A Perspective on Molecular Structure and Experimental-Computational Characterization of a Novel Cd(II) Pyridine-Terminal Salamo-like Coordination Polymer

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Abstract—A structurally novel Cd(II) coordination polymer (CP), $[Cd(H_2L)_2(NCS)_2]_n$, has been self-assembled from a newly designed salamo-like ligand H₂L bearing double terminal pyridine groups, Cd(II) and KSCN. The Cd(II) ion is not located in the N₂O₂ cavity of the ligand H₂L and forms a six-coordinate octahedral geometrical configuration. Using Cd(II) ion as a node, the undeprotonated salamo-like ligand H₂L units extend in space to give a Cd(II)CP with a 2D pore structure. Spectroscopic analysis of the ligand H₂L and its Cd(II)CP is performed using IR, UV-Vis and fluorescence spectroscopy. Various short-range interactions in the Cd(II)CP are investigated on the basis of Hirshfeld surfaces analysis.

Keywords: salamo-like ligand, coordination polymer, synthesis, crystal structure; Hirshfeld surface analysis

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INTRODUCTION

Coordination polymers (CPs) [1–6] have received considerable attention due to their regular pore channels and large porosity [7, 8]. In 2013, N. Ma and co-authors [9] developed a Cu-CP, the coordination polymer that enabled single-crystal to single-crystal conversion by changing humidity and could absorb CO_2 efficiently in a humid environment [9]. A.B. Stephanie and R.T. David [10] obtained seven coordination polymers and studied the effect of solvents on polymer structures.

Salen- [11–14] and salamo-like compounds [15–18] have been studied as versatile ligands in coordination and organometallic chemistry. Due to introduction of electronegative oxygen atoms in alkyl chain, salamo-like compounds demonstrate high stability. Salamo-like ligands and their transition metal complexes [19, 20] have valuable potentials in ions recognition [21–28], antibacterial activity [29–31], catalysis [32], and some more. Their detailed theoretical studies have been carried out by DFT calculations [33] and the Hirshfeld surfaces analysis [34–36].

Introduction of terminal pyridines in salamo-like ligands can provide active coordination sites because in case of addition of metal ions to the salamo-like ligands, the terminal pyridine N atoms could participate in coordination and give various novel complexes via self-assembly. Based on the studies of salamo-like ligands [19–22], a new type of such ligand (H₂L) was designed and synthesized. By self-assembly of the H₂L ligand and Cd(II) ions, a structurally novel coordination polymer was obtained. This type of coordination polymer had not been reported earlier for salamo-like metal complexes.

EXPERIMENTAL

All chemicals were analytical grade and obtained from Shanghai Meiruier Chemical Technology Co., Ltd. The solvents were of analytical grade, obtained from Tianjin Chemical Reagent Factory, and used without further purification. Melting points were measured on a microscope melting point apparatus (Beijing Taike Instrument Limited Co), and a thermometer was uncorrected. X-Ray single crystal structure analysis was carried out on a SuperNova, Dual (Cu at zero) AtlasS2 CCD diffractometer. The data for the Cd(II) CP were collected on a graphite monochromated MoK_{α} radiation $(\lambda = 0.71073 \text{ Å})$ at 297 K. In the structure refinement of the Cd(II)CP the solvent molecules could not be located because of their high thermal disorder, and the final structure model was refined without the solvent molecules by using a SQUEEZE routine of PLATON. The H atoms were included in the calculated positions and constrained

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to ride on their parent atoms. The supplementary crystallographic data is deposited (CCDC-2019252). Other instruments used in the experiments were the same as reported in our earlier publication [29].

Synthesis of 3-*tert*-butyl-5-bromosalicylaldehyde and 3-*tert*-butyl-5-(4-pyridinyl) salicylaldehyde was carried out according to the known method [37].

Synthesis of H_2L . 1,2-Bis(aminooxy)ethane was synthesized according to the reported method [30].

