

# Synthesis and Characterization of a Metal-Organic Framework Bridged by Long Flexible Ligand

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## Abstract

3D supramolecular network with considerable volume pores was created via hydrogen bond & C-H- $\pi$ . By tightening of The metal-organic frameworks (MOF) namely  $[\text{Ni}(\mu\text{-pmb})_2(\text{H}_2\text{O})_2]_n$  (pmb = 3,5-bis(4-pyridylmethylenoxy) benzoate, that have been synthesized by hydrothermal method. Complex 1 crystallizes in triclinic P-1 space group and consists of 1D semi zigzag chain.

## Keywords

Coordination Polymer, Hydrogen Bond, Metal-Organic Frameworks, Ligand

## 1. Introduction

In recent years, the area of inorganic-organic open framework materials has become one of the intense research activity [1]-[7] not only for the intellectual challenge in controlling and manipulating the self-assembly process but also for their fascinating structural diversities and potential applications in catalysis, molecular adsorption, magnetism, nonlinear optics and molecular sensing [8]-[13]. By closely controlling the properties of the ligands such as the shape [14] [15], functionality [16] [17], flexibility [18] [19], conformation [20] [21], and symmetry [22] [23], frameworks with fantastic structures and desirable properties can be created. Multidentate N or O donor ligands have been extensively employed in the construction of extended structures, such as the flexible ligands 1,2-bis(4pyridyl)ethane (bpe) [24] [25], and its analogues [26] [27], which can adopt the gauche and anti-conformations. Metal-organic frameworks (MOFs) constructed by mixing pyridyl and carboxylate groups are applicable for the reason

that it incorporates the interesting properties of the different functional groups [28]. It has been well recognized that the noncovalent intermolecular forces such as hydrogen bond interactions are reasonably strong [29] [30] and can be used as structural-directing tools in generating many molecular solids with novel properties [31] [32]. Incorporation of both metal-ligand covalent bonds and hydrogen bonds to control the arrangement of the molecule in the space has been considered one of the most rational design strategies. Taking account of all above depicted points, we have employed a long flexible ligand 3,5-bis(4-pyridylmethylenoxyl)benzoate to construct a coordinated infinite framework, formulated as



## 2. Experimental

### 2.1. General

All chemicals purchased were of reagent grade and used without further purification. Elemental analyses (C, H, and N) were performed on a Perkin-Elmer 2400CHN Elemental Analyzer. FT-IR spectra were recorded in the range 400 - 4000  $\text{cm}^{-1}$  on an Alpha Centaur FT/IR Spectrophotometer as KBr pellets. The emission/excitation spectra were recorded on a Varian Cary Eclipse spectrometer. TGA experiments were performed from room temperature to 700°C using a Perkin-Elmer TG-7 analyzer under nitrogen at a heating rate of 10°C/min.

### 2.2. Synthesis of 3,5-PMB

3,5-dihydroxybenzoic acid (15.4 g), ethanol (53 ml), and sulphuric acid (4 ml) were mixed and refluxed for 6 - 7 h. The excess ethanol was removed under vacuum, and then extracted by ethylacetate. The extracted solution was washed by water for one time, and an other time by  $\text{NaHCO}_3$ , after drying by  $\text{Na}_2\text{SO}_4$  the solvent was evaporated. To 20 mmol (3.64 g) of the product, 40 mmol (6.5 g) of 4-pycolyl chloride hydrochloride and 120 mmol (4.8 g) of NaOH and 10 mls DMF were added, the mixture was heated at 70°C for 10 h., then the product was washed by ethanol and filtered. The solid product was transferred to 100 ml round bottom flask, 50 ml of 5% NaOH solution was added and refluxed under 70°C for 5 hours, after transferred into 400 ml beaker, 50 ml of distilled water was added, then concentrated HCL was added drop by drop, at pH = 6, light brown precipitate was obtained, washed by ethanol and filtered. Yield: 72%. Calcd (%) for  $\text{C}_{19}\text{H}_{16}\text{N}_2\text{O}_4$  (336.11): C, 67.85; H, 4.79; N, 8.33%. Found: C, 67.91; H, 4.98; N, 8.2.

