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Research Article

Synthesis, Crystal Structure, Hirschfeld Surface Analysis and Catalytic Activity of a New Binuclear Zn(II) Complex Based on Homophthalic Acid and 2,2'-Bipyridine Ligands

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Abstract

A new binuclear Zn(II) complex, $[Zn_2L_2(BIPY)_2(H_2O)_2]$ (1) (H_2L = homophthalic acid, BIPY = 2,2'-bipyridine) has been synthesized by one-pot method of homophthalic acid, 2,2'-bipyridine, zinc acetate dihydrate, and NaOH in water/ethanol (v:v = 1:1) solution. The structure of complex (1) was characterized by IR and X-ray single-crystal diffraction analysis. The results show that each Zn(II) ion is five-coordinated with two carboxylic O atoms from two homophthalate ligands (O2, O3 or O2a, O3a), two N atoms from two 2,2'-bipyridine ligands (N1, N2 or N1a, N2a) and one O atom from coordinated water molecule (O5 or O5a), and forms a distorted trigonal bipyramid coordination geometry. Complex (1) forms 1D chained structure and 3D network structure by the π-π interaction of 2,2'-bipyridine ligands. The Hirschfeld surface analysis of complex (1) was calculated. The catalytic performance of complex (1) has also been investigated for the oxidation of benzyl alcohol under O_2 atmosphere. The optimal reaction temperature and pressure were 100 °C and 0.3 MPa for complex (1).

Keywords: Homophthalic acid; Binuclear Zn (II) complex; Synthesis; Structural characterization; Hirschfeld surface analysis; Catalytic activity

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1. Introduction

Zn(II) complexes have been widely studied by chemists during the past decades. Because they display many potential applications in chemosensor [1], antibacterial activity [2,3], magnetic property [4,5], luminescence [6–9], nonlinear optical property [10], anticancer activity [11–13], electrochemical property [14,15], and catalytic activity such as oxidation of benzyl alcohol [16], chemical fixation of CO_2 into cyclic carbonates [17], cyanosilylation of aldehydes [18], decomposition reaction of H_2O_2 [19], A3 coupling reaction [20], cyanosilylation reaction [21]. Some transition metal complexes of homophthalic acid-based ligand have been synthesized and exhibited excellent properties such as magnetic property and fluorescence property [22], photocatalytic degradation [23], and catalytic oxidation [24]. In our previous research work, some Zn(II) complexes have been synthesized, and their structure and properties have also been investigated [25–28]. At the same time, the catalytic activity of some metal complexes has also been investigated [29–34]. To

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further investigate the novel structure and catalytic property of metal complexes, in this paper, a new dinuclear Zn(II) complex, bis[homophthalate-2,2'-bipyridine-aquazinc (II)], has been synthesized by homophthalic acid (Figure 1), 2,2'-bipyridine, NaOH and zinc acetate dihydrate.

2. Materials and Methods

2.1 Materials and Measurements

The materials of homophthalic acid, 2,2'-bipyridine, zinc acetate dihydrate, NaOH were purchased from Jilin Chinese Academy of Sciences-Yanshen Technology Co., Ltd.. IR spectra were carried on a Nicolet AVATAR 360 FTIR spectrophotometer with KBr discs (Nicolet Instrument Inc., Madison, WI, USA) (range 4,000–400 cm^{-1}). The crystal data of complex (**1**) were obtained at 100 K on a SuperNova diffractometer (Bruker, Billerica, MA, USA). Selective oxidation of benzyl alcohol was conducted in a 10 mL stainless-steel high-pressure reactor equipped with magnetic stirring and a temperature controller at 90–110 °C under 0.1 MPa–0.5 MPa O_2 pressure mild conditions.

2.2 Synthesis of Complex (**1**)

An amount of 0.0900 g homophthalic acid (0.5 mmol), 0.0781 g 2,2'-bipyridine (0.5 mmol) and 0.040 g NaOH (1.0 mmol) were added to added to the solution of 20 mL ethanol-water (v:v = 3:2) and stirred at R.T. After the solid was dissolved, 0.1097 g zinc(II) acetate dihydrate (0.5 mmol) solid was added. Then the mixture was stirred and kept at 70 °C for 4 h. After the mixture was cooled and filtered, the colourless crystals of bis[homophthalic acid-2,2'-bipyridine-aquazinc(II)] were received from the filtrate in two weeks with yield 66%.

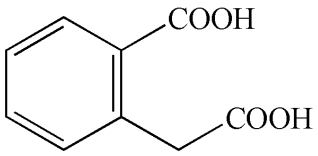

Figure 1. The molecular structure of homophtalic acid.