The mixture of anhydrous ethanol (10 mL) with 1,2-bis(aminooxy)ethane (0.21 g, 2.28 mmol) was added to 1.14 g (4.47 mmol) of 3-*tert*-butyl-5-(4-pyridyl)-salicylaldehyde in 20 mL of anhydrous ethanol. The reaction proceeded at 60°C for 12 h. Upon cooling it down to 20°C, a white solid was precipitate. Yield 57.6%, mp 129–131°C. *I*R (KBr) spectrum, v, cm⁻¹: 3450 s, 1603 s, 1260 s. ¹H NMR spectrum (500 MHz, CDCl₃), δ , ppm: 10.55 s (1H, OH), 8.62 d (2H, *J* = 6.0 Hz, ArH), 8.34 s (1H, N=CH), 7.57 s (1H, ArH), 7.43 d (2H, *J* = 6.2 Hz,

Table 1. The Cd(II)CP crystal data

Parameter	Value		
Empirical formula	C ₇₀ H ₇₆ CdN ₁₀ O ₈ S ₂		
Formula weight	1361.92		
Т, К	297		
Wavelength, Å	$MoK_{\alpha} (\lambda = 0.71073)$		
Crystal system	Triclinic		
Space group	<i>P</i> -1		
<i>a</i> , Å	10.7774(15)		
b, Å	15.853(2)		
<i>c</i> , Å	17.9611(18)		
α, deg	64.559(12)		
β, deg	82.055(10)		
γ, deg	81.733(12)		
<i>V</i> , Å ³	2732.3(7)		
Ζ	1		
$D_{\text{calc}}, \text{g/cm}^3$	0.828		
Absorption coefficient, mm ⁻¹	0.276		
F(000)	710.0		
Crystal size, mm	$0.13 \times 0.12 \times 0.11$		
2θ range, deg	4.404 to 50		
Index ranges	$-12 \le h \le 10$		
	$-18 \le k \le 18$		
	$-21 \le l \le 21$		
Reflections collected	17606		
Independent reflections	9618 [$R_{\rm int} = 0.0790$,		
	$R_{\sigma} = 0.1516$]		
Data/restraints/parameters	9618/168/430		
GOF	0.997		
Final R_1 , wR_2 indices	0.0956, 0.2281		
R_1 , wR_2 indices (all data)	0.1471, 0.2627		
I argest diff neak and hole $(\rho/Å^3)$	1 44/_0 41		



Fig. 1. (a) View of the molecular structure of the Cd(II)CP (hydrogen atoms are omitted for clarity). (b) Coordination polyhedron of Cd(II) ion.

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ArH), 7.29 s (1H, ArH), 4.56 s (2H, CH₂), 1.47 s (9H, CH₃). Found, %: C 72.34; H 6.71; N 9.82. C₄₆H₃₈N₄O₄. Calculated, %: C 72.06; H 6.76; N 9.89 (Scheme 1).

Synthesis of Cd(II)CP. The H_2L (5.75 mg, 0.01 mmol) dissolved in 2 mL of acetone was added dropwise to the mixture of Cd(OAc)₂·H₂O dissolved in 2 mL anhydrous ethanol and 1.05 mg (0.011 mmol) of KSCN dissolved in 2 mL of anhydrous methanol. The mixture immediately turned pale yellow. Stirring was continued for 25 min, then the mixture was filtered, sealed and stored for 2.5 weeks. A colorless blockshaped monocrystal suitable for X-ray diffraction was formed. Yield 46%. Although the single-crystal diffraction indicated the formula $[Cd(H_2L)_2(NCS)_2]_n$ formula of the product could be presented as $[Cd(H_2L)_2(NCS)_2]_n \cdot 2nCH_3COCH_3 \cdot n CH_3CH_2OH on$ the basis of microanalysis. Found, %: C 62.52; H 6.05; N 9.11; Cd 7.31. C₇₈H₉₄CdN₁₀O₁₁S₂. Calculated, %: C 61.47; H 6.22; N 9.19; Cd 7.38 (Scheme 2).