### 2.3. Synthesis of $[\text{Ni}(\mu\text{3,5-PMB})_2(\text{H}_2\text{O})_2]_n$ (1)

A mixture of  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (0.030 mmol, 0.072 g), pmb (0.20 mmol, 0.073 g), NaOH (0.6 mmol 0.024 g) and (8 ml) distilled water was heated in sealed Teflon lined steal at 150°C for three days, then cooled to room temperature at a rate of 10°C/h. Green block crystals were obtained. Yield: 54%. Elemental analysis calcd

(%) for  $C_{38}H_{34}N_4O_{14}Ni$  (829.39): C, 55.03; H, 4.13; N, 6.76%. Found: C, 54.98; H, 4.09; N, 6.72.6.

## 2.4. X-Ray Crystallography

Suitable single crystal with approximate dimensions  $0.31 \times 0.26 \times 0.24$  mm (1), was mounted on a glass fiber and used for X-ray diffraction analyses. Data were collected at 293(2) K on a Bruker ApexII CCD diffractometer using the  $\omega$  scan technique with Mo  $K\alpha$  radiation ( $\lambda = 0.71069$  Å). Absorption corrections were applied using the multi-scan technique [33]. The structures were solved by the Direct Method and refined by full-matrix least-square techniques on F2 using SHLXL-97 [34].

## 3. Result and Discussion

Complexes 1 were successfully synthesized hydrothermally. In a typical procedure for synthesizing 1, a mixture of  $NiCl_2 \cdot 6H_2O$ , pmb and NaOH in a ratio of 1:2/3:2/3:2 was used.

### 3.1. Description of Crystal Structure

A summary of crystal data, experimental details and refinement results for compounds 1 - 2 are listed in **Table 1**. Selected bond lengths and angles for 1 - 2 are given in **Table 2**. Complex 1 crystallizes in the triclinic space group P-1 with a formula  $[Ni(\mu\text{-pmb})_2(H_2O)_2]_n$ . The asymmetric unit contains one central Ni atom, two pmb ligands and two water molecules as shown in **Figure 1**. The Ni atom is six-coordinated by two oxygen atoms of different carboxylate groups from two pmb ligands with  $Ni-O(1) = 2.05$  Å and two nitrogen atoms of different pmb in a distance of 2.116 Å and two oxygen atoms of different water molecules ( $Ni-O(3) = 2.094$  Å), to form a distorted octahedral geometry. Adjacent Ni atoms are linked by two pmb ligands to form a neutral 1D zigzag polymeric chain spanning along the a-axis (**Figure 2**). The distance between each two neighboring Ni atoms intersecting the circuit made by two pmb moieties is 11.979 Å. The uncoordinated pyridyl rings are oriented up- or down-side with respect to the Ni atom in the apexes of the zigzag chain, this orientation plays an important role in the packing into a higher network through hydrogen bonding interactions. It should be mentioned that adjacent chains recognize each other through the very short hydrogen bond between the oxygen atom of water molecule in the chain and the nitrogen of pyridyl ring in the adjacent chain (bond length is 2.77 Å and oxygen atoms of different carboxylate groups from two pmb ligands with  $Ni-O(1) = 2.05$  Å and two nitrogen atoms of different pmb in a distance of 2.116 Å and two oxygen atoms of different water molecules ( $Ni-O(3) = 2.094$  Å), to form a distorted octahedral geometry. Adjacent Ni atoms are linked by two pmb ligands to form a neutral 1D zigzag polymeric chain spanning along the a-axis (**Figure 2**). The distance between each two neighboring Ni atoms intersecting the circuit made by two pmb moieties is 11.979 Å. The uncoordinated

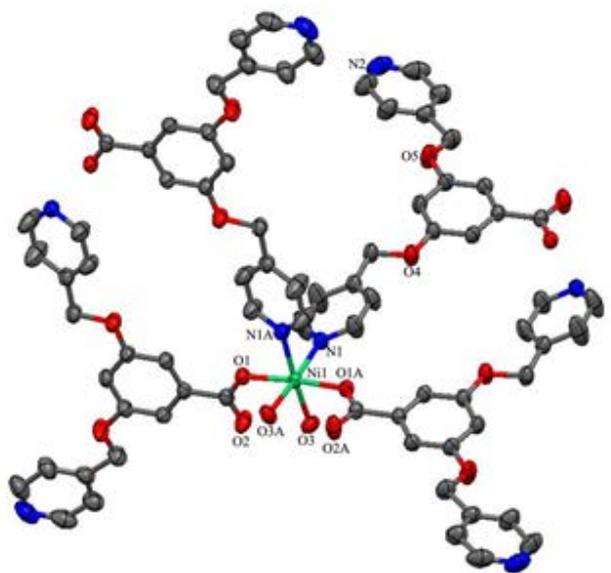
**Table 1.** Crystallographic data for complexes 1.

