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Research Article

# Synthesis, Structural Characterization of a New Ni(II) Complex and Its Catalytic Activity for Oxidation of Benzyl Alcohol

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## Abstract

In ethanol-water (v:v = 1:1), a new Ni(II) complex, $[Ni(L)_2(H_2O)_2]$ (1) (HL = 6-phenylpyridine-2-carboxylic acid) was synthesized using 6-phenylpyridine-2-carboxylic acid, NaOH and $Ni(CH_3COO)_2.4H_2O$. The structure of complex **1** has been determined by elemental analysis and single crystal X-ray diffraction. The single crystal analysis shows that complex 1 contains one Ni(II) ion, two L ligands and two coordinated water molecules. In **1**, the Ni(II) ion is six-coordinated to two O atoms and two N atoms from L ligands and two O atoms from coordinated water molecules, respectively, which form a distorted octahedral coordination geometry. The whole unit of complex **1** is interconnected to each other through intermolecular N−H···O hydrogen bonds involving oxygen atom of coordinated water molecule and the oxygen atoms of L ligand to form 1D molecular architecture. The catalytic activity of complex **1** for oxidation of benzyl alcohol with $O_2$ was investigated. The complex 1 shows good catalytic performance for the oxidation of benzyl alcohol, the benzyl alcohol conversion, benzaldehyde selectivity, and benzaldehyde yield were 49.1%, 92.0%, and 45.2%, respectively, at 90 °C under 0.7 Mpa $O_2$ for 2 h. Moreover, complex **1** could be recovered easily by centrifugation and used repetitively for at least four times.



***Keywords***: 6-Phenylpyridine-2-carboxylic acid; Ni(II) complex; Synthesis; Single crystal structure; Catalytic activity

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## 1. Introduction

As an important member of the coordination chemistry family, the synthesis, novel structure and properties of Ni(II) complex are of chemists' interest [1]. They also exhibit wide applications in anticorrosion performance [2], electrocatalytic properties [3–5], magnetic properties [6–8], photocatalytic properties [9–11], antibacterial activities [12–14], fluorescence properties [15,16], antitumor activities [17,18], catalytic properties [19–22]. Particularly, Ni(II) complex has been used as catalyst in catalyzing the oxidation of benzyl alcohol [23,24]. Mobinikhaledi *et al.* [25] synthesized Y zeolite-encapsulated Ni(II) complexes of the 2-[(2-hydroxyphenylimino)-methyl]-4-(4-chloro-3-nitro-phenylazo)-phenol ligand using the flexible ligand method. They found that it showed good catalytic performance for benzyl alcohol oxidation, the benzyl alcohol conversion and selectivity of benzaldehyde were 52.68% and 100%, respectively. Lin *et al.* [26] reported that $[H_3PMo_8V_6O_{46}][Ni(en)_2]\}.2[Ni(en)_2].5H_2O$ showed high catalytic activity for selective oxidation of benzyl alcohol to benzaldehyde, the

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benzyl alcohol conversion reaches 56.5% and benzaldehyde selectivity reaches 70.1% at the optimal conditions of catalyst concentration, 10 mg/mL, $H_2O_2$/benzyl alcohol, 1.25:1 (v/v), reaction time, 4 h at 120 °C. Nickel(II) riboflavin complex showed excellent yield of benzaldehyde (91% at 70°C for 4 h) for benzyl alcohol oxidation [27]. However, in general, there are relatively few studies on benzyl methanol oxidation as a catalytic catalyst by nickel metal complexes.

