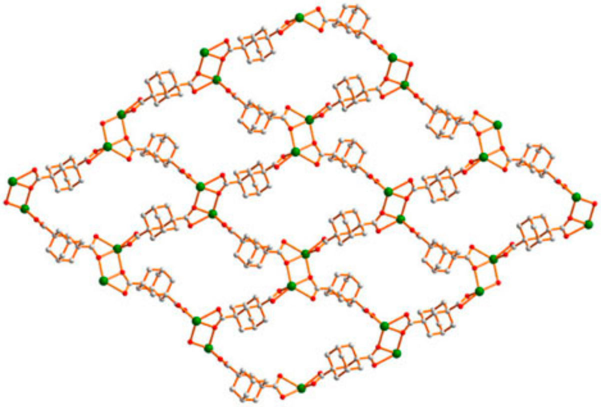

Figure 2. The 2-D structure of **1** formed by carboxylate ligands and Cd(II) ions.

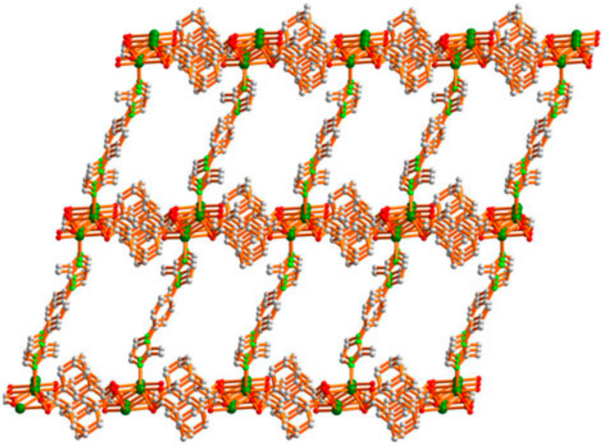

Figure 3. The 3-D structure for **1**.

3.1.2. [Ni(obb)(bmib)]$_n$ (2). X-ray single-crystal diffraction analysis reveals that **2** is a polycatenated 2-D+2-D→3-D framework based on undulated 4^4-sql single sheets. It crystallizes in the triclinic crystal system with space group of *P-1* with an asymmetric unit that contains one Ni(II), one obb^{2-}, and two halves of bmib ligands. As depicted in figure 5, Ni (II) is six-coordinate by four carboxylate oxygens [Ni–O, ranging from 2.0708(14) Å to 2.2399(15) Å] and two nitrogens [Ni(1)–N(1) = 2.0599(14) Å and Ni(1)–N(3) = 2.0395(16) Å]. Due to the flexible obb^{2-} ligands, a highly undulated sheet is formed. From a topological viewpoint, this sheet contains one type of node (Ni ion) and two types of linkers (obb^{2-} and bmib ligands). In **2**, the octahedral Ni(II) ions as four-connected nodes are bridged by two-connected bmib and obb^{2-} ligands, so the 2-D sheet can be simplified to be a 4^4-**sql** sheet, which contains a window of 13.435 × 13.829 Å and exhibits highly undulated character with a thickness of about 17.337 Å (figure 6).

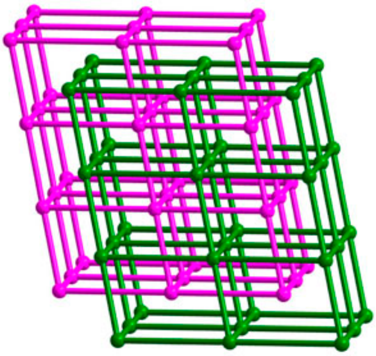

Figure 4. The twofold interpenetrated pcu topological net of **1**.

