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Synthesis of coordination polymers with d^{10} metal ions and a new linear ligand: X-ray structural and luminescence studies

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Dedicated to Alfred Werner on the 100th anniversary of his Nobel prize in chemistry in 1913.

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1. Introduction

Recent years have witnessed an explosive growth in the construction of coordination polymers (CPs) for their potential applications in gas adsorption [1–5], molecular storage [6,7], heterogeneous catalysis [8-11], proton conduction [12-15], magnetism [16-20], luminescence [21,22] and so on. The design and characteristic features of ligands such as rigidity and flexibility, bulkiness, chirality and so on play important roles in dictating overall topology of the CPs. Additionally, the tendency of metal ions to adopt certain geometries and the external experimental conditions imposed also influences the overall structure of the frameworks. Most researchers have focused on the synthesis and utilization of symmetric, bridging ligands [23] for the construction of CPs. However, unsymmetrical ligands incorporating multiple binding sites with different donors, can assembled metal centers around to afford a variety of CPs with novel topological features [24,25]. Usually ligands having both nitrogen [26] and carboxylate [27-30] donors have been used extensively for this purpose. Motivated by this, we have designed a semi-flexible linear ligand flanked on either side by benzimidazole and carboxylate units (Scheme 1). This design provides more conformational freedom for the coordination environment around transition metal ions that exhibit strong coordination preferences. Our attempted synthesis

ABSTRACT

Four new coordination polymers (CPs), $\{[Zn_2(L)_4]\cdot 7H_2O\}_n$ (1), $\{[Zn_2(L)_4]\cdot 4H_2O\}_n$ (2), $\{[Zn(L)_2]\cdot 3/2H_2O\}_n$ (3) and $\{[Cd(L)_2]\cdot 3H_2O\}_n$ (4) (HL = 4-benzoimidazol-1-yl-methyl benzoic acid) have been synthesized under hydrothermal conditions. All the complexes have been characterized by single crystal X-ray crystallography (XRD), IR spectroscopy, elemental analysis and thermogravimetry (TGA). Complexes 1–4 show similar 2D, non-interpenetrated, uninodal 4-connected layers that are further connected by H-bonding interactions to 3D supramolecular structures. For the complexes 1–3, the overall architecture is controlled by the template used. All complexes exhibit high thermal stability losing coordinated water molecules at ~110 °C. Upon excitation at 343 nm, compounds 1–3 exhibit solid-state luminescence at room temperature. The intensity of luminescence gradually decreases with an increase in the number of lattice water molecules per Zn(II) center due to vibrational quenching by water molecules.

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of mixed ligand CPs with different N-donor co-ligands did not lead to results. However, nature of the co-ligands are found to influence the overall architecture. In this paper, we report the synthesis and crystal structural description of four new coordination polymers of Zn(II) and Cd(II) ions having the formula $\{[Zn_2(L)_4] \cdot 7H_2O\}_n$ (1), $\{[Zn_2(L)_4] \cdot 4H_2O\}_n$ (2), $\{[Zn(L)_2] \cdot 3/2H_2O\}_n$ (3) and $\{[Cd(L)_2] \cdot 3H_2O\}_n$ (4).

2. Experimental

2.1. General methods and materials

Benzimidazole (98%), methyl 4-(bromomethyl)benzoate (98%), dabco, 1,2-di(4-pyridyl)ethylene (dpe), potassium iodide, Zn(NO₃)₂·6H₂O (98%) and Cd(NO₃)₂·4H₂O (98%) were purchased from Aldrich, USA and used as received. All solvents and anhydrous K₂CO₃ were procured from S.D. Fine Chemicals, India. All solvents were purified prior to use following standard methods.

Infrared spectra were recorded on a Perkin-Elmer Model 1320 spectrometer (KBr disk, 400–4000 cm⁻¹). ¹H NMR spectra were recorded on a JEOL-ECX 500 FT (500 MHz) instrument in DMSO- d_6 using Me₄Si as the internal standard. ESI-mass spectra were collected on a WATERS Q-TOF Premier mass spectrometer. Thermogravimetric analyses (TGA) were recorded using a Mettler Toledo Star System (heating rate of 5 °C/min). Microanalyses for the compounds were obtained using a CE-440 elemental analyzer (Exeter Analytical Inc.). Solid-state photoexcitation and emission





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Scheme 1. Schematic representation of ligand HL.

spectra were recorded using double UV–Vis-NIR spectrophotometer (Varian Model Cary 5000) and Jobin Yvon Horiba Fluorolog-3 spectrofluorimeter at room temperature. Powder X-ray diffraction (Cu K α radiation, scan rate 3 °/min, 293 K) was performed on a Bruker D8 Advance Series 2 powder X-ray diffractometer.