The bidentate ligands containing N and O atoms are widely used to construct metal complexes with rich coordination structures [28-30]. 6-Phenylpyridine-2-carboxylic acid and its deprotonated anions, as excellent bidentate ligands, can provide various coordination modes for coordination with metal ions, and our group has synthesized and characterized several metal complexes [31–35]. In this work, as part of our ongoing investigation of the coordination behavior and properties of 6-phenylpyridine-2-carboxylic acid complexes, a new Ni(II) complex, $[Ni(L)_2(H_2O)_2]$ (**1**) (HL = 6-phenylpyridine-2-carboxylic acid) was synthesized using 6-phenylpyridine-2-carboxylic acid, NaOH, and $Ni(CH_3COO)_2.4H_2O$. The structure of complex **1** was determined by elemental analysis and single crystal X-ray diffraction. The catalytic activity of complex **1** as a catalyst for the oxidation of benzyl alcohol with $O_2$ was also investigated.

## 2. Materials and Method

### 2.1 Materials and Measurements

6-Phenylpyridine-2-carboxylic acid (A. R.) was purchased from Jilin Chinese Academy of Sciences-Yanshen Technology Co., Ltd.. $Ni(CH_3COO)_2 \cdot 4H_2O$ (A. R.) and NaOH (A. R.) were purchased from Sinopharm Chemical Reagent Co., Ltd.. The contents of C, H, and N were determined using an Elementar Vario III EL elemental analyzer (Hanau, Germany). Crystal data of complex **1** were obtained with a Bruker Smart CCD diffractometer (Bruker, Billerica, MA, USA). Liquid products were analyzed using a gas chromatography spectrometer (GC-6890, Purkinje General Instrument Co., Ltd., China) equipped with a flame ioniza-

Table 1. The important crystal data and structure refinement for $[Ni(L)_2(H_2O)_2]$ (**1**).

| | |
|---|---|
| Empirical formula | $C_{24}H_{20}N_2NiO_6$ |
| Formula weight | 491.13 |
| Temperature/K | 199.99(10) |
| Crystal size/mm$^3$ | 0.13 × 0.10 × 0.08 |
| Crystal system | monoclinic |
| Space group | $I1_2/a1$ |
| $a$/Å | 13.7381(7) |
| $b$/Å | 17.4278(9) |
| $c$/Å | 19.3220(9) |
| $\alpha$/° | 90 |
| $\beta$/° | 108.785(5) |
| $\gamma$/° | 90 |
| Volume/Å$^3$ | 4379.7(4) |
| $Z$ | 8 |
| $\rho_{calc}$, mg/mm$^3$ | 1.490 |
| $\mu$/mm$^{-1}$ | 0.930 |
| $S$ | 1.011 |
| $F$(000) | 2032 |
| Index ranges | $-14 \le h \le 16$, $-20 \le k \le 16$, $-22 \le l \le 22$ |
| Reflections collected | 9883 |
| $\theta$/° | 2.5650-29.2170 |
| Independent reflections | 3869 [$R$(int) = 0.0271] |
| Data/restraints/parameters | 3869/0/308 |
| Goodness-of-fit on $F^2$ | 1.011 |
| Refinement method | Full-matrix least-squares on $F^2$ |
| Final $R$ indexes [I>=2$\sigma$ ($I$)] | $R_1$ = 0.0325, $wR_2$ = 0.0741 |
| Final $R$ indexes [all data] | $R_1$ = 0.0407, $wR_2$ = 0.0801 |

tion detector (FID) and a SE-54 capillary column to determine conversion and yield.

2.2 Synthesis of $[Ni(L)_2(H_2O)_2]$ (1)

A solution of 248.8 mg $Ni(CH_3COO)_2 \cdot 4H_2O$ (1.0 mmol) in 15 mL ethanol was added a solution of 199.2 mg 6-phenylpyridine-2-carboxylic acid (1.0 mmol) and 40 mg NaOH (1.0 mmol) in 15 mL distilled water with stirring. The above solution was heated at 70 °C for 3 h with stirring, and stirred for 2 h after cooled room temperature. The mother liquid was volatized slowly after the solution was filtered at room temperature. The blue crystals of $[Ni(L)_2(H_2O)_2]$ (**1**) were obtained in 72% yield from after 15 days. Elemental analysis (%) calculated for $C_{24}H_{20}N_2NiO_6$: C, 58.64; H, 4.07; N, 5.70. Found (%): C, 58.39; H, 4.36; N, 5.59.

