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Positional isomeric effect of phenylenediacetate on the construction of mixed-ligand Cd^{II} coordination frameworks

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ABSTRACT

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Keywords: Cd(II) complexes Phenylenediacetate Isomeric effect Luminescent properties Three Cd^{II} coordination polymers merging two types of flexible ligands, phenylenediacetate isomers (*o/m/p*pda = 1,2/1,3/1,4-phenylenediacetate) and 1,4-bis(imidazol-1-ylmethyl)benzene (bix), have been hydrothermally synthesized and characterized. [Cd(*o*-pda)(bix)]_n (**1**) and [Cd(*m*-pda)(bix)_{0.5}(H₂O)]_n (**2**) have 2D coordination networks containing hetero-helical subunits, in which **1** features a (3,5)-connected (4²·6) (4²·6⁷·8) topology, but **2** shows a (3,4)-connected V₂O₅-type net with (4²·6)(4²·6³·8) topology. Different from **1** and **2**, [Cd(*p*-pda)(bix)]_n (**3**) displays a 5-fold interpenetrating **dia** framework, indicating structuredirecting effect of the positions of acetate groups in pda.

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The world-wide interest in metal-organic frameworks (MOFs) focuses not only on seeking their potential application in gas storage, ion-exchange, catalysis, luminescence, magnetic and electronic devices, but also for the intriguing variety of architectures and topologies [1,2]. As a well known fact, the judicious choice of appropriate metal ions and tailor-made organic precursors is the dominating factor to building well-defined coordination frameworks. In this point, the flexibility of conformational freedom ligands favors the formation of alluring topological structures [3]. Despite the lack of predictability, some flexible N/O-donor ligands with multiform conformations have been used as excellent candidates for rationally constructing attractive networks. Among them, pyridyl/imidazolyl/ triazolyl ligands with alkyl spacers and flexible polycarboxylate ligands are outstanding examples [4]. However, the research about the positional isomeric effect of such flexible ligands on structural assemblies of coordination networks remains less explored yet [5].

As for the aromatic dicarboxyl ligands, the rigid isomers such as 1,2-, 1,3-, and 1,4-benzene-dicarboxylicate, have been extensively investigated [6]. In contrast, the flexible analogs, especially phenylenediacetate isomers with conformationally flexible pendants, have been seldom used for the construction of coordination polymers [4c,d,5]. Most recently, using phenylenediacetate isomers and flexible dipyridylligands, we have successfully synthesized a series of Zn^{II} and Cd^{II} complexes, where single or mixed conformations of such two types of flexible ligands are found [7]. As an extension of our work, herein, we report three Cd^{II} coordination polymers with three phenylenediacetate isomers (o/m/p-pda = 1,2/1,3/1,4-phenylenediacetate) and a flexible co-ligand 1,4-bis(imidazol-1-ylmethyl) benzene (bix), $[Cd(o-pda) (bix)]_n$ (1), $[Cd(m-pda) (bix)_{0.5}(H_2O)]_n$ (2), and $[Cd(p-pda)(bix)]_n$ (3). Their corresponding structural patterns vary from (3,5)- or (3,4)-connected 2D nets to a 5-fold interpenetrating 3D diamond motif, which demonstrate the positional isomeric effect of phenylenediacetate isomers on forming such coordination architectures. Further, the luminescent properties and thermal stabilities of **1–3** were also discussed.

Crystalline products of **1**, **2** and **3** were hydrothermally prepared by reacting Cd(CH₃COO)₂·2H₂O (0.1 mmol), o/m/p-pda (0.1 mmol), bix (0.05 mmol), NaOH (8.0 mg, 0.20 mmol) and H₂O (10 mL) at 160 °C [8], respectively, and characterized by elemental analysis, IR, PXRD and single crystal X-ray diffraction [9]. Single crystal X-ray diffraction reveals that **1** has a double-layer coordination motif. As illustrated in Fig. 1a, the asymmetric unit of **1** consists of one Cd^{II} ion, one bix ligand, and one fully deprotonated *o*-pda ligand. Each Cd^{II} center is seven—coordinated by five carboxylate oxygen donors from three individual μ_3 -o-pda and two nitrogen atoms from two *trans*-bix ligands, taking a pentagonal bipyramid (CdN₂O₅) geometry. The bond angles around Cd^{II} range from 50.84(5) to 162.29(6)°. The Cd–O (2.295(9)–2.546(2) Å) and Cd–N (2.281(0)–2.301(8) Å) bond lengths are in the normal range [10].