2.2. Synthesis

The ligand **HL** was synthesized following an earlier reported [31] procedure with slight modifications, as given in supporting information.

2.2.1. Synthesis of $\{[Zn_2(L)_4], 7H_2O\}_n$ (1)

A mixture containing **HL** (20 mg, 0.08 mmol), $Zn(NO_3)_2 \cdot 6H_2O$ (60 mg, 0.20 mmol) in 3 mL water and 0.1 mL of aq. ammonia solution were sealed in a Teflon-lined autoclave and heated under autogenous pressure to 120 °C for three days and then allowed to cool to room temperature at the rate of 1 °C per minute. Block-shaped pale-yellow crystals of **1** were collected in ~40% yield. The crystals were repeatedly washed with water followed by acetone and air-dried. *Anal.* Calc. for $C_{60}H_{58}N_8O_{15}Zn_2$: C, 57.11; H, 4.63; N, 8.88%. Found: C, 57.21; H, 4.72; N, 8.75%. IR (KBr, cm⁻¹): 3450(m), 3090(m), 1611(m), 1590(s), 1538(s), 1509(w), 1463(m), 1397(s), 1294(m), 1264(m), 1202(w), 1113(w), 1018(s), 977(w), 953(w), 912(w), 859(m), 799(m), 777(s), 751(s).

2.2.2. Synthesis of $\{[Zn_2(L)_4] \cdot 4H_2O\}_n$ (2)

A mixture of Zn(NO₃)₂·6H₂O (50 mg, 0.17 mmol), **HL** (30 mg, 0.12 mmol) and dpe (10 mg, 0.06 mmol) in 3 mL H₂O and 0.1 mL aqueous Et₄NOH solution were placed in a Teflon-lined stainless steel autoclave and heated to 120 °C for three days under autogenous pressure, and then allowed to cool to room temperature at the rate of 1 °C per minute. Block-shaped pale-yellow crystals of **2** were collected in ~43% yield. The crystals were repeatedly washed with water followed by acetone and air-dried. *Anal.* Calc. for C₆₀H₅₂N₈O₁₂Zn₂: C, 59.66; H, 4.34; N, 9.28%. Found: C, 59.24; H, 4.39; N, 9.56%. IR (KBr, cm⁻¹): 3476(m), 3090(m), 1591(s), 1540(s), 1463(s), 1398(s), 1294(s), 1264(s), 1203(m), 1018(s), 911(s), 859(s), 799(s), 777(s), 751(s).

2.2.3. Synthesis of $\{[Zn(L)_2] \cdot 3/2H_2O\}_n$ (3)

A mixture containing **HL** (20 mg, 0.08 mmol), $Zn(NO_3)_2 \cdot 6H_2O$ (60 mg, 0.20 mmol) and dabco (10 mg, 0.08 mmol) in 3 mL water and 0.1 mL of NaOH solution were sealed in a Teflon-lined autoclave and heated under autogenous pressure to 120 °C for three days and then allowed to cool to room temperature at the rate of 1 °C per minute. Block-shaped yellow crystals of **3** were collected

in ~51% yield. The crystals were repeatedly washed with water followed by acetone and air-dried. *Anal.* Calc. for $C_{60}H_{50}N_8O_{11}Zn_2$: C, 60.56; H, 4.24; N, 9.42%. Found: C, 60.69; H, 4.35; N, 9.12%. IR (KBr, cm⁻¹): 3414(m), 3089(m), 1605(s), 1561(s), 1513(s), 1485(w), 1381(s), 1296(m), 1265(w), 1206(w), 1179(m), 1134(m), 1114(w), 1018(m), 921(m), 799(s), 753(s), 627(m).

2.2.4. Synthesis of ${[Cd(L)_2] \cdot 3H_2O}_n$ (4)

A mixture containing **HL** (30 mg, 0.12 mmol), $Cd(NO_3)_2\cdot 4H_2O$ (90 mg, 0.29 mmol) in 3 mL water and 0.1 mL of aq. ammonia solution were sealed in a Teflon-lined autoclave and heated under autogenous pressure to 120 °C for three days and then allowed to cool to room temperature at the rate of 1 °C per minute. Block-shaped light-yellow crystals of **4** were collected in ~50% yield. The crystals were repeatedly washed with water followed by acetone and air-dried. *Anal.* Calc. for $C_{30}H_{28}N_4O_7Cd$: C, 53.66; H, 4.20; N, 8.34%. Found: C, 53.13; H, 4.28; N, 8.21%. IR (KBr, cm⁻¹): 3424(m), 3097(m), 1611(s), 1560(s), 1512(s), 1382(s), 1297(m), 1183(m), 1017(m), 923(s), 798(s), 750(s),693(w), 663(w).