2.3 Crystal Structure Determination

A blue single crystal of $[Ni(L)_2(H_2O)_2]$ (**1**) (0.13 mm × 0.10 mm × 0.08 mm) was used for X-ray diffraction analysis using a SuperNova, Dual, Cu at zero, AtlasS2 diffractometer with graphite-monochromated Mo-*Ka* radiation ($\lambda$ = 0.71073 Å) at 199.99(10) K. The structure of **1** was solved by direct method (SHELXT 2018/2 [36]) and then refined by full-matrix least squares ( SHELXL 2017/1 [37]) on $F^2$. Absorption corrections were applied by using multi-scan program (Olex2 [38]). The hydrogen atoms were positioned geometrically (C–H = 0.93 Å and O–H = 0.85 Å). Their $U_{iso}$ values were set to 1.2 or 1.5 $U_{eq}$ of the parent atoms. The important crystal data and structure refinement for **1** are listed in Table 1.

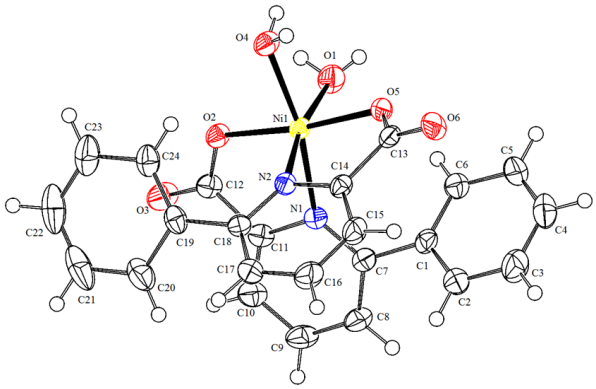


Figure 1. The molecular of $[Ni(L)_2(H_2O)_2]$ (**1**).

2.4 General Procedure for the Oxidation of Benzyl Alcohol

The selective oxidation of benzyl alcohol with $O_2$ as oxidant was performed in a 20 mL stainless-steel high-pressure reactor. In the catalytic test, benzyl alcohol (1 mmol, 108 mg), complex **1** (25 mg), and tetrahydrofuran (THF, 7 mL) were added into the reactor. Then the reactor was sealed, purged with $O_2$ for four times, and then heated to 90 °C. The oxidation reaction was carried out for 2 h under 0.5 MPa $O_2$ pressure. After cooling down to room temperature naturally, the liquid products was collected via centrifugation. The reaction products were analyzed using a gas chromatography (GC-6890, Purkinje General instrument Co., Ltd., Beijing, China) equipped with a flame ionization detector (FID) and a SE-54 capillary column (30 m×0.25 mm×0.25 mm). The reusability of the catalyst was analyzed by separating the complex **1** from the reaction mixture. The complex **1** was dried at 60 °C for 12 h. After each cycle, the complex **1** was collected by similar method and reused for the subsequent cycles.

## 3. Results and Discussion

3.1 Structural Description of $[Ni(L)_2(H_2O)_2]$ (1)

The molecular structure of $[Ni(L)_2(H_2O)_2]$ (**1**) and the coordination environment of Ni(II) are shown in Figure 1. Selected bond lengths and angles of $[Ni(L)_2(H_2O)_2]$ (**1**) are listed in Table 2. The 1D chain of 1 forming by intermolecular hydrogen bonds is shown in Figure 2. The 3D network of **1** forming by intermolecular