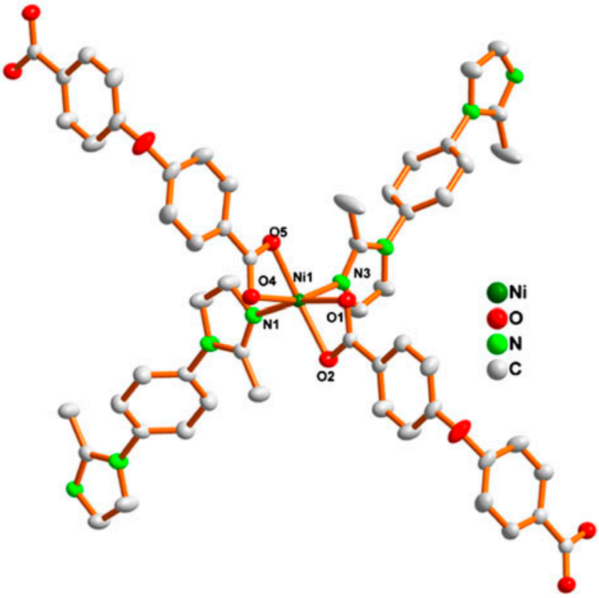

Figure 5. Crystal structure of **2**. Thermal ellipsoid (30%) plot of **2** showing the coordination environment of Ni (II).

A more intriguing structural characteristic of **2** is that each highly undulated 2-D 4^4-**sql** single sheet is simultaneously penetrated by the two nearest neighboring ones (one above and the other below), which have parallel but not coincident mean planes (The interlayer distance of 2-D sheets is 6.40 Å) (figure 7). In contrast to simple 2-D→2-D interpenetration, this resulting structure can be described by a 2-D→3-D "polycatenated network" because this network results in the overall network with a higher dimensionality than each component of the single motif with density of catenation (Doc = 2) and index of separation of (Is = 1). The high degree of entanglement is due to: (i) the large windows of the net; (ii) the highly undulated nature of the single 4^4-**sql** sheet. Among reported interpenetrated 4^4-**sql** networks, most examples show 2-D→2-D parallel interpenetration or 2-D→3-D

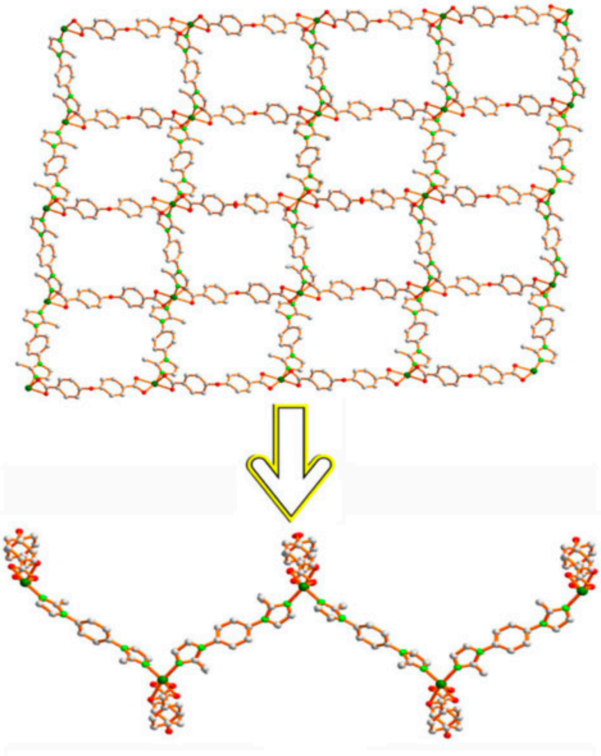

Figure 6. The view of 2-D undulated sheet along two different orientations for **2**.

inclined polycatenation and only limited 2-D→3-D polycatenated structures have been observed in a parallel mode [23–28].

3.2. *Thermogravimetric analyses and XRPD analyses*

To investigate the thermal stability of **1** and **2**, TG analyses were carried out with a NETSCHZ STA-449C thermoanalyzer with a heating rate of 10 °C/min under nitrogen. The TG curve of **1** shows that **1** is stable at 235 °C, and then a weight loss to 800 °C may to attributed to loss of organic ligands (obs. 71.53%, Calcd 71.70%) (figure S1a, see online supplemental material at http://dx.doi.org/10.1080/00958972.2014.957198). The TG curve for **2** exhibits an initial weight loss starting at 240 °C with the observed weight loss of 86.29% (Calcd 86.50%) (figure S1b). To confirm the purity of the samples, **1** and **2** are measured by XRPD. Experimental and simulated XRPD patterns for **1** and **2** are shown in figure S2. All peaks in the measured curves approximately match the simulated curves generated from single-crystal diffraction data, which clearly confirms the phase purity of the as-synthesized products.

