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Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gcoo20>

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Hai-Wei Kuai ^a, Xiao-Chun Cheng ^a & Xiao-Hong Zhu ^a

^a Faculty of Life Science and Chemical Engineering, Huaiyin Institute of Technology, Huaian 223003, P.R. China

Published online: 04 May 2011.

To cite this article: Hai-Wei Kuai, Xiao-Chun Cheng & Xiao-Hong Zhu (2011) Synthesis, structure, and properties of a coordination polymer based on N- and O-donors, Journal of Coordination Chemistry, 64:9, 1636-1644, DOI: [10.1080/00958972.2011.578134](https://doi.org/10.1080/00958972.2011.578134)

To link to this article: <http://dx.doi.org/10.1080/00958972.2011.578134>

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Synthesis, structure, and properties of a coordination polymer based on N- and O-donors

HAI-WEI KUAI*, XIAO-CHUN CHENG and XIAO-HONG ZHU

Faculty of Life Science and Chemical Engineering,
Huaiyin Institute of Technology, Huaian 223003, P.R. China

(Received 18 January 2011; in final form 18 March 2011)

A ternary coordination polymer, $[\text{Cd}(\text{L})(\text{pbda})_{0.5}]_n$ [HL = 3,5-bis(2-pyridylmethyl)aminobenzoic acid, pbda = dianion of 1,4-benzenedicarboxylic acid], has been synthesized and characterized by elemental analysis, FT-IR spectroscopy, powder X-ray diffraction, thermogravimetric analysis, and single-crystal X-ray diffraction analysis. The single-crystal X-ray crystallography reveals that the complex is a 2-D wave-like network. The cadmium has an unsymmetrical seven-coordinate $[\text{CdN}_2\text{O}_5]$ geometry, coordinated by two nitrogens and five oxygens from L^- and pbda. Hydrogen bonds between the uncoordinated (2-pyridylmethyl)amino groups from adjacent layers form dimers across the inversion center, superposing different layers to construct a 3-D framework. To the best of our knowledge, $[\text{Cd}(\text{L})(\text{pbda})_{0.5}]_n$ represents the first example of a complex containing 3,5-bis(2-pyridylmethyl)aminobenzoate.

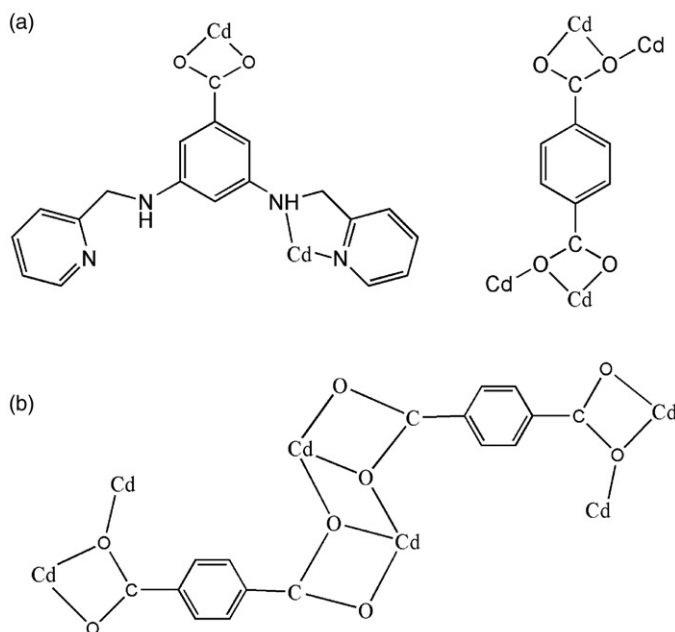
Keywords: Crystal structure; Mixed-ligand; Hydrogen bonding dimer

1. Introduction

Design and synthesis of supramolecular frameworks causes incessant interest, justified due to their intriguing architectures and potential applications in heterogeneous catalysis, ion-recognition, nonlinear optics, chemical absorption, electroconductivity, and magnetism [1, 2]. A series of open frameworks with abundant structural motifs have been discussed in some comprehensive reviews [3]. An effective strategy for constructing such extended porous frameworks is to select suitable ligands as building blocks that enable the control of structural motifs and functional properties [4].