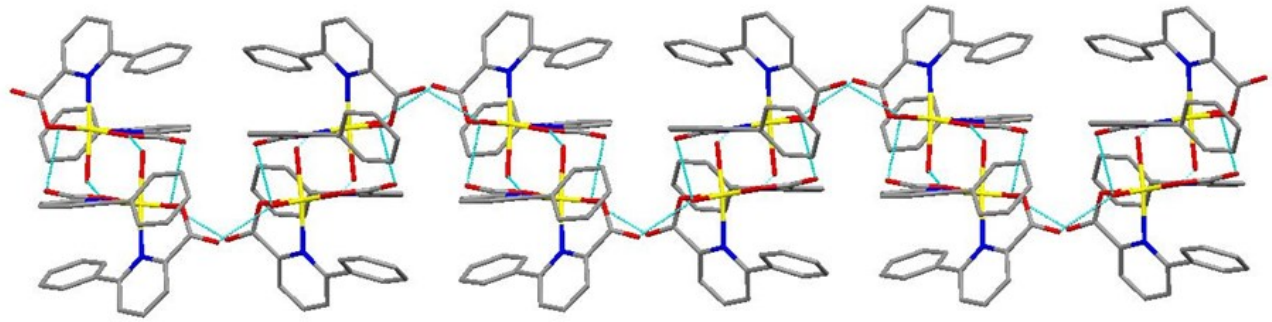

Figure 2. 1D chain structure of $[Ni(L)_2(H_2O)_2]$ (**1**). (The yellow for Ni, blue for N, red for O, and gray for C).

hydrogen bonds and π–π interaction is shown in Figure 3. The hydrogen bond parameters are given in Table 3. Complex **1** contains one Ni(II) ions, two L ligands and two coordinated water molecules. The Ni(II) ion in **1** is six-coordinated by two L ligands connected to Ni(II) in a bidentate chelating mode by two N atoms (N1, N2) and two O atoms (O2, O5) of carboxylate groups, two O atoms (O1, O4) of two coordinated water molecules, respectively. The coordination polyhedron around Ni(II) can be described as a distorted octahedral. The sum of bond angles around Ni(II), O4−Ni1−O1 (89.83(7)°), N2−Ni1−O4 (95.39(7)°), N2−Ni1−N1 (87.11(7)°), O1−Ni1−N1 (89.59(7)°) equals to 361.92° and the bond angle of O5−Ni1−O2 is 172.93(6)°, indicating that the Ni(II) is coplanar plane with O1O4N1N2 atoms and the O2 and O5 atoms are located in the axial position. The bond distances around Ni(II) are 2.0984(19) Å (Ni1−O1), 2.0170(14) Å (Ni1−O2), 2.0767(16) Å (Ni−O4), 2.0015(14) Å (Ni1−O5), 2.1726(19) Å (Ni1−N1), 2.1580(19) Å (Ni1−N2), respectively. As shown in Figure 2, the complex **1** forms 1D chain structure by intermolecular O−H···O hydrogen bonds (Table 3) forming by carboxyl O atoms of L ligands and O atoms of coordinated water molecules. And the 1D chains further form a 3D network structure through intermolecular interactions of O−H···O hydrogen bonds and π–π interactions (Figure 3).

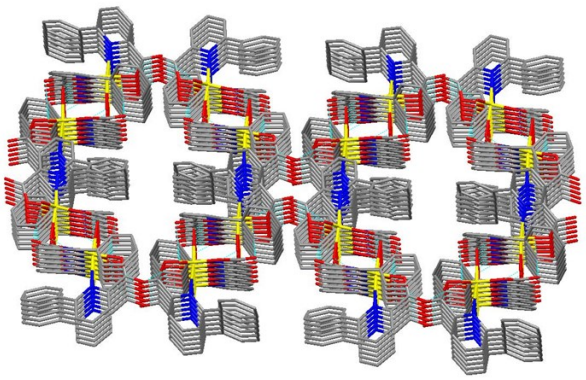

Figure 3. 3D network structure of $[Ni(L)_2(H_2O)_2]$ (**1**). (The yellow for Ni, blue for N, red for O, and gray for C).