3.3. *Luminescent properties*

The fluorescence emission spectrum of **1** was measured in the solid state at room temperature (figure 8). Intense luminescence emission band of **1** is observed at 405 nm

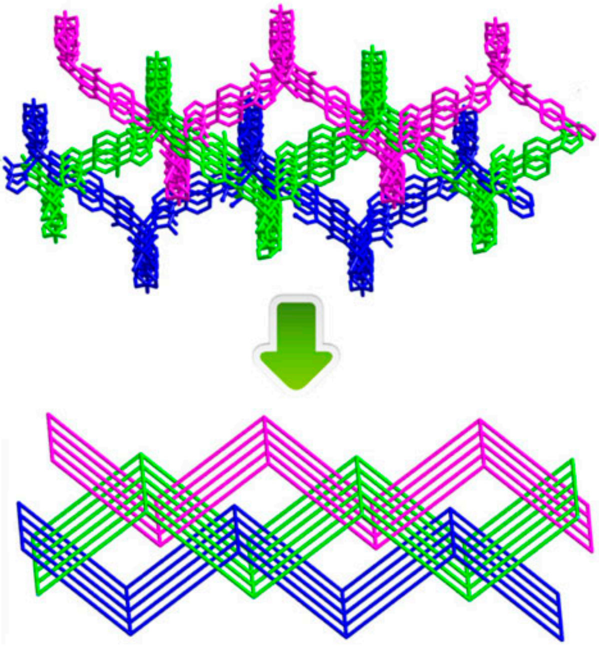

Figure 7. Schematic drawing of the 2D+2D→3D polycatenated network.

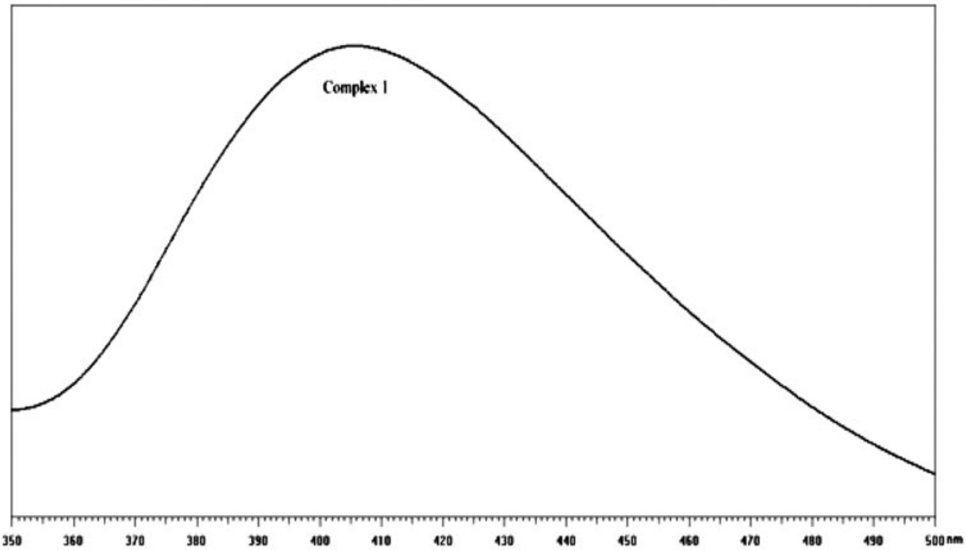

Figure 8. The luminescence for **1** in the solid state at room temperature.

