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Research Article

Crystal Structure and Catalytic Activity of A Novel Cd(II) Coordination Polymer Formed by Dicarboxylic Ligand

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Abstract

A new Cd(II) coordination polymer, {[$Cd_3(L)_2(DMF)_2(H_2O)_2$]·H_2O}n (H_2L = 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylic acid) was synthesized by one-pot synthesis method from 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylic acid, NaOH, DMF, and $Cd(NO_3)_2·4H_2O$. Its structure was determined by elemental analysis and single crystal X-ray diffraction. Structural analysis shows that three Cd(II) ions are all six-coordinated with four oxygen atoms of four 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylate ligands and two O atoms from two DMF molecules (Cd1) or two oxygen atoms of two coordinated H_2O molecules (Cd2 and Cd3) to form an octahedral coordination geometry. The Cd(II) coordination polymer displays a 1D chained structure by the bridging carboxylate groups from 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylate ligands. The conversion of benzaldehyde is 90.9%, which is 40~50% higher than those of the other three aldehydes (4-methylbenzaldehyde, *p*-methoxybenzaldehyde and 3-chlorobenzaldehyde), so the Cd(II) coordination polymer catalyst shows better catalytic activity for the coupling reaction of benzaldehyde, phenylacetylene, and piperidine than the other three aldehydes.

Keywords: 1,3-Bisbenzyl-2-imidazolidine-4,5-dicarboxylic acid; Cd(II) coordination polymer; Synthesis; Structural characterization; Catalytic property

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1. Introduction

Many studies show that the metal-organic coordination polymer materials have attracted wide interests among chemists because of their structural diversities and potential applications in many fields such as antitumor and antibacterial activities [1-5], luminescent properties [6-9], catalytic activities [10-12], magnetic properties [13-16], DNA-binding properties [17,18], and gas adsorption [19,20], and so on.

Owing to the abundant coordination modes, the ligands containing carboxylate group have occupied an important role in the construction of metal-organic coordination polymer materials [21-27]. However, so far, the researches on the catalytic property of Cd(II) complexes are less. Our research group has carried on the studies on the synthesis, novel structure and property of metal-organic coordination polymer materials [28,29].

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As part of our studies, in this paper, we have synthesized a novel Cd(II) coordination polymer by one-pot synthesis method from 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylic acid, NaOH, DMF and $Cd(NO_3)_2 \cdot 4H_2O$. And its structure has been determined by elemental analysis and X-ray single crystal diffraction. The catalytic activities for the coupling reaction of aldehydes (benzaldehyde, *p*-tolualdehyde, *p*-anisaldehyde and *m*-chlorobenzaldehyde), phenylacetylene, and piperidine with 1,4-dioxane as solvent at 120 °C for 12 h have been investigated. Interestingly, the Cd(II) coordination polymer catalyst shows better catalytic activity for the coupling reaction of benzaldehyde, phenylacetylene, and piperidine than the other three aldehydes.

2. Materials and Methods

2.1 Materials and measurements

1,3-Bisbenzyl-2-imidazolidine-4,5-dicarboxylic acid, NaOH, DMF, $Cd(NO_3)_2 \cdot 4H_2O$, and solvent used were reagent grade and purchased from Jinan Henghua Chemical Reagent Company. Element analyses (C, H and N) were carried out with an Elementar Vario III EL elemental analyzer (Hanau, Germany). The single crystal data of Cd(II) coordination polymer were obtained by means of a Bruker Smart CCD diffractometer (Bruker, Billerica, MA, USA).