Rigid or flexible N-, O-containing multidentate ligands are regarded as excellent candidates for building the blocks of desirable frameworks [5]. However, compared with other N- or O-donors, those containing N,N-chelating bidentate group {such as [(2-pyridylmethyl)amino]} and carboxylate can exhibit distinctive features. The intrinsic N,N-chelating may induce new structural evolution and maintain the desired specific coordinating configuration [6]. Moreover, other rigid aromatic carboxylate ligands, such as 1,4-benzenedicarboxylic acid (H_2pbda), can be introduced as a bridging rod. Thus, structural prediction of the resulting polymeric species may be possible.

*Corresponding author. Email: hytshy@126.com



Scheme 1. (a) Schematic representation of the coordination modes of the L^- and $pbda^{2-}$ in the complex; (b) centrosymmetrical carboxylate-bridged binuclear secondary building unit (SBU) $[Cd_2(COO)_2]$.

Second, this kind of ligand contains both rigid and flexible coordinating groups, providing a greater degree of structural diversity reflected by the unique structural features of the complex. Third, due to the presence of N–H, hydrogen bonding interactions are available to extend the network [7].

In this contribution we describe the synthesis, crystallography, and properties of a ternary Cd(II) coordination complex with 3,5-bis(2-pyridylmethyl)aminobenzoic acid (HL) $[Cd(L)(pbda)_{0.5}]_n$ (scheme 1). As expected, the complex is a 2-D wave-like layer, which further connected into a fascinating 3-D supramolecular framework through hydrogen bonds between uncoordinated (2-pyridylmethyl)amino groups from the adjacent 2-D layers. The uncoordinated (2-pyridylmethyl)amino groups create strong intermolecular N–H \cdots N hydrogen bonds, important in stabilization and, due to the presence of the uncoordinated (2-pyridylmethyl)amino groups, the coordination compound may be utilized as Lewis base in heterogeneous catalysis [8]. FT-IR spectroscopy, powder X-ray diffraction (PXRD), and thermal stability have been studied. $[Cd(L)(pbda)_{0.5}]_n$ represents the first example of a complex containing 3,5-bis(2-pyridylmethyl)aminobenzoate.

2. Experimental

2.1. Materials and characterization

Commercially available chemicals and solvents of reagent grade were used as received. HL was readily prepared according to the literature [9]. Elemental analysis for C, H,

and N was performed on a Perkin-Elmer 240C Elemental analyzer. Thermogravimetric analysis (TGA) was carried out on a simultaneous SDT 2960 thermal analyzer under nitrogen with a heating rate of $10^{\circ}\text{C min}^{-1}$. FT-IR spectroscopy was recorded from 400 to 4000 cm^{-1} on a Bruker Vector22 FT-IR spectrophotometer using KBr pellets. PXRD patterns were measured on a Shimadzu XRD-6000 X-ray diffractometer with Cu-K α ($\lambda = 1.5418\text{ \AA}$) radiation at room temperature.

2.2. Synthesis of $[\text{Cd}(\text{L})(\text{pbda})_{0.5}]_n$

A mixture of $\text{Cd}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (49.4 mg, 0.1 mmol), HL (33.4 mg, 0.1 mmol), H_2pbda (8.3 mg, 0.05 mmol), and NaOH (8.0 mg, 0.2 mmol) in 10 mL of H_2O was stirred for 10 min in air, then transferred to a 16 mL Teflon-lined stainless steel container and heated at 180°C for 3 days. After the mixture was cooled to room temperature, light brown block crystals were obtained (yield: 37% based on HL). Anal. Calcd for $\text{C}_{23}\text{H}_{19}\text{N}_4\text{O}_4\text{Cd}$ (%): C, 52.34; H, 3.63; N, 10.61. Found (%): C, 52.30; H, 3.81; N, 10.44.

2.3. X-ray crystallography

Diffraction data for the complex were collected at 293(2) K with a Siemens SMART CCD diffractometer using graphite-monochromated (Mo-K α) radiation ($\lambda = 0.71073\text{ \AA}$), ω scans mode at different fixed positions of φ ; 5604 reflections were collected. The cell parameters were determined from full-matrix least-squares fit of 3390 reflections with θ from 2.41° to 27.70° . The structure was solved by direct methods and refined by full-matrix least-squares on F^2 to $R_1 = 0.0374$ ($wR_2 = 0.0938$) for 3458 reflections [$I > 2\sigma(I)$] collected from $2.00^{\circ} \leq \theta \leq 26.00^{\circ}$. Intensity data were corrected for Lorentz and polarization effects and a multi-scan absorption correction was performed. All non-hydrogen atoms were refined anisotropically. Hydrogens except on N3 and N4 were added geometrically and allowed to ride on their respective parent atoms. Hydrogens on N3 and N4 were located directly from the Fourier map. The contribution of these hydrogens was included in the structure factor calculations. All calculations were carried out on a PC computer using SHELXL-97 [10]. Details of crystal data, collection, and refinement are listed in table 1.