Table 1. Crystallographic data of complex (**1**).

Parameters	Values
Empirical formula	$C_{19}H_{16}N_2O_5Zn$
Formula weight	417.71
Temperature/K	100
Crystal system	triclinic
Space group	*P*-1
a/Å	8.3155(8)
b/Å	10.6542(11)
c/Å	11.5311(12)
α/°	69.035(10)
β/°	70.958(9)
γ/°	87.008(8)
Volume/Å^3	899.35(17)
Z	2
ρ_{calc}, mg/mm^3	1.542
μ/mm^{-1}	1.399
S	1.042
F(000)	428
Index ranges	$-9 \le h \le 9$, $-12 \le k \le 10$, $-13 \le l \le 12$
Reflections collected	5831
2θ/°	4.104-49.992
Independent reflections	3157 [*R*(int) = 0.0301]
Data/restraints/parameters	3157/162/245
Goodness-of-fit on F^2	1.055
Refinement method	Full-matrix least-squares on F^2
Final *R* indexes [I>=2*σ* (*I*)]	R_1 = 0.0346, wR_2 = 0.0701
Final *R* indexes [all data]	R_1 = 0.0404, wR_2 = 0.0738
Largest diff. peak/hole / e Å^{-3}	0.41/-0.34

2.3 Crystal Structure Determination

A suitable crystal (0.12 mm × 0.11 mm × 0.10 mm) of complex (**1**) was selected to collect data using Olex2 [35] on a SuperNova, Dual (Cu at zero) diffractometer with an Atlas detector at 100(10) K. The structure was solved with the SHELXT [36] structure solution program using Intrinsic Phasing and refined with the SHELXL [37] refinement package. Crystallographic data of complex (**1**) are shown in Table 1. The Hirschfeld surface analysis of complex (**1**) was calculated by the CrystalExplorer software 21.5 [38]. The crystallographic data for the structure reported in this paper has been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC 2207232. The CIF file can be obtained conveniently from the website: https://www.ccdc.cam.ac.uk/structures.

2.4 Selective Oxidation of Benzyl Alcohol

Selective oxidation of benzyl alcohol was conducted in a 10 mL stainless-steel high-pressure reactor equipped with magnetic stirring and a temperature controller at 90–110 °C under 0.1 MPa–0.5 MPa O_2 pressure mild conditions. In a typical experiment, 1.0 mmol (108.1 mg) benzyl alcohol, 15 mg complex (**1**), and 7.0 mL tetrahydrofuran (THF) were mixed in the stainless-steel high-pressure reactor. After the reactor was sealed, the reactor was heated to reaction temperature (90–110 °C) and maintained 2 h. Then the reactor was cooled naturally to room temperature. The mixture was centrifuged to remove the complex (**1**) completely. The remaining solution was analyzed with a gas chromatograph (GC-6890) equipped with SE-54 capillary column and flame ionization detector. The vaporizing chamber and detector temperatures were 250 °C. The GC analysis program was as follows: initial column temperature of 50 °C to 250 °C at 10 °C/min, and held for 10 min.

3. Results and Discussion

3.1 Infrared Spectra

The infrared spectra of homophthalic acid, 2,2'-bipyridine and the complex (**1**) are shown

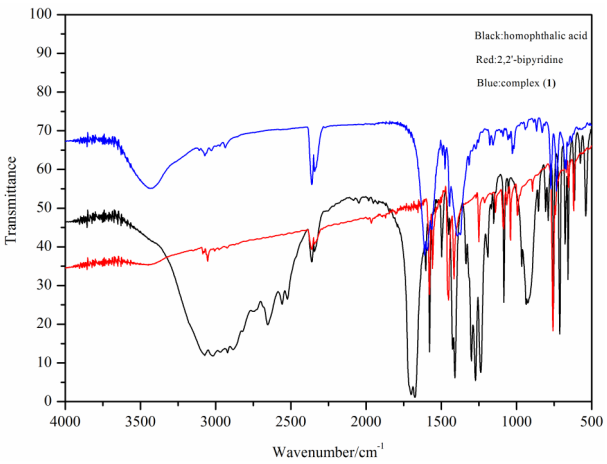

Figure 2. The infrared spectra of homophthalic acid, 2,2'-bipyridine and the complex (**1**).

Table 2. Selected bond lengths (Å) and bond angles (°) for complex (**1**).