Table 1. Crystallographic data and structure refinement for Cd(II) coordination polymer

Empirical formula	$C_{44}H_{50}Cd_3N_6O_{14}$
Formula weight	1111.70
Temperature/K	293(2)
Crystal system	Monoclinic
Space group	*C*2/*c*
a/Å	28.053(6)
b/Å	10.956(2)
c/Å	31.933(6)
α/°	90
β/°	111.04(3)
γ/°	90
Volume/Å^3	9160(3)
Z	8
ρ_{calc}, mg/mm^3	1.612
μ/mm^{-1}	1.002
S	1.025
F(000)	4512
Index ranges	$-33 \le h \le 33$, $-13 \le k \le 12$, $-37 \le l \le 37$
Reflections collected	33741
θ/°	3.07-25.01
Independent reflections	7967 [*R*(int) = 0.1681]
Data/restraints/parameters	7967/0/596
Goodness-of-fit on F^2	1.025
Refinement method	Full-matrix least-squares on F2
Final *R* indexes [I>=2*σ* (*I*)]	$R_1 = 0.0529$, $wR_2 = 0.0624$
Final *R* indexes [all data]	$R_1 = 0.1294$, $wR_2 = 0.0800$
Largest diff. peak/hole / e Å^{-3}	1.207 /-1.604

2.2 Synthesis of Cd(II) coordination polymer

The 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylic acid ligand (1.0 mmol, 0.3544 g) and NaOH (4.0 mmol, 0.160 g) were dissolved in 10 mL ethanol solution. A white precipitate formed immediately when $Cd(NO_3)_2 \cdot 4H_2O$ (0.5 mmol, 0.1543 g) was added to the above solution. Then 5.0 mL DMF was added and the precipitate dissolved. The reactants were continuously stirred at *ca.* 70 °C for 5 h. The resultant solution was filtered and left aside for crystallization at room temperature. The colorless block crystals appeared for 20 days (56 % yield). Elemental analysis (%) calculated for $C_{44}H_{50}Cd_2N_6O_{14}$: C, 47.49; H, 4.50; N, 7.56. Found (%): C, 47.58; H, 4.29; N, 7.41.

2.3 Crystal structure determination

The colorless block single crystal of Cd(II) coordination polymer (0.21×0.20×0.18 mm) was measured on a Bruker Smart APEX CCD diffractometer with graphite-monochromated Mo-*Kα* radiation (λ = 0.71073 Å) at 293(2) K. The structure was solved by direct method using SHELXS-97 program [30] and refined by full-matrix least squares on F^2 by means of the program SHELXL-97 [30]. The crystallographic data and processing parameters for Cd(II) coordination polymer are summarized in Table 1.

2.4 General procedure for the three component coupling reaction (A^3)

The A^3 coupling reactions of aldehyde (0.13 mmol), piperidine (0.15 mmol, 12.8 mg), phenylacetylene (0.17 mmol, 16.9 mg), 1,4-dioxane (1.5 g) using Cd(II) coordination polymer (40 mg) as catalyst were stirred for 12 h at 120 °C. After completion of the reaction, the mixtures were cooled to room temperature and the products obtained by centrifugation. The Cd(II) coordination polymer catalyst was dried at 60 °C under vacuum for 3 h and stored in a desiccator for its use in subsequent catalytic runs. The product was analysed by GC ((GC-1100, capillary column SE-54) using *n*-nonane as the external standard. The conversion and selectivity were calculated by Equations (1) and (2), respectively.

$$C_{benzaldehyde} = \frac{n_{initial} - n_{after\ reaction}}{n_{initial}} x100\% \quad (1)$$

$$S = \frac{n_{propargylamine}}{n_{initial} - n_{after\ reaction}} x100\% \quad (2)$$