Interestingly, the two carboxylate groups of *o*-pda in **1** are situated at the two sides of the benzene ring with the *trans*-configuration (see Fig. S1a) and a dihedral angle of 10.4° . This kind of distortion between the separated flexible functional groups is mostly inclined to form helical structures [5a,7a,d11]. As expected, the *o*-pda ligands with chelating carboxylate groups connect the Cd^{II} centers into the leftand right-handed helical subunits that are parallel to the [010] direction (see Fig. 1b), with the Cd···Cd separation of 9.543 Å (length of the *b* axis). Furthermore, two hereto-handed helical arrays are

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Fig. 1. Views of **1**. (a) Coordination environment of Cd^{II} (50% probability displacement ellipsoids; H-atoms being omitted for clarity). Symmetry codes: #1 = -1 + x, 1 + y, 1 + z; #2 = x, 1 + y, z (b) The left- (plum) and right- (olive) handed helical chains based on *o*-pda and Cd^{II}. (c) 1D double-helical chains (left) containing 4-membered rings and 18-membered rings (right). (d) 2D double-layer. (e) 3D supramolecular network formed by $\pi \cdots \pi$ stacking. (f) Topological view of the 2D network (teal for Cd^{II} and olive for *o*-pda).

bridged via O3 atoms of carboxylates to form a double-helical array, which contains alternate 4-membered and 18-membered rings with the Cd…Cd distances of 3.970 and 7.111 Å, respectively (see Fig. 1c).

On the other hand, the bidentate bix ligands adopt the *trans*conformation to pillar these double helical chains into a 2D doublelayer (see Fig. 1d), in which the Cd···Cd distance across the bix spacer is 15.636 Å. The adjacent 2D patterns exhibit a parallel stacking along [101] in an ABAB fashion (see Fig. 1e), with the presence of π ··· π interactions resulting from interlayer imidazole rings of bix (centroid-to-centroid distance = 3.780 Å and dihedral angle = 21.9°).

Topologically, each *o*-pda acts as a 3-connected node, and the Cd^{II} ion can be regarded as a 5-connected node. Thus, the 2D layer shows a novel (3,5)-connected topology (see Fig. 1f) with the point symbol of $(4^2.6)(4^2.6^3.8)$, as analyzed by TOPOS program [12].

Complex **2** displays a wave-like 2D coordination layer, and the asymmetric unit contains one Cd^{II} ion, one fully deprotonated *m*-pda, one coordinated water molecule, and half a bix ligand. The Cd^{II} center adopts a distorted octahedral geometry, provided by two μ_3 -*m*-pda



Fig. 2. Views of **2.** (a) Coordination environment of Cd^{II} (50% probability displacement ellipsoids; H-atoms being omitted for clarity). Symmetry codes: #1 = -x, 1 - y, 1 - z; #2 = 1 + x, y, -1 + z; #3 = 1 - x, 2 - y, -1 - z. (b) 1D double-helical chains (left) containing 8-membered rings and 20-membered rings (right). (c) 2D wave-like layer. (d) 3D architecture formed by O-H···O bonds. (e) Topological view of the 2D V₂O₅-type net (teal for Cd^{II} and olive for *m*-pda). (f) Topological view showing the 3D trinodal (3,5,5)-connected net (teal for Cd^{II}, olive for *o*-pda, and plum for aqua ligand).