(λ_{ex} = 330 nm), while the bmib ligand itself has an emission band at 408 nm (λ_{max} = 326 nm) [20]. Several factors might serve for the emissions, such as a change in the highest occupied and lowest unoccupied molecular orbital energy levels of deprotonated dicarboxylate anions and neutral ligands coordinating to metal centers, a charge-transfer transition between ligands and metal centers, and a joint contribution of the intraligand or charge-transfer transitions between the coordinated ligands and metal centers. However, because Cd^{II} ions are difficult to oxidize or reduce, these bands should be assigned to the intraligand fluorescent emissions that are tuned by the metal–ligand interactions and the deprotonation effect of the dicarboxyl ligands [29, 30].

3.4. *Magnetic properties*

Magnetic measurements were performed on polycrystalline samples of **2** using a SQUID magnetometer under an applied field of 1000 Oe from 2 to 300 K. The temperature dependence of magnetic susceptibility as $\chi_M T$ and χ_M *versus* T curves of **1** are given in figure 9. At room temperature, the value of $\chi_M T$ is 1.34 $cm^3\,Kmol^{-1}$, larger than the spin-only value of one Ni(II) center, which might be caused by the spin–orbit coupling characteristic for Ni(II) complexes with an $^3A_{2\ g}$ ground state resulting in an increasing g factor. Upon cooling to 25 K, the $\chi_M T$ values gradually decrease to 1.23 $cm^3\,Kmol^{-1}$, indicating the presence of an antiferromagnetic interaction. Then, the $\chi_M T$ value decreases rapidly to 0.571 $cm^3\,Kmol^{-1}$, which should be assigned to zero-field splitting within the ground state at low temperature, as often found in Ni(II) complexes [31, 32]. The magnetic susceptibilities of **1** can be fitted by the expression in Equation (1) deduced from the spin Hamiltonian $H = -JS_1S_2$ [33]:

$$
\begin{gathered}
\chi_M = \frac{N\beta^2 g^2}{\kappa T}\frac{(2+A\alpha+B\alpha^2)}{(3+C\alpha+D\alpha^2+E\alpha^3)} \\
A = 0.021,\ B = 0.741,\ C = 4.566, \\
D = 3.232,\ E = 5.834 \\
\alpha = \frac{|J|}{\kappa T}
\end{gathered}
\tag{1}
$$

The best fit parameters are $J = -0.93\ cm^{-1}$, $g = 2.40$, $R = 1.79 \times 10^{-4}$.

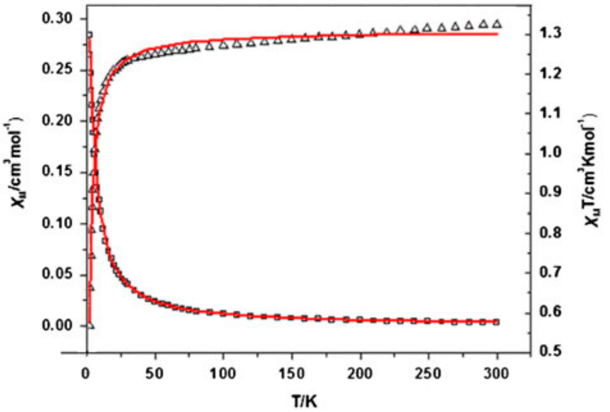

Figure 9. The plots of χ_M vs. T and $\chi_M T$ vs. T of **2** in a range from 2 to 300 K under a field of 1000 Oe. The hollow triangle and rectangle represent the experimental data and the red line represents the theoretical simulation.

4. Conclusion

A number of Cd(II) or Ni(II) coordination polymers based on carboxylate and imidazole containing different structures have been constructed [34–38]. In this paper, we constructed two new coordination polymers based on carboxylate ligands in the presence of imidazole-containing ligand. Complex **1** features a six-connected twofold interpenetrated pcu toplogy and **2** shows 2-D + 2-D→3-D polycatenated framework. Furthermore, **1** exhibits emission in the solid state at room temperature and **2** shows antiferromagetic properties.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC reference numbers: 991984 (for **1**) and 991985 (for **2**). These data can be obtained free of charge at http://www.ccdc.cam.ac.uk (or Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44 1223 336 033; E-mail: deposit@ccdc.cam.ac.uk).

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