3. Results and Discussion

3.1 Structural description of Cd(II) coordination polymer

The coordination environment of Cd(II) ions is shown in Figure 1 and the selected bond lengths and bond angles for Cd(II) coordination polymer are listed in Table 2. Structural analysis shows that $\{[Cd_2(L)_2(DMF)_2(H_2O)_2]\cdot H_2O\}_n$ crystallizes in monoclinic C_2/c space group, and its asymmetric unit contains two Cd(II) ions, two 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylate ligands, two DMF molecules, two coordinated water molecules and one lattice water molecule. As shown in Figure 1, Cd1 is six-coordinated by four O atoms of four bridging carboxylate groups from 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylate ligands, two O atoms from two DMF molecules and adopts an octahedral coordination geometry, in which two O atoms (O4 and O6A) are at the axial positions, and four O atoms (O8, O9A, O11, and O12) are in the equatorial plane with deviation 0.0324 Å. Cd2 and Cd3 are also six-coordinated by four O atoms of four bridging carboxylate groups from 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylate ligands, two O atoms from two coordinated water molecules and adopt an octahedral coordination geometry (for Cd2: O9 and O2W are at the axial positions, O3, O3A, O9A, and O2WA are in the equatorial plane; for Cd3: O1 and O1A are at the axial positions, O7B, O7C, O2W and O2WA are in the equatorial plane). The Cd(II) ions are interlinked by the bridging carboxylate groups from 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylate ligands to form 1D chained structure as shown in Figure 2. In addition, Figure 3 displays a 3D

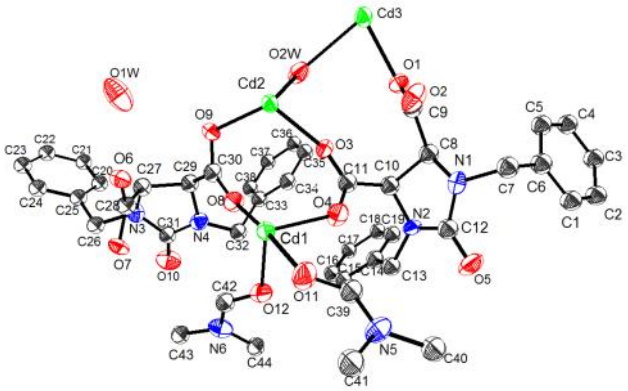

Figure 1. The coordination environment of Cd(II) ion

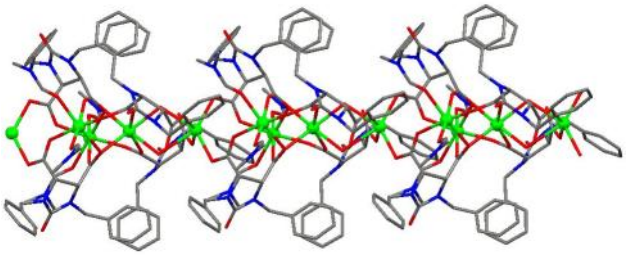

Figure 2. 1D chained structure of Cd(II) coordination polymer by bridging O atoms