ligands (Cd–O=2.252(2)–2.406(2) Å) as well as one water and one bix ligands (Cd–O=2.348(2) and Cd–N=2.263(2) Å), as depicted in Fig. 2a. Similar to *o*-pda of **1**, the *m*-pda ligand shows the μ_3 -linking mode and takes the *trans*-configuration (see Fig. S1b) but with the dihedral angle between two carboxylate groups of 60.3°. Therefore, the *o*-pda ligands also connect the Cd^{II} ions to form double-helical arrays containing both 8-membered and 20-membered rings, with the Cd···Cd distances of 4.697 and 7.907 Å, respectively (see Fig. 2b). Compared with **1**, due to the presence of water ligands occupying the coordination sites in **2**, the *trans*-conformation bix ligands alternately bridge these double chains to result in a 2D wave-like layer, with the Cd···Cd distance of 14.824 Å separated by bix (see Fig. 2c).

Remarkably, $O-H\cdots O$ H-bonds are observed between the water ligands (05W) and carboxylate oxygen atoms (O2 and O4) (for intralayer: O5W–H \cdots O2, 2.748 Å and 152.7°; for interlayer: O5W– $H\cdots O4$, 2.747 Å and 172.2°), which not only stabilize the 2D layers but also extend them to form a 3D architecture (see Fig. 2d). Topological analysis reveals that **2** has a (3,4)-connected V₂O₅-type coordination net with the point symbol of (4².6)(4².6³.8), with *m*-pda and Cd^{II} serving as 3- and 4-connected nodes, respectively (see Fig. 2e). Further, if the H-bonds are taken into account, the water ligands can be regarded as 3-connected nodes, while both Cd^{II} and *m*pda²⁻ can be treated as 5-connected nodes. Thus, the overall 3D network is trinodal (3,5,5)-connected with the point symbol of (3.4.5) $(3.4^2.5.6^4.7.8)(3.4^3.5^2.6^2.7^2)$ (see Fig. 2f).

Complex 3 has a 5-fold interpenetrating diamond framework, and the fundamental building unit of **3** is composed of one Cd^{II} ion, a disordered *p*-pda and an unordered bix ligand. Each Cd^{II} ion takes a highly distorted octahedral sphere, completed by four carboxylate oxygen atoms (Cd-O1 = 2.48(4) and Cd-O2 = 2.293(2) Å) from two *p*-pda anions and two imidazole nitrogen atoms from two bix ligands (Cd-N=2.217(4) Å), as shown in Fig. 3a. Each *p*-pda adopts the transbis(chelating) binding mode to bridge the Cd^{II} centers into 1D infinite chains along the [001] axis, with the Cd \cdots Cd separation of 12.048 Å. Further, the trans-conformation bix tectons extend such 1D chains (Cd···Cd distance across the bix spacer of 15.782 Å) to afford a 3D dia network(see Fig. 3b). Notably, the large adamantanoid cages in a single dia network have the maximum dimension of $17.811 \times 35.610 \times 39.477$ Å 3 (see Fig. 3c), which allows the other selfsame dia nets to penetrate and thus, affords a 5-fold interpenetrating **dia** framework (see Fig. 3d). Analysis of the interpenetrating fashion in **3** with TOPOS suggests the so-called 'normal' type for diamondoid frames [13], which belongs to class Ia with the interpenetration vector being equal to the *b* axis [14].

Structural diversification of **1–3** may mainly arise from two factors: (i) the phenylenediacetate isomers with different orientations, torsions,



Fig. 3. Views of **3.** (a) Coordination environment of Cd^{II} (50% probability displacement ellipsoids; H-atoms being omitted for clarity). Symmetry codes: #1 = -x, y, 0.5 – z; #2 = -x, 2 – y, –z; #3 = 0.5 - x, -0.5 - y, 1 – z. (b) 3D network (left) and a single 3D **dia** net (right). (c) A single **dia** cage unit. (d) Topological representation of 5-fold interpenetrating framework (left) and 5-fold interpenetrating nets viewed along the [010] direction (right).