### 3.2 Catalytic Studies

The catalytic performance of the complex **1** for benzyl alcohol oxidation were determined with $O_2$ as a green oxidant using THF as solvent. The effect of reaction temperature and reaction pressure were studied to optimize the reaction condition. The results are shown in Table 4. The catalytic activity of the blank (without catalyst) is very low for the benzyl alcohol oxidation at 90 °C within 2 h under 0.5 MPa of $O_2$. As shown in Table 4, the reaction

Table 2. Selected bond lengths and angles of $[Ni(L)_2(H_2O)_2]$ (1).

| Bond | *d* | Angle | (°) |
|---|---|---|---|
| Ni1−O1 | 2.0984(19) | O1−Ni1−N1 | 89.59(7) |
| Ni1−O2 | 2.0170(14) | O1−Ni1−N2 | 169.71(8) |
| Ni1−O4 | 2.0767(16) | O2−Ni1−O1 | 83.98(8) |
| Ni1−O5 | 2.0015(14) | O2−Ni1−O4 | 88.46(6) |
| Ni1−N1 | 2.1726(19) | O2−Ni1−N1 | 79.64(6) |
| Ni1−N2 | 2.1580(19) | O2−Ni1−N2 | 104.97(6) |
| C12−O2 | 1.266(3) | O4−Ni1−O1 | 89.83(7) |
| C12−O3 | 1.239(3) | O4−Ni1−N1 | 168.08(6) |
| C13−O5 | 1.268(3) | O4−Ni1−N2 | 95.39(7) |
| C13−O6 | 1.237(2) | O5−Ni1−O1 | 91.10(8) |
| C7−N1 | 1.352(3) | O5−Ni1−O2 | 172.93(6) |
| C11−N1 | 1.349(3) | O5−Ni1−O4 | 86.45(6) |
| C14−N2 | 1.348(3) | O5−Ni1−N1 | 105.47(7) |
| C18−N2 | 1.357(3) | O5−Ni1−N2 | 80.41(6) |
| | | N2−Ni1−N1 | 87.11(7) |

Table 3. Hydrogen bond geometry of $[Ni(L)_2(H_2O)_2]$ (1).

| D–H···A | d(D–H) (Å) | d(H···A) (Å) | d(D–A) (Å) | ∠DHA (°) |
|---|---|---|---|---|
| O1−H1A···O2$^{i}$ | 0.82 | 2.07 | 2.886(3) | 174 |
| O4−H1A···O6$^{ii}$ | 0.85 | 1.83 | 2.664(2) | 165 |
| O4−H4B···O3$^{i}$ | 0.85 | 2.01 | 2.841(3) | 167 |

Symmetry codes: (i) 3/2-*x*,1/2-*y*,1/2-*z*; (ii) 3/2-*x*, +*y*, 1-*z*.

temperature has a remarkable effect on the benzyl alcohol conversion, benzaldehyde selectivity, and benzaldehyde yield for complex **1**. As the reaction temperature increases, the conversion of benzyl alcohol increases over the complex **1**. The benzyl alcohol conversions were 4.1%, 39.2%, and 66.5% at 80 °C, 90 °C, and 100 °C, respectively. The selectivities of benzaldehyde were 99.8% and 99.2% at 80 °C and 90 °C, respectively. However, the selectivity toward benzaldehyde decreased significantly (46.5%) when the reaction temperature increased to 100 °C. The yield of benzaldehyde were 4.1%, 38.9%, and 30.9% at 80 °C, 90 °C, and 100 °C, respectively. The benzyl alcohol conversion, benzaldehyde selectivity, and yield were also found to be strongly dependent upon the reaction pressure. The benzyl alcohol conversion and benzaldehyde selectivity were 16.1% and 99.7%, 39.2% and 99.2%, and 49.1% and 92.0% at 90 °C under 0.3 MPa, 0.5MPa, and 0.7 MPa, respectively. The conversion of benzyl alcohol increased with increasing of reaction pressure, while the benzaldehyde selectivity decreased with increasing of reaction pressure. The highest yield of benzaldehyde (45.2%) was obtained at 90 °C under 0.7 MPa $O_2$.