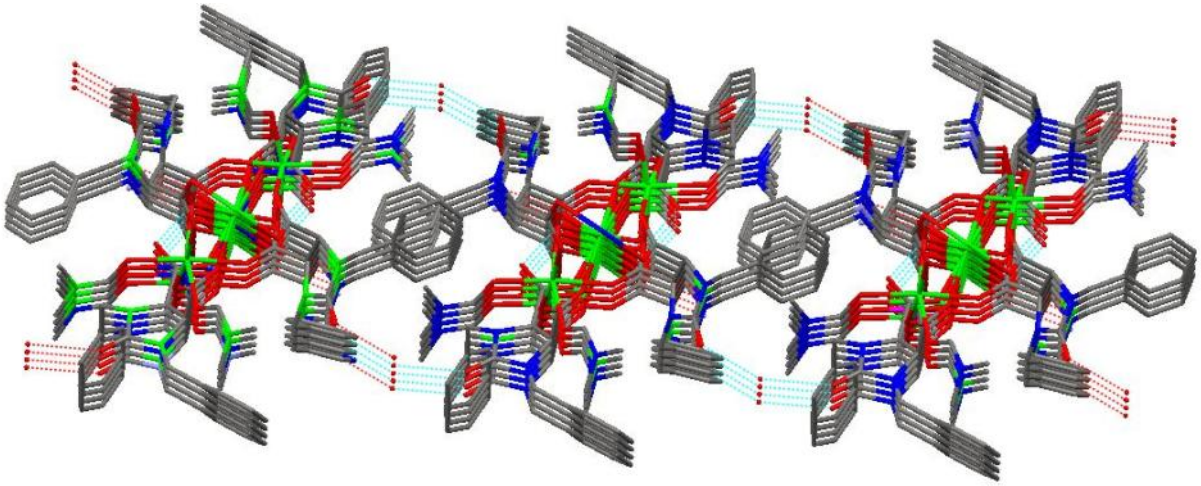

Figure 3. 3D network structure of Cd(II) coordination polymer by hydrogen bonds and π-π stacking

network structure formed by the 1D chain. And the weak interaction of O–H…O hydrogen bonds and π-π stacking play an important role in the stability of the network structure. The hydrogen bonds of Cd(II) coordination polymer are listed in Table 3.

3.2 Catalytic studies of three component coupling reaction

The catalytic activities of Cd(II) coordination polymer catalyst was studied for the synthesis of propargylamines in the A^3 coupling reaction (Figure 4). The conversions of aldehydes were given in Table 4. From the catalytic results, it can be seen that the conversion of benzaldehyde was 90.9%, which shows that the Cd(II) coordination polymer catalyst shows high catalytic activity for the coupling reaction of benzaldehyde, phenylacetylene, and piperidine with 1,4-dioxane as solvent at 120 °C for 12 h. The catalyst features 100 % selectivity to the product of propargylamine for the A^3 coupling reaction without any byproduct. The reusabil-

Table 2. Selected bond lengths (Å) and bond angles (°) for Cd(II) coordination polymer (Symmetry codes: (A) *−x, y, −z+3/2*; (B) *x, y-1, z*; (C) *−x, y-1, −z+3/2*)

Bond	*d*	Angle	(°)
Cd1-O4	2.209(4)	O4-Cd1-O6A	174.27(17)
Cd1-O6A	2.250(4)	O4-Cd1-O11	83.34(16)
Cd1-O11	2.262(4)	O6A-Cd1-O11	92.33(15)
Cd1-O8	2.298(4)	O4-Cd1-O8	96.91(16)
Cd1-O12	2.298(4)	O8-Cd1-O6A	87.81(15)
Cd1-O9A	2.393(4)	O11-Cd1-O8	173.57(15)
Cd2-O3	2.236(4)	O4-Cd1-O12	93.95(16)
Cd2-O3A	2.236(4)	O12 -Cd1-O6A	90.20(15)
Cd2-O9A	2.288(4)	O11 -Cd1-O12	95.50(16)
Cd2-O9	2.288(4)	O8-Cd1-O12	78.06(15)
Cd2-O2W	2.356(4)	O4-Cd1-O9A	92.69(15)
Cd2-O2WA	2.356(4)	O6A-Cd1-O9A	84.26(13)
Cd3-O7B	2.235(4)	O11-Cd1-O9A	99.08(15)
Cd3-O7C	2.235(4)	O8-Cd1-O9A	87.34(14)
Cd3-O1	2.237(4)	O12-Cd1-O9A	164.58(15)
Cd3-O1A	2.237(4)	O3-Cd2-O3A	153.4(2)
Cd3-O2W	2.653(4)	O3-Cd2-O9A	94.58(14)
Cd3-O2WA	2.653(4)	O3A-Cd2-O9A	103.60(14)
		O3-Cd2-O9	103.60(14)
		O3A-Cd2-O9	94.58(14)
		O9-Cd2-O9A	93.8(2)
		O3-Cd2-O2WA	86.53(14)
		O2WA-Cd2-O3A	73.07(14)
		O9A-Cd2-O2WA	94.05(14)
		O9-Cd2-O2WA	166.67(13)
		O3-Cd2-O2W	73.07(14)
		O3A-Cd2-O2W	86.53(14)
		O9A-Cd2-O2W	166.67(13)
		O9-Cd2-O2W	94.05(14)
		O2WA-Cd2-O2W	80.5(2)
		O7B-Cd3-O7C	125.3(2)
		O7B-Cd3-O1	92.01(15)
		O7C-Cd3-O1	99.14(15)
		O7B-Cd3-O1A	99.14(15)
		O7C-Cd3-O1A	92.01(15)
		O1A-Cd3-O1	155.6(2)