2.3. X-ray structural studies

Single crystal X-ray data on complexes 1-4 were collected at 100 K on a Bruker SMART APEX CCD diffractometer using graphite monochromated Mo K α radiation (λ = 0.71073 Å). The linear absorption coefficients, scattering factors for the atoms, and the anomalous dispersion corrections were taken from the International Tables for X-ray Crystallography [32]. The data integration, reduction, empirical absorption correction and structure solution were carried out as described earlier [33–36]. The H atoms were refined as follows: the H atoms attached to C atoms were positioned geometrically and treated as riding atoms using SHELXL default parameters [37]. The hydrogen atoms of lattice water molecules were located in difference Fourier maps and refined freely keeping the O–H bond distances constrained to \sim 0.85 Å with the DFIX command. The crystal and refinement data are collected in Table 1 while selective bond distances and angles are given in Table S1 (supporting information).

3. Results and discussion

The ligand is designed such that there is a possibility of rotational degree of freedom about C–N bond to adopt different conformations to generate different coordination polymeric structures with d^{10} metal ions. The IR spectra of **1–4** show strong absorption bands between 1414 and 1611 cm⁻¹ attributable to [38,39] coordinated carboxylate groups (see Supporting information). The broad peak in the region, 3414–3476 cm⁻¹ indicates [40] the presence of lattice water molecules.

3.1. Crystal structure of $\{[Zn_2(L)_4], 7H_2O\}_n$ (1)

Compound **1** crystallizes in the monoclinic space group C2/*c*. Single-crystal X-ray analysis reveals that the asymmetric unit of **1** contains one Zn(II), two L^{-1} and three and a half lattice water molecules. The coordination mode of L^{-1} is given in Fig. 1. Here, the metal ion shows distorted tetrahedral ZnN₂O₂ coordination from two monodentate carboxylate O (Zn-O = 1.960(2)–1.998(2) Å) and two benzimidazole N (Zn-N = 2.024(2)–2.042(3) Å) from four different L^{-1} ligand units.

A 2D non-interpenetrated rectangular-grid is generated by combination of four ligands and four metal centers. The dimension of the rectangular-grid is approximately 6.3×8.1 Å. These 2D layers stack in $\dots ABAB \dots$ fashion through $\pi \dots \pi$ interactions (3.714(1) Å) between aromatic rings of two parallel layers that is further

Table 1			
Crystal and str	ucture refinem	nent data fo	r 1–4.

Complex	1	2	3	4
Empirical formula	$C_{60}H_{58}N_8O_{15}Zn_2$	$C_{60}H_{52}N_8O_{12}Zn_2$	$C_{60}H_{50}N_8O_{11}Zn_2$	C30H28N4O7Cd
Formula weight	1261.88	1207.83	1189.81	671.38
Crystal system	monoclinic	triclinic	monoclinic	monoclinic
Space group	C2/c	ΡĪ	C2/c	C2/c
a (Å)	25.349(6)	11.779(5)	25.161(4)	24.987(5)
b (Å)	11.761(4)	11.811(6)	11.539(2)	11.986(6)
<i>c</i> (Å)	21.405(5)	20.942(3)	21.399(5)	22.025(4)
α (°)	90.000	75.872(4)	90.000	90.000
β(°)	121.855(5)	81.890(5)	123.182(5)	124.209(5)
γ (°)	90.000	77.105(7)	90.000	90.000
U (Å ³)	5420(3)	2742.5(18)	5199.7(17)	5455(3)
Ζ	4	2	4	8
$ ho_{calc}$ (g/cm ³)	1.544	1.460	1.517	1.624
μ (mm ⁻¹)	0.966	0.947	0.996	0.857
F(000)	2608	1244	2448	2704
Reflections collected	13466	13899	13138	13819
Independent reflections	3860	7166	3362	3931
Goodness-of-fit (GOF)	1.062	1.073	1.027	1.073
Final R indices $[I > 2\sigma(I)]$	$R_1 = 0.0463$	$R_1 = 0.0793$	$R_1 = 0.0558$	$R_1 = 0.0455$
	$wR_2 = 0.1202$	$wR_2 = 0.1967$	$wR_2 = 0.1387$	$wR_2 = 0.1172$
R indices (all data)	$R_1 = 0.0576$	$R_1 = 0.0978$	$R_1 = 0.0796$	$R_1 = 0.0562$
	$wR_2 = 0.1288$	$wR_2 = 0.2142$	$wR_2 = 0.1539$	$wR_2 = 0.1254$
CCDC	881367	786124	881368	881369



Fig. 1. Coordination modes of ligand \mathbf{L}^{-1} in **1**.