RESULTS AND DISCUSSION

IR spectrum of the ligand H_2L did not change significantly upon its complexation, which indicated that H_2L did not undergo deprotonation, and Cd(II) ion did not participate in coordination with the N₂O₂ cavity. The absorption bands at about 696 and 625 cm⁻¹ were attributed to the C=S bond of the coordinated ion NCS⁻, and the band at ca 451 cm⁻¹ indicated bending vibrations of the C=S bond [38]. The band at ca 422 cm⁻¹ was attributed to formation of the Cd–N bond between Cd(II) ions and N atoms of the undeprotonated ligand H₂L unit and NCS⁻ [39].

UV-Vis spectra of the free ligand H₂L demonstrated a strong band at ca 326 nm, which could be attributed to $\pi-\pi^*$ transition in the oxime groups [40]. The red-shifted



Fig. 2. Pore configuration in the Cd(II)CP.

Bond	<i>d</i> , Å	Bond	<i>d</i> , Å	Bond	d, Å
Cd ¹ –N ⁴	2.397(5)	Cd ¹ –N ^{1#1}	2.402(5)	Cd ¹ -N ^{5#3}	2.331(6)
Cd ¹ –N ⁵	2.331(6)	Cd ¹ -N ^{4#3}	2.397(5)	Cd ¹ -N ^{1#4}	2.402(5)
Bond angles	φ, deg	Bond angles	φ, deg	Bond angles	φ, deg
$N^{4}-Cd^{1}-N^{5}$	89.03(19)	N ^{1#1} -Cd ¹ -N ⁵	89.6(2)	N ^{1#4} -Cd ¹ -N ^{5#3}	90.4(2)
$N^{1#1}$ – Cd^1 – N^4	88.33(19)	N ^{4#3} Cd ¹ N ⁵	90.97(19)	N ^{1#1} -Cd ¹ -N ^{1#4}	180.00
N ⁴ -Cd ¹ -N ^{4#3}	180.00	N ⁵ -Cd ¹ -N ^{5#3}	180.00	N ^{4#3} -Cd ¹ -N ^{5#3}	89.03(19)
N ^{1#4} -Cd ¹ -N ⁴	91.67(19)	N ^{1#4} -Cd ¹ -N ⁵	90.4(2)	N ^{1#4} -Cd ¹ -N ^{4#3}	88.33(19)
N ⁴ -Cd ¹ -N ^{5#3}	90.97(19)	N ^{1#1} -Cd ¹ -N ^{4#3}	91.67(19)	N ^{1#4} Cd ¹ N ^{5#3}	89.6(2)

Table 2. Selected bond lengths and angles in the Cd(II)CP^a

^a (#1) -1+x, 1+y, -1+z; (#3) 1-x, 2-y, -z; (#4) 2-x, 1-y, 1-z.

Table 3. Geometrical parameters of hydrogen bonds and C–H $\cdots \pi$ interactions in the Cd(II)CP

	-			-	
D–H…A	<i>d</i> (D–H)	d(H–A)	d(D-A)	D–X–A angle	
O^1 – H^1 ··· N^2	0.82	1.85	2.584(8)	149	
O^4 – H^4 ··· N^3	0.82	1.84	2.567(13)	147	
C^{14} - H^{14C} O^{1}	0.96	2.28	2.920(10)	123	
$C^{15}\!\!-\!\!H^{15A}\!\cdots\!O^{1}$	0.96	2.47	3.058(11)	119	
$C^{18}\!\!-\!\!H^{18B}\!\cdots\!N^2$	0.97	2.32	2.752(15)	106	
C^{31} – H^{31A} ···O ⁴	0.96	2.30	2.919(14)	121	
C^{33} - H^{33C} O^4	0.96	2.40	3.026(17)	123	
$C-H\cdots\pi$	H–Cg	X–Cg	C–H–Cg angle		
C^{17} – H^{17A} ··· Cg^1	2.85	3.821(10)	178		

band (329 nm) of low intensity could be assigned to the ligand to metal charge transfer [41]. Such data were consistent with the earlier report and similar to the ligand to metal charge transition in salen- and salamo-like metal complexes [32].

According to the titration curves of the ligand H_2L by Cd^{2+} followed by addition of the NCS-ion indicated the optimal ratio of Cd^{2+} : H_2L : NCS⁻ as 2 : 1 : 2, which was consistent with the crystal structure of the complex determined by X-ray diffraction analysis.