ity of Cd(II) coordination polymer catalyst was investigated in the A^3 coupling reaction of benzaldehyde, phenylacetylene, and piperidine in 1,4-dioxane at 120 °C. The result of recyclability of Cd(II) coordination polymer catalyst in A^3 coupling reaction of benzaldehyde, phenylacetylene, and piperidine was listed in Table 5. In four successive cycles, the conversion of benzaldehyde was 90.9%, 72.6%, 52.6%, and 46.1% at 120 °C for 12 h, respectively.

5. Conclusions

In summary, we demonstrated that treatment of $Cd(NO_3)_2 \cdot 4H_2O$ with 1,3-bisbenzyl-2-imidazolidine-4,5-dicarboxylic acid, NaOH and DMF formed a new $\{[Cd_2(L)_2(DMF)_2(H_2O)_2] \cdot H_2O\}_n$ coordination polymer. The catalytic activity of Cd(II) coordination polymer was evaluated for the A^3 coupling reactions of aldehyde, piperidine and phenylacetylene with 1,4-dioxane as solvent. The Cd(II) coordination polymer catalyst shows high catalytic activity for the coupling reaction of benzaldehyde, phenylacetylene, and piperidine.

Table 3. Hydrogen bonds data for Cd(II) coordination polymer

Donor-H…Acceptor	D-H	H…A	D…A	D-H…A	Symmetry transformation
O1W-H1WB…O10	0.96	2.06	2.905(8)	146	x, y, z
O1W-H1WA…O5	0.96	2.25	2.913(8)	125	-1/2+x, 1/2+y, z
O2W-H2WA…O3	0.95	2.29	2.735(6)	108	
O2W-H2WB…O2	0.86	1.74	2.578(6)	165	-x, y, 3/2-z

Table 4. Coupling of aldehyde, alkyne, and amine catalyzed by Cd(II) coordination polymer catalyst in dioxane at 120 °C

Entry	Cat.	R^1	R^2R^3NH	R^4	Time (h)	Conversion (%)
1	Cd(II) complex	Ph	piperidine	Ph	12	90.9
2	Cd(II) complex	4-$CH_3C_6H_4$	piperidine	Ph	12	35.9
3	Cd(II) complex	4-$CH_3OC_6H_4$	piperidine	Ph	12	41.3
4	Cd(II) complex	3-ClC_6H_4	piperidine	Ph	12	50.5

Table 5. Recyclability of Cd(II) coordination polymer catalyst in A^3 coupling reaction of benzaldehyde, phenylacetylene, and piperidine

Run	Solvent	Temperature (°C)	Time (h)	Conversion(%)
1	1,4-dioxane	120	12	90.9%
2	1,4-dioxane	120	12	72.6%
3	1,4-dioxane	120	12	52.6%
4	1,4-dioxane	120	12	46.1%

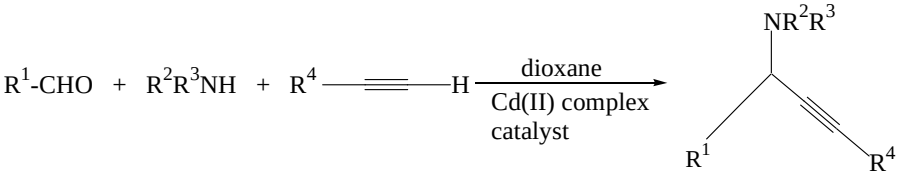

Figure 4. A^3 coupling reaction of aldehyde, alkyne, and amine catalyzed by Cd(II) coordination polymer catalyst

Acknowledgments

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