Bond	*d*	Angle	(°)
Zn1-O2a	1.9956(17)	O5-Zn1-O2a	92.86(7)
Zn1-O3	1.9778(16)	O2a-Zn1-N1	94.19(8)
Zn1-O5	2.200(2)	O2a-Zn1-N2	129.55(7)
Zn1-N1	2.140(2)	O2a-Zn1-O3	106.49(7)
Zn1-N2	2.075(2)	O5-Zn1-O3	85.71(7)
C9-O1	1.229(3)	O3-Zn1-N1	100.77(7)
C9-O2	1.296(3)	O3-Zn1-N2	123.96(7)
C7-O3	1.295(3)	N1-Zn1-O5	168.64(7)
C7-O4	1.234(3)	O5-Zn1-N2	90.42(8)
		N1-Zn1-N2	78.23(8)
		C9-O2-Zn1a	116.59(16)
		C7-O3-Zn1	117.40(16)
		C15-N1-Zn1	114.18(18)
		C19-N1-Zn1	127.20(18)
		C10-N2-Zn1	125.48(18)
		C14-N2-Zn1	115.54(17)
		O4-C7-O3	124.5(2)
		O1-C9-O2	123.8(2)
		O3-C7-C6	115.6(2)
		O4-C7-C6	119.9(2)

Symmetry transformations: a: 1-*x*, 1-*y*, -*z*.

in Figure 2. The homophthalic acid ligand showed bands at ca. 3000, 1697, 1676, 1577, 1409, 1299, 1272, 1238, 1190, 923, and 613 cm^{-1}. The 2,2'-bipyridine ligand showed bands at ca. 1577, 1416, 1250, 1083, 993, and 758 cm^{-1}. The complex (**1**) showed bands at ca. 3423, 2360, 1591, 1373, and 735 cm^{-1}. Comparing the positions of the ligand and complex absorption peaks, both homophthalic acid and 2,2'-bipyridine coordinated to zinc ions.

3.2 Structural Description of Complex (**1**)

The asymmetrical unit of complex (**1**) is shown in Figure 3. The selected bond lengths (Å) and angles (°) for complex (**1**) are given in Table 2. The 1D chained structure and the 3D network structure of complex (**1**) are given in Figure 4 and Figure 5, respectively. As displayed in Figure 3, the dinuclear Zn(II) complex contains two Zn(II) ions, two homophthalate ligands, two 2,2'-bipyridine ligands and two coordinated water molecules. The carboxylate of deprotonated homophthalic acid ligands adopt monodentate chelate coordination modes. Each Zn(II) ion is five-coordinated with two carboxylic O atoms from two deprotonated homophthalic acid ligands (O2, O3 or O2a, O3a), two N atoms from two 2,2'-bipyridine ligands (N1, N2 or N1a, N2a) and one O atom from coordinated water molecule (O5 or O5a), and forms a distorted trigonal bipyramid coordination geometry. The bond distances of Zn-O and Zn-N are 1.9955(17)Å (Zn1-O2a), 1.9769(15) Å (Zn1-O3), 2.2005(19) Å (Zn1-O5), 2.140(2) Å (Zn1-N1) and 2.075(2) Å (Zn1-N2), respectively, which is consistent with those reported in the literature [39,40]. The dihedral angles of bipyridyl's ring 1 (C1-C2-C3-C4-C5-C6) and ring 2 (C1a-C2a-C3a-C4a-C5a-C6a), ring 3 (N2-C10-C11-C12-C13-C14-C15-C16-C17-C18-C19-N1) and ring 4 (N2a-C10a-C11a-C12a-C13a-C14a-C15a-C16a-C17a-C18a-C19a-N1a) are 0.0°, respectively, showing that ring 1 and ring 2, ring 3 and ring 4 are coplanar. However, the dihedral angle of ring 1 and ring 3 is 70.85°, indicating that the whole molecule is not coplanar. The Zn(II) complex molecules form 1D chained, which then grew into 3D network structure due to the π-π interaction of 2,2'-bipyridine ligands with the distance of 3.356 Å.