The Cd(II)CP crystal data are listed in Tables 1 and 2.

The Cd(II) ion had a six-coordinate octahedral stereometrical configuration (Fig. 1). Unlike the previously reported transition metal complexes [32], in this case Cd(II) ion was surrounded by 6 N atoms (N¹, N⁴, N⁵, N^{1#}, N^{4#}, and N^{5#}). Among those the atoms N¹, N⁴, N^{1#} and N^{4#} were originated from the terminal pyridine N atoms of the four undeprotonated ligand H₂L units and characterized as coplanar. The other atoms (N⁵ and N^{5#}) were provided by two NCS⁻ anions and coordinated to the axial positions of the Cd(II) ion. The Cd(II) coordination polymer had a large cavity (Fig. 2), in which the ligand H₂L had a V-shape stereo structure (angle 104°) with C¹⁷ atom as the node.

There were identified 7 pairs of intramolecular hydrogen bonds ($O^1-H^1\cdots N^2$, $O^4-H^4\cdots N^3$, $C^{14}-H^{14C}\cdots O^1$,



Fig. 3. Graphical presentation of intramolecular hydrogen bonds in the Cd(II)CP.



Fig. 4. The C–H··· π interactions in the Cd(II)CP.

C¹⁵–H^{15A}····O¹, C¹⁸–H^{18B}····N², C³¹–H^{31A}····O⁴ and C³³–H^{33C}····O⁴) in Cd(II) CP (Table 3, Fig. 3) and one kind of C–H··· π interaction (C¹⁷–H¹⁷···Cg¹) (Fig. 4). Overall the Cd(II)CP formed a 2-D supramolecular structure via C–H··· π stacking interactions (Fig. 5).

The Hirshfeld surface analysis [42] was carried out by selecting the smallest symmetrical unit of the Cd(II) CP. In Fig. 6 the Hirshfeld surfaces of the Cd(II)CP are mapped to dnorm, shape index and curvature. In the map of dnorm, the red region represents the O···H/H···O interactions in the Cd(II)CP, the darker the color, the stronger the interaction; the other regions represent different short-range interactions. In the two-dimensional fingerprint, gray represents the whole fingerprint, while the blue area represents the proportion of each shortrange effect. Taking the O···H/H···O interactions as an example, the O···H interaction is mainly concentrated in the peak position at the bottom right of the fingerprint, while the H…O interaction is mainly concentrated in the peak position at the top left of the fingerprint. The proportion of O···H/H···O interactions is 5.3 %. The proportions of C···H/H···C, N···H/H···N, S···H/H···S and H···H interactions in the Cd(II)CP are 8.9, 3.4, 10.6,



Fig. 5. The 2-D supramolecular structure of the Cd(II)CP.

and 58.3%, respectively. Therefore, $H \cdots H$ interactions are the main interactions in the Cd(II)CP.

In fluorescence spectra of H₂L (5×10^{-5} M) and the Cd(II)CP (5×10^{-5} M) the ligand demonstrated an emission peak at ca 461 nm, which could be due to π - π * transition [43]. The emission peak of Cd(II)CP at 466 nm did not change significantly, only a small increase in fluorescence intensity was observed, and there was a small blue-shifts to ca 456 nm which was attributed to the ligand metal charge transition (LMCT) [44].

CONCLUSIONS

A novel salamo-like ligand H_2L containing double terminal pyridine groups has been synthesized and characterized. The structurally novel Cd(II)CP, [Cd(H₂L)₂(NCS)₂]_n, has been obtained. The salamo-like coordination polymer (CP) is self-assembled from Cd(II) ions, the undeprotonated salamo-like ligand H₂L units and NCS⁻ anions. The N₂O₂ cavity is not involved in the coordination. UV-Vis titration results are consistent with the obtained single crystal structure. Fluorescence properties of H₂L and its derivative Cd(II)CP have been studied. Theoretical approach to various interactions in



Fig. 6. The Hirshfeld surface analysis mapping and various 2D fingerprints of the Cd(II)CP.

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the Cd(II)CP has been based on the Hirshfeld surface analysis.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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