The reusability study of complex **1** was carried out on the selective oxidation of benzyl alcohol in THF at 90 °C under 0.5 MPa $O_2$. The complex **1** showed good stability for the oxidation of benzyl alcohol. In four successive cycles, benzyl alcohol conversions were 39.2%, 39.5%, 38.7%, and 38.3% at 90 °C under 0.5 MPa for 2 h. The selectivities and yields toward benzaldehyde were 99.2% and 38.9%, 99.5% and 39.3%, 99.2% and 38.4%, and 99.5% and 38.1% in the first, second , third, and fourth cycles (Table 5). The complex **1** can be easily recycled and used repetitively at least four times with slightly decrease in catalytic activity.

The molecular of $[Ni(L)_2(H_2O)_2]$ (**1**) contains two water molecules, which can be easily removed by heating before catalyzing, and the coordinatively unsaturated nickel was formed which could act as catalytic site of benzyl alcohol oxidation. We speculate that the reaction mechanism of $[Ni(L)_2]$ for the selective oxidation benzyl alcohol to the benzaldehyde is initiated by oxidative dehydrogenation of alcohol taking place on unsaturated nickel [39]. The hydroxyl group of benzyl alcohol first coordinate with unsaturated nickel to obtain the intermediate nickel-alcoholate species. The portion in the hydroxyl group is abstracted to $[Ni(L)_2]$ to form surface adsorbed H species and alkoxide intermediates. Then the alkoxide intermediates undergo a $\beta$-hybride elimination to give the target product benzaldehyde. Meanwhile, nickel-hydride species are reacted with molecular oxygen to give water and to regenerate the catalyst for further reaction.

Table 4. The benzyl alcohol conversion, selectivity, and benzaldehyde yield for complex **1** in the selective oxidation of benzyl alcohol.

| Sample | Temperature (°C) | Pressure (MPa) | Conversion (%) | Selectivity (%) | Yield (%) |
|---|---|---|---|---|---|
| Blank | 90 | 0.5 | 8.5 | 32.1 | 2.7 |
| complex **1** | 80 | 0.5 | 4.1 | 99.8 | 4.1 |
| complex **1** | 90 | 0.5 | 39.2 | 99.2 | 38.9 |
| complex **1** | 100 | 0.5 | 66.5 | 46.5 | 30.9 |
| complex **1** | 90 | 0.3 | 16.1 | 99.7 | 16.1 |
| complex **1** | 90 | 0.7 | 49.1 | 92.0 | 45.2 |

Reaction condition: benzyl alcohol 1 mmol, THF 7 mL, complex 1 25 mg, 2 h

Table 5. The benzyl alcohol conversion, selectivity, and benzaldehyde yield for complex **1** in the selective oxidation of benzyl alcohol at 90 °C under 0.5 MPa $O_2$.

| Entry | Conversion (%) | Selectivity (%) | Yield (%) |
|---|---|---|---|
| Blank | 8.5 | 32.1 | 2.7 |
| Fresh | 39.2 | 99.2 | 38.9 |
| Run 1 | 39.5 | 99.5 | 39.3 |
| Run 2 | 38.7 | 99.2 | 38.4 |
| Run 3 | 38.3 | 99.5 | 38.1 |

Reaction condition: benzyl alcohol 1 mmol, THF 7 mL, complex 1 25 mg, 90 °C, 0.5 Mpa, 2 h

## 4. Conclusions

In summary, we synthesized a new Ni(II) complex based on 6-phenylpyridine-2-carboxylic acid, NaOH and $Ni(CH_3COO)_2.4H_2O$. Single crystal X-ray analysis demonstrates the structure of Ni(II) complex. The crystal packing of **1** shows 3D networks formed by intermolecular interactions of O−H···O hydrogen bonds and π–π interactions. The catalytic activity shows that the complex **1** as catalyst were good stability, and presented excellent selectivity to benzaldehyde, the selectivity of benzaldehyde.

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