3. Results and discussion

3.1. Preparation

The hydrothermal reaction at 180°C of stoichiometric amounts of cadmium perchlorate hexahydrate with HL and H_2pbda in the presence of NaOH provided brown single crystals, $[\text{Cd}(\text{L})(\text{pbda})_{0.5}]_n$, which are stable in the open air.

3.2. Structural description

X-ray crystallographic analysis reveals that the complex is a 2-D wave-like layer. It crystallizes in the triclinic system with space group $\text{P}\bar{1}$. In the asymmetrical unit, there

Table 1. Crystallographic data and structure refinement for the complex.

Empirical formula	C ₂₃ H ₁₉ N ₄ O ₄ Cd
Formula weight	527.82
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	Pī
Unit cell dimensions (Å, °)	
<i>a</i>	9.8381(6)
<i>b</i>	10.1685(7)
<i>c</i>	10.997(7)
α	70.803(1)
β	77.667(1)
γ	84.283(1)
Volume (Å ³), <i>Z</i>	1014.46(11), 2
Calculated density (Mg m ⁻³)	1.728
Absorption coefficient (mm ⁻¹)	1.117
<i>F</i> (000)	530
Crystal dimension (mm ³)	0.20 × 0.18 × 0.18
θ range for data collection (°)	2.00–26.00
Limiting indices	−12 ≤ <i>h</i> ≤ 12; −12 ≤ <i>k</i> ≤ 9; −13 ≤ <i>l</i> ≤ 8
Reflections collected/unique	5604
Independent reflection	3920 [<i>R</i> (int) = 0.0493]
Completeness to $\theta = 26.00$ (%)	98.0
Absorption correction	Multi-scan
Max. and min. transmission	0.8243 and 0.8075
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	3920/0/285
Goodness-of-fit on <i>F</i> ²	1.013
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0374, <i>wR</i> ₂ = 0.0938
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0422, <i>wR</i> ₂ = 0.0963
Largest difference peak and hole (e Å ⁻³)	1.215 and −1.461

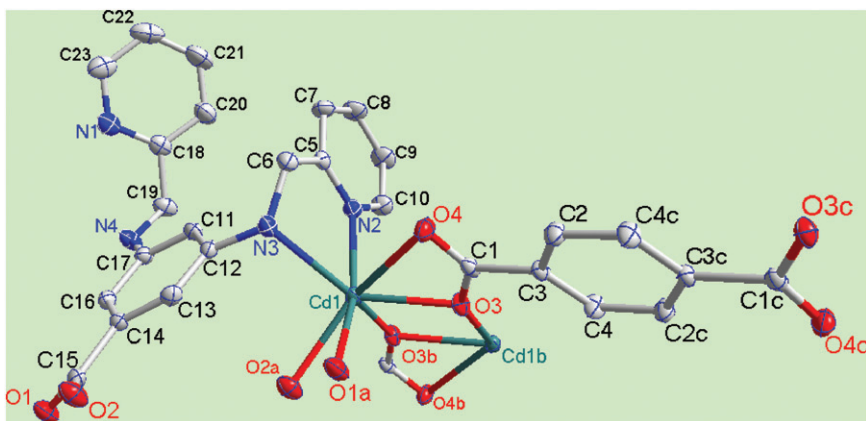


Figure 1. Diagram showing the coordination geometry of Cd(II) with 30% probability displacement ellipsoids. Hydrogens were omitted for clarity. Symmetry transformations: a: $-x, 1-y, 1-z$; b: $-x, 1-y, 2-z$; c: $-x, 2-y, 2-z$.

are one Cd, one L[−], and 0.5 pbda. Each Cd(II) is seven-coordinate with two nitrogens from L[−] (figure 1), two oxygens from L[−], and three oxygens from two different pbda to furnish a hemidirected [CdN₂O₅] geometry. Selected bond distances and angles are listed in table 2. Pbda plays a very important role in the construction of the complex.

Table 2. Selected bond lengths (Å) and angles (°) for the complex.