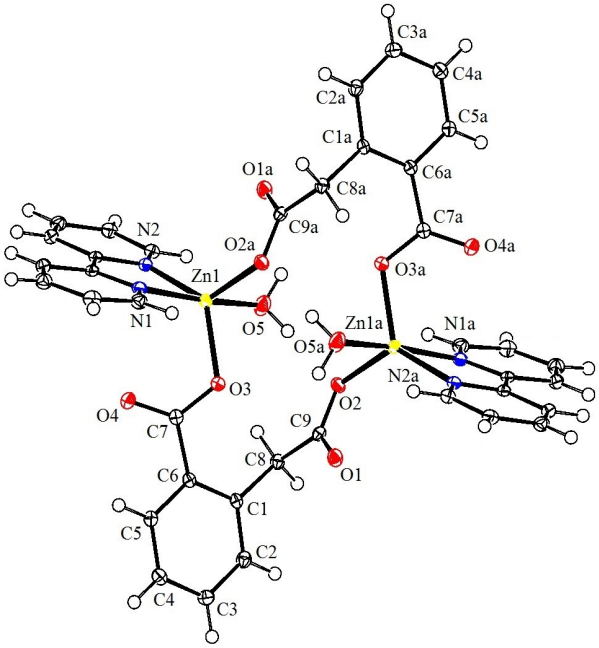

Figure 3. The asymmetrical unit of complex (**1**).

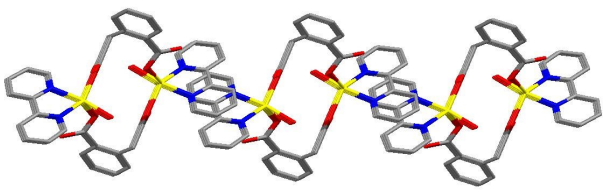

Figure 4. 1D chained structure of complex (**1**).

3.3 The Hirschfeld Surface of the Complex (**1**)

The Hirschfeld surface of the complex (**1**) was analyzed by the CrystalExplorer software 21.5. As shown in Figure 6, the Hirschfeld surfaces mapped over dnorm, di and de of the crystal (a-c), and the two-dimensional (2D) fingerprint plots represented overall and the top three interactions (H···H, C···H/H···C and

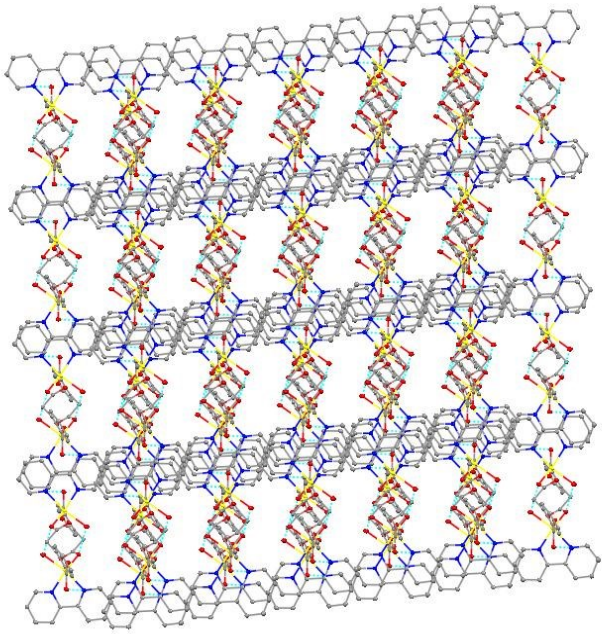

Figure 5. 3D network structure of complex (**1**).

O···H/H···O) are shown in (d-g). Based on the calculations, it can be concluded that the H···H contacts represented the largest contribution (43.5%) to the Hirschfeld surface, followed by C···H/H···C and O···H/H···O contacts with contributions of 22.5% and 18.1%, respectively. It's worth noting that the π-π stacking interactions play a subordinate role in forming the crystal for the C···C contacts with a Hirschfeld surface contribution percentage of 6.7%.

3.4 Catalytic Studies

The catalytic activities of the as-prepared complex (**1**) for benzyl alcohol oxidation were determined with O_2 as a green oxidant using THF as solvent. The results are shown in Table 3. To avoid producing benzoic acid and benzyl benzoate, a series of reactions to obtain optimal reaction conditions to produce benzaldehyde were conducted. The effects of reaction temperature and reaction pressure on benzyl alcohol conversions, benzaldehyde selectivity, and yields of complex (**1**) were studied. The benzyl alcohol conversion (9.3%) and benzaldehyde yield (3.2%) were low for the oxidation of benzyl alcohol at 100 °C within 2 h under 0.3 MPa of O_2. The conversion of benzyl alcohol increased with the increasing of reaction temperature and pressure. However, the selectivity of benzaldehyde decreased with the increasing of reaction temperature and reaction pressure. The optimal reaction temperature and pressure were 100 °C and 0.3 MPa for complex (**1**), respectively. The highest yield (39.0%) of benzaldehyde were obtained at 100 °C under 0.3 MPa of O_2. The yields of benzaldehyde were 50.8% for complex $[Zn_3(L_1)_4(L_2)_2(CH_3COO)_2]$ (HL_1 = 6-phenylpyridine-2-carboxylic acid, L_2 = bis(4-pyridyl)amine) at 90 °C with THF as solvent under 0.5 MPa O_2 within 3 h [16]. The benzyl alcohol conversion and benzaldehyde yield over $ZnL_4(Phen)_2$ (HL=3-bromo-2-hydroxybenzaldehyde-pyridine-2-carbohydrazone) were 37.1% and 1.9% at 90 °C within 4 h under 0.5 MPa of O_2 [41]. Although