Fig. 2. Diagram showing $\pi \cdots \pi$ interactions between aromatic rings of two different layers in **1**.



Fig. 3. 3D view along *a*-axis in 1 showing lattice water molecules in space-fill model.

reinforced by strong non-covalent H-bonding interactions involving solvent molecules to form an overall 3D structure (Figs. 2 and 3).

Solvent accessible volume for **1**, calculated with the PLATON [41] program, is 12.0% of the total unit cell volume. Topological analysis using the Topos software [42] shows that the structure can be described as a uninodal 4-connected *sql* net with Schläfli symbol as $\{4^4.6^2\}$. (Fig. 4).

3.2. Crystal structure of $\{[Zn_2(L)_4] \cdot 4H_2O\}_n$ (2)

In this case, use of **dpe** as a co-ligand and the pH shifted to more basic condition, led to the formation of a 2-fold interpenetrated 2D coordination polymer. Interestingly, the co-ligand itself does not bind to the metal. This compound crystallizes in the triclinic space group $P\bar{1}$. The asymmetric unit contains two Zn(II), four L^{-1} ligands and four lattice water molecules. Both the Zn(II) metal ions show



Fig. 4. Topological view of (4,4)-connected net in 1.

distorted tetrahedral geometry with ligation from two monodentate carboxylate O (Zn–O = 1.950(3)–1.959(3) Å) and two benzimidazole N (Zn–N = 2.021(4)–2.014(4) Å) from four different \mathbf{L}^{-1} ligands. The structure consists of rectangular-grid 2D sheets generated along the crystallographic *ab* plane through combination of four \mathbf{L}^{-1} and four Zn (II) ions (Fig. 5).

These 2D interpenetrated sheets stack in $\dots ABAB \dots$ fashion through non-covalent interactions involving solvent molecules to form an overall 3D structure (Figs. 6 and 7).

The four water molecules themselves form a linear chain via strong H-bonding interactions $(OW1\cdots OW2 = 2.939(11) \text{ Å})$, $(OW2\cdots OW3 = 2.797(9) \text{ Å})$ and $(OW3\cdots OW4 = 2.788(11) \text{ Å})$. In addition, both OW1 and OW4 $(O8\cdots OW1 = 3.000(9), OW4\cdots O8 = 2.832(10) \text{ Å})$ show strong H-bonding interactions with the available carboxylate O (Fig. 8). This structure like **1** is densely packed with solvent accessible volume only 9.0% of the total unit cell volume.

3.3. Crystal structure of $\{[Zn(L)_2] \cdot 3/2H_2O\}_n$ (3)

When dabco is used as a co-ligand with NaOH base, a different structure is formed. Single crystal X-ray diffraction studies reveal



Fig. 6. ... ABAB... stacking mode between two 2D sheets in 2.

that **3** crystallizes in the monoclinic space group C2/c. The asymmetric unit consists of one Zn(II), two L^{-1} and one and a half lattice water molecules. The metal ions show distorted tetrahedral geometry with ligation from two monodentate carboxylate O (Zn–O = 1.961(3) – 1.993(3) Å) and two benzimidazole N (Zn–N = 2.050(3)–2.061(3) Å) from four independent L^{-1} ligands. A non-interpenetrated 2D sheet is formed along the crystallographic *bc* plane by four L^{-1} and four Zn (II) ions (Fig. 9).

These 2D sheets further connect to give a 3D framework through the multi-point H-bonding interactions between lattice water molecules, non-coordinated carboxylate O atoms as well as $\pi \cdots \pi$ stacking interactions between adjacent layers (Fig. 10). The solvent accessible volume again is very low; only 7.3% of the total unit cell volume.

3.4. Crystal structure of $\{[Cd(L)_2] \cdot 3H_2O\}_n$ (4)

Compound **4** also crystallizes in the monoclinic space group C2/c as a non-interpenetrated structure. The asymmetric unit contains two **L**⁻¹, one Cd(II) and three lattice water molecules. Each Cd(II) is six coordinated with a distorted octahedral geometry (Fig. 11), with ligation from four O (Cd–O = 2.270(3)–2.474(3) Å) of two ligands **L**⁻¹ in chelating mode and two N (Cd–N = 2.262(3)–2.273(3) Å) of two benzimidazole moieties. In this case also, a non-interpenetrated 2D sheet is formed along the crystallographic



Fig. 5. 2D sheet view along *ab* plane in 2.