Cd(1)–O(3)	2.287(2)
Cd(1)–O(4)	2.655(3)
Cd(1)–N(2)	2.313(3)
Cd(1)–N(3)	2.459(3)
Cd(1)–O(1a)	2.322(3)
Cd(1)–O(2a)	2.385(3)
Cd(1)–O(3b)	2.355(2)
O(3)–Cd(1)–O(4)	52.14(10)
O(3)–Cd(1)–N(2)	100.06(11)
O(3)–Cd(1)–N(3)	132.80(10)
O(1a)–Cd(1)–O(3)	87.09(11)
Cd(1)–O(3)–Cd(1b)	103.99(9)
O(2a)–Cd(1)–O(3)	120.41(11)
O(3)–Cd(1)–O(3b)	76.01(9)
O(4)–Cd(1)–N(2)	87.10(10)
O(4)–Cd(1)–N(3)	80.74(10)
O(1a)–Cd(1)–O(4)	81.08(10)
O(2a)–Cd(1)–O(4)	136.26(10)
O(3b)–Cd(1)–O(4)	124.63(9)
N(2)–Cd(1)–N(3)	71.75(10)
O(1a)–Cd(1)–N(2)	158.18(11)
O(2a)–Cd(1)–N(2)	132.69(11)
O(3b)–Cd(1)–N(2)	83.85(11)
O(1a)–Cd(1)–N(3)	88.21(10)
O(2a)–Cd(1)–N(3)	94.13(10)
O(3b)–Cd(1)–N(3)	144.25(10)
O(1a)–Cd(1)–O(2a)	55.26(11)
O(1a)–Cd(1)–O(3b)	117.95(11)
O(2a)–Cd(1)–O(3b)	83.38(10)

Symmetry transformations used to generate equivalent atoms: a: $-x$, $1-y$, $1-z$; b: $-x$, $1-y$, $2-z$.

Each carboxylate from pbda is $\mu_2\text{-}\eta^2\text{:}\eta^1$ -chelating/bridging coordination mode, resulting in the formation of a carboxylate-bridged binuclear secondary building unit {(SBU) $[\text{Cd}_2\text{COO}]_2$, (scheme 1)}. The Cd1...Cd1 distance $\#d(-x, 1-y, 2-z)$ in each SBU is 3.659 Å, indicating weak Cd...Cd contacts within the binuclear units [11]. Two nitrogens N2 and N3 from L^- chelate with the angle subtended at cadmium being 71.76(10)°; the chelating conformation leads to the generation of a five-membered non-coplanar N,N-chelating metallacycle. The other pyridyl ring does not coordinate, which implies that the complex may be utilized as a Lewis base in heterogeneous catalysis. The carboxylate O1C15O2 from L^- shows $\mu_1\text{-}\eta^1\text{:}\eta^1$ -chelating coordination (scheme 1). The L^- ligands doubly interconnect the SBUs $[\text{Cd}_2(\text{COO})_2]$ to yield one-dimensional zigzag chains (figure 2). Progression of the 1-D chain through pbda forms a fascinating 2-D network (figure 3).

Hydrogen bonds $[\text{N4-H4N}\cdots\text{N1}\#e, \text{N4}\#e\text{-H4N}\#e\cdots\text{N1} (\#e: 1-x, -y, 1-z); \text{N4-H4N}, 0.92 \text{ \AA}; \text{H4N}\cdots\text{N1}, 2.10 \text{ \AA}; \text{N4}\cdots\text{N1}, 2.98 \text{ \AA}; \angle\text{N4-H4N}\cdots\text{N1}, 162^\circ]$ existing between uncoordinated (2-pyridylmethyl)amino from adjacent 2-D layers form dimers across the inversion center. The strong intermolecular interactions are rather significant in stabilizing the supramolecular architecture. This system of strong hydrogen bonds superposes different layers to construct a three-dimensional framework (figure 4).