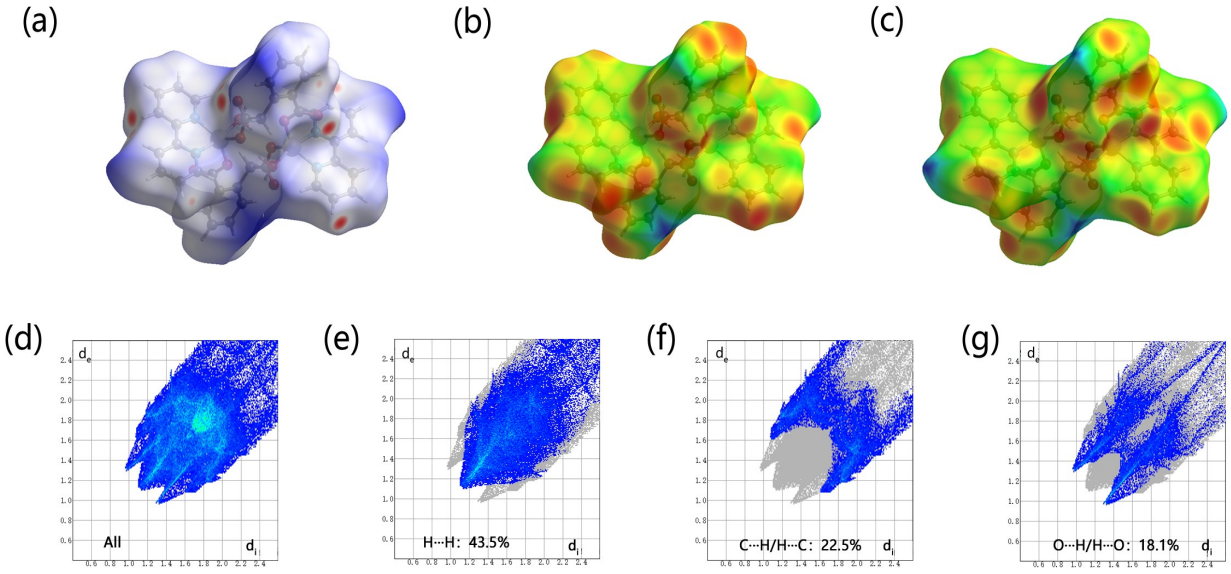

Figure 6. The Hirschfeld surface of the complex (**1**).

Table 3. The benzyl alcohol conversion and benzaldehyde yield for complex (**1**) in the benzyl alcohol oxidation.

Sample	Temperature (°C)	Pressure (MPa)	Conversion (%)	Selectivity (%)	Yield (%)
blank	100	0.3	9.3	34.4	3.2
Complex (**1**)	90	0.3	24.3	100	24.3
Complex (**1**)	100	0.3	64.3	60.7	39.0
Complex (**1**)	110	0.3	98.5	12.4	12.2
Complex (**1**)	100	0.1	36.4	99.5	36.2
Complex (**1**)	100	0.5	82.5	26.1	21.5

Reaction condition: benzyl alcohol 1 mmol, THF 7 mL, complex (**1**) 15 mg, 2h.

the benzaldehyde yield was lower than that of $[Zn_3(L_1)_4(L_2)_2(CH_3COO)_2]$ (50.8%), complex (**1**) catalyst produces higher yields (39.0%) than $ZnL_4(Phen)_2$ (1.9%) catalyst.

4. Conclusions

In summary, a new binuclear Zn(II)-homophthalate complex has been successfully synthesized by one-pot reaction and structurally characterized by IR and X-ray single-crystal diffraction analysis. The Hirschfeld surface analysis of complex (**1**) shows that the H···H contacts represented the largest contribution (43.5%) to the Hirschfeld surface and the π-π stacking interactions play a subordinate role in forming the crystal, whereas selective oxidation of benzyl alcohol with complex (**1**) as catalyst shows that the optimal reaction temperature and pressure were 100 °C and 0.3 MPa.

Acknowledgments

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