Fig. 7. Water chain supported by carboxylate oxygen running along b axis in 2.



Fig. 8. 3D architecture showing embedded water molecules in space-fill model along *a* axis in 2.



Fig. 9. 2D view along bc plane in 3.

bc plane by four L^{-1} and four Cd(II) ions (Fig. 12). These 2D sheets stack in *…ABAB…* fashion through non-covalent interactions involving lattice water molecules to form an overall 3D structure (Fig. 13). The 3D structure is further stabilized by a weak C–H*…* π interactions (3.834(3) Å) between the benzimidazole moieties. The solvent accessible volume again is quite low i.e., 10.7% of the total unit cell volume.

Thermal stabilities of all the complexes were examined (see Supporting information). Compound **1** shows a weight loss of 10.01% (expected = 9.99%) in the temperature range, 80-110 °C corresponding to the loss of seven lattice water molecules. Decom-

position of the framework is achieved only above 350 °C. Compound **2** exhibits a weight loss of 5.95% (expected = 5.96%) in the range, 80–110 °C that corresponds to loss of four lattice water molecules and the framework decomposes above 310 °C. Compound **3** shows a weight loss of 2.27% (expected = 2.27%) due to removal of lattice water molecules and the framework shows stability upto 350 °C. Likewise, **4** shows a weight loss of 8.05% (expected = 8.04%) in the range, 80–120 °C corresponding to loss of lattice water molecules. It is also stable upto 350 °C and its decomposition starts beyond 350 °C. The complex **1** was heated at 110 °C under vacuum for 4 h. The PXRD pattern of evacuated complex **1** (i.e., complex **1**') shows that the framework remains intact upon removal of water molecules (see Supporting information).

4. Luminescence properties

Complexes showing luminescence properties are of immense current interest because of their variety of applications in photochemistry, chemical sensors, as well as in electroluminescent displays [43–45]. Luminescence properties of complexes of d^{10} metal ions with nitrogen and carboxylate donor ligands have been reported in the literature [46]. The luminescence properties of 1-4 and metal-free HL were investigated in the solid-state at room temperature and are shown collectively in Fig. 14. Upon excitation at 343 nm. **1–3** exhibit unsymmetrical emission as a pair of bands at 510, 540 and 530 nm and correspondingly at 450, 440 and 420 nm, respectively. The emission bands at higher energy could possibly be due to intra-ligand interaction as the profile of the emission band is similar to that of HL, while the low energy band probably originates from ligand-to-metal charge transfer [47]. For complexes 1-3, the luminescence intensity gradually decreases as the number of water molecules per Zn(II) center increases due to



Fig. 10. 3D view along *c*-axis with lattice aqua molecules in space-fill model in 3.



Fig. 11. Coordination modes of ligand L^{-1} in **4**.

vibrational guenching [48] by water molecules. To probe the effect of water molecules on the intensity of luminescence, the evacuated complex 1' is excited at 343 nm. The lower wavelength intra-ligand charge transfer band disappears with concomitant significant increase in intensity of the longer wavelength band. This band, attributed to ligand-to-metal charge transfer, is greatly enhanced due to absence of vibrational quenching by lattice water molecules as in **1**. The red-shift of this band by \sim 35 nm may be due to slight changes in the structure around the metal ions upon removal of lattice water molecules. Complex 4 also exhibits two emission bands centering at \sim 450 and 490 nm due to intra-ligand and ligand to metal charge transfer transitions [49] as in 1-3. Compared with the ligand, which shows very less luminescence, the luminescence enhancement in these complexes result from increased rigidity of the ligand upon metal coordination that reduces loss of energy by radiationless decay [50]. The Zn...Zn (more than 6.6 Å) and Cd…Cd (more than 6.5 Å) distances are long enough to contribute to cluster-centered transition caused by metal---metal interaction in these complexes.



Fig. 12. 2D view along bc plane in 4.



Fig. 13. 3D view along *c*-axis with lattice water molecules in space-fill model in 4.



Fig. 14. Solid-state emission spectra at room temperature.

5. Conclusion

In conclusion, we have described construction of four new coordination polymers under hydrothermal conditions. We have chosen unsymmetrical semi-flexible mixed donor based organic ligand for construction of coordination polymers with d^{10} metal ions. Solid-state emissions of the complexes have been achieved at room temperature. We are in process to synthesize similar type ligands for constructing coordination polymers for various applications.

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Appendix A. Supplementary data

CCDC 786124, 881367–881369 contain the supplementary crystallographic data for 1–4. 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) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/ 10.1016/j.poly.2012.06.055.

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