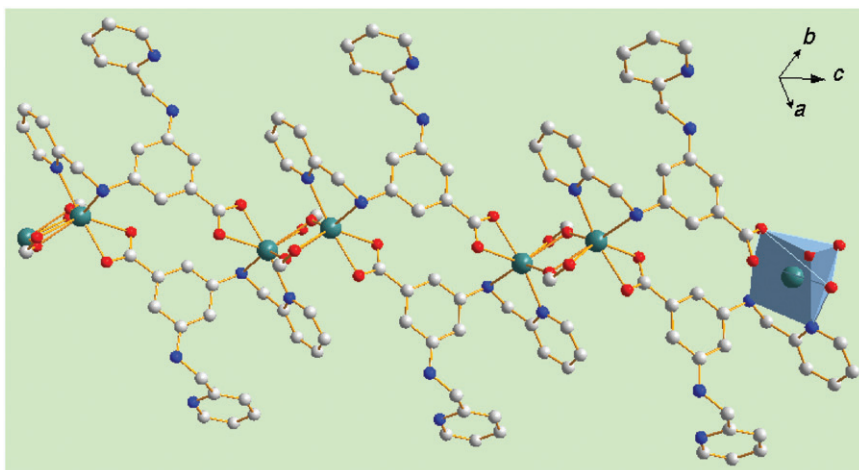


Figure 2. View of the 1-D zigzag chain linked *via* the L^- doubly interconnecting the SBUs.

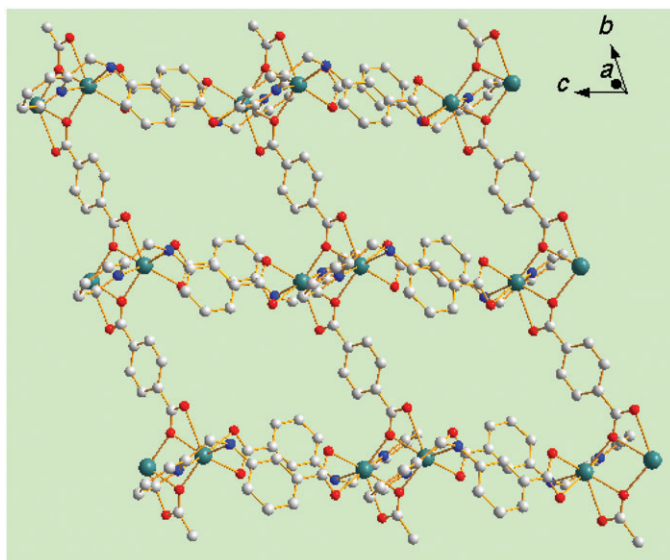


Figure 3. View of the 2-D wave-like network formed through pbda ligands bridging 1D chains in the complex. Uncoordinated (2-pyridylmethyl)amino groups were omitted for clarity.

3.3. FT-IR spectroscopy

The absence of bands at $1690\text{--}1730\text{ cm}^{-1}$ indicates complete deprotonation of the carboxylic group [12]. Although there are two different kinds of coordination of carboxylate in the complex (chelating and chelating/bridging), from the IR spectrum only one group of split asymmetric and symmetric stretching bands of COO^- are observed (1607 cm^{-1} , 1550 cm^{-1} and 1409 cm^{-1} , 1350 cm^{-1} , respectively). The bands at 1518 cm^{-1} may be assigned to stretching vibrations of C–H in the benzene rings [12], and the band at 3066 cm^{-1} attributable to stretching vibrations of C–H in pyridyl rings [13].

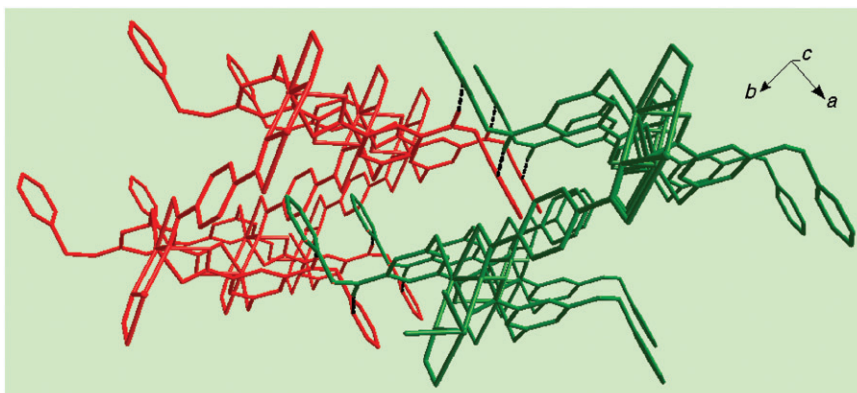


Figure 4. View of the 3-D supramolecular framework along the c direction constructed by hydrogen bonds between the uncoordinated (2-pyridylmethyl)amino groups from adjacent 2-D layers. The (2-pyridylmethyl) of the coordinated (2-pyridylmethyl)amino is omitted for clarity.