and binding modes of the carboxylate groups (see Fig. S1); (ii) the diverse coordination geometries of Cd^{II} (7-coordinated in **1** and 6-coordinated in **2/3**). In **1** and **2**, the μ_3 -o/m-pda ligands with *trans*-conformation and suitable torsion of the carboxylate groups link the Cd^{II} centers to form double-helical chains, in which the carboxylate oxygen atoms occupy the equatorial planes of Cd^{II} spheres. Therefore, the *exo*-bidentate bix ligands extend these chains from the apical positions Cd^{II} spheres to result in different 2D networks in **1** and **2**. In contrast, the *trans*-bis(chelating) *p*-pda ligands in **3** act as the linkers to extend the six-coordinated Cd^{II} centers into 1D infinite arrays, and further, the *trans*-bix connectors bridge these chains by holding the two remanent sites of Cd^{II} spheres to form a **dia** framework.

For the PXRD patterns for **1**, **2** and **3**(see Fig. S2), although the experimental patterns have a few unindexed diffraction peaks and some are slightly broadened in comparison with those simulated from the single-crystal diffraction data, it can still be regarded that the bulk as-synthesized materials represent the pure phase of **1**, **2** and **3**. Thermogravimetric analysis (see Fig. S3) of **2** indicates that the weight loss between 130 and 180 °C corresponds to the release of coordination water molecules (obsd 4.26% and calcd 4.08%), and the anhydrous composition begins to decompose at 289 °C. The TGA curves of **1** and **3** exhibit similar weight loss stage. There are no lattice/ coordination water molecules therein and thus, decomposition of the organic components occurs at 295 °C for **1** and 303 °C for **3**, respectively.

To examine the luminescent properties of the d¹⁰ metal complexes, solid-state fluorescence spectra of **1–3** were investigated at room temperature. As illustrated in Fig. 4, the intense broad emission peaks appear at 412 nm ($\lambda_{ex} = 340$ nm) for **1**, 373 nm ($\lambda_{ex} = 300$ nm) for **2**, and 383 nm ($\lambda_{ex} = 320$ nm) for **3**. As for the free organic ligands, the emissions are observed at 392 nm ($\lambda_{ex} = 340$ nm) for bix [15], 392 nm ($\lambda_{ex} = 300$ nm) for o-pda [7c], 367 nm ($\lambda_{ex} = 300$ nm) for *m*-pda [7a], and 466 nm ($\lambda_{ex} = 360$ nm) for *p*-pda [7b]. Thus, the emissions for **1–3** are neither metal-to-ligand charge transfer (MLCT) nor ligand to metal transfer (LMCT), since the Cd^{II} ions are difficult to oxidize or reduce, and are likely attributed to the intraligand $\pi \rightarrow \pi^*$ or $\pi \rightarrow n$ transitions. Similar emission bands in the region of 350~420 nm for other d¹⁰ metal coordination polymers with pda^{2–} and bix ligands have been observed [4,5,7].

A series of Cd^{II} coordination frameworks with mixed-ligands of phenylenedicarboxylate isomers (o/m/p-pda) and 1,4-bis(imidazol-1-ylmethyl)benzene (bix) have been hydrothermally synthesized and structurally characterized. The corresponding structural patterns vary from (3,5)- or (3,4)-connected 2D nets (o-pda for 1 or m-pda for 2) to a 5-fold interpenetrating **dia** framework (p-pda for 3). These results



Fig. 4. Solid-state fluorescent emission spectra of 1–3 (blue for 1, green for 2, and red for 3).

will enrich the current research of isomeric effect of flexible ligands on constructing coordination polymers and provide new insights into its application in designing such crystalline materials with desired structures and properties.

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Appendix A. Supplementary Data

CCDC 787445–787447 contain the supplementary crystallographic data for complexes **1**, **2** and **3**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www. ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found in the section of supporting information. Supplementary data to this article can be found online at doi:10.1016/j.inoche.2011.01.029.

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