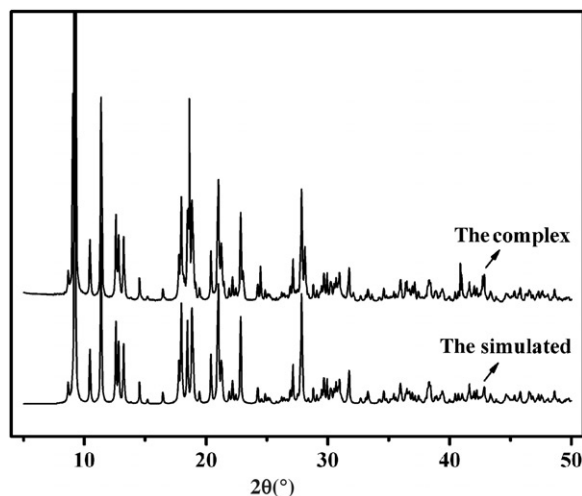


Figure 5. PXRD patterns for the complex recorded (upper) and (below) the simulated PXRD pattern calculated from the single-crystal structure. The diffraction peaks of both simulated and experimental patterns match well.

The characteristic peak of the uncoordinated N–H from L^- appears at 3340 cm^{-1} , whereas the coordinated shift to 3262 cm^{-1} [14], agreeing with single-crystal X-ray analysis.

3.4. PXRD and TGA

The PXRD pattern for the complex is recorded (upper) and (below) the simulated PXRD pattern is calculated from the single-crystal structure (figure 5). The diffraction peaks of both simulated and experimental patterns match well in key positions.

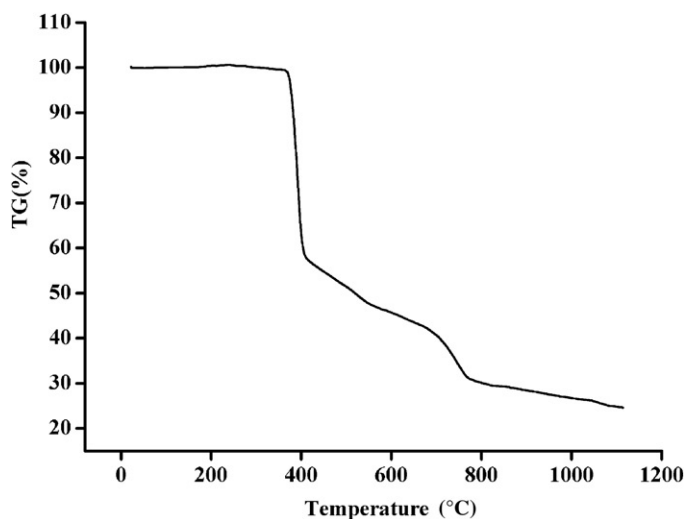


Figure 6. TGA curve of the complex.

The thermal behavior of the coordination compound has been deduced from the TGA curve in N_2 with a heating rate of $10^\circ C\ min^{-1}$. The TGA indicates that the compound is thermally stable to $376^\circ C$, above which a sharp weight loss followed by a long tail is observed due to the framework decomposing rapidly. The total weight loss from 28 to $1114^\circ C$ is 75.56%, which is in agreement with the weight loss calculated for the loss of ligands leading to the formation of CdO (75.67%) as final residuals above $1100^\circ C$ (figure 6).

4. Conclusion

We have designed HL and obtained a ternary coordination polymer, $[Cd(L)(pbda)_{0.5}]_n$, with 2D wave-like networks through hydrothermal reaction at $180^\circ C$ of stoichiometric amounts of cadmium perchlorate hexahydrate with HL and H_2pbda in the presence of NaOH. Due to HL and the presence of H_2pbda as bridging rods, the structural features of the resulting polymer could have been predicted to some extent. The intermolecular hydrogen bonding interactions superpose different 2-D layers to construct a 3-D framework. The polymer has been fully characterized by elemental analysis, FT-IR spectroscopy, PXRD, TGA, and single-crystal X-ray diffraction. Further investigations on properties of the complex will be carried out in our future research.

Supplementary material

Complete listings of bond distances and angles, hydrogen atom coordinates and isotropic displacement parameters, anisotropic displacement parameters, and least-squares plans are available either quoting the deposition number CCDC-803810 (12 Union Road, Cambridge, CB2 1EZ, UK) or from the authors on request.

Acknowledgments

The authors gratefully acknowledge the Natural Science Foundation of Jiangsu Province of China (BK2008195) and the Science Research Foundation of Huaiyin Institute of Technology (HGQ0649 and 2517045) for financial support of this work.

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