| Parameter   | <b>1</b>  |
|---|---|
| <i>Empirical formula</i>  | C <sub>38</sub> H <sub>34</sub> N <sub>4</sub> O <sub>14</sub> Ni |
| <i>T (K)</i>  | 293(2)  |
| <i>Wavelength (Å)</i>   | 0.71069   |
| <i>Crystal system</i>   | Triclinic   |
| <i>Space group</i>  | P-1   |
| <i>a (Å)</i>  | 18.796(5)   |
| <i>b (Å)</i>  | 10.121(5)   |
| <i>c (Å)</i>  | 20.513(5)   |
| <i>α</i>  | 90.000(5) deg.  |
| <i>β</i>  | 98.312(5) deg.  |
| <i>γ</i>  | 90.000(5) deg.  |
| <i>V (Å<sup>3</sup>)</i>  | 3861(2)   |
| <i>Z</i>  | 4   |
| <i>D<sub>calc</sub> (mg·m<sup>-3</sup>)</i>                             | 1.427   |
| <i>Absorption coefficient (mm<sup>-1</sup>)</i>                         | 0.576   |
| <i>F(000)</i>   | 1720  |
| <i>Crystal size (mm)</i>  | 0.31 × 0.26 × 0.24  |
| <i>θ (Range of data collection)</i>                                     | 2.01 to 28.27 deg.  |
| <i>Limiting indices</i>   | -22 ≤ h ≤ 24,<br>-13 ≤ k ≤ 11,<br>-26 ≤ l ≤ 26                    |
| <i>Data [1 &gt; 2σ(1)]</i>  | 4667/0/266  |
| <i>Goodnes of fit on F<sup>2</sup></i>                                  | 0.943   |
| <i>R<sub>1</sub>, wR<sub>2</sub> indices (1 &gt; 2σ(1))<sup>a</sup></i> | R <sub>1</sub> = 0.0517, wR <sub>2</sub> = 0.1396                 |
| <i>R<sub>1</sub>, wR<sub>2</sub> (all data)</i>                         | R <sub>1</sub> = 0.0747, wR <sub>2</sub> = 0.1544                 |

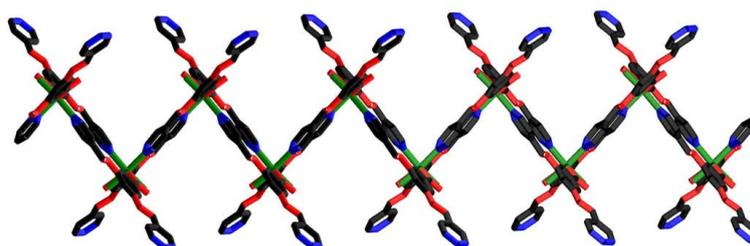
**Table 2.** Selected bond length (Å) and angles (°) for complexes 1.

| <b>1</b>            |            |                     |            |
|---------------------|------------|---------------------|------------|
| Ni(1)-O(1)          | 2.0511(17) | Ni(1)-O(1)#1        | 2.0511(17) |
| Ni(1)-O(3)#1        | 2.093(2)   | Ni(1)-O(3)          | 2.093(2)   |
| Ni(1)-N(1)#1        | 2.115(2)   | Ni(1)-N(1)          | 2.115(2)   |
| O(1)-Ni(1)-O(1)#1   | 175.25(9)  | O(1)-Ni(1)-O(3)#1   | 87.36(8)   |
| O(1)#1-Ni(1)-O(3)#1 | 89.48(8)   | O(1)-Ni(1)-O(3)     | 89.48(8)   |
| O(1)#1-Ni(1)-O(3)   | 87.36(8)   | O(3)#1-Ni(1)-O(3)   | 96.47(13)  |
| O(1)-Ni(1)-N(1)#1   | 90.81(7)   | O(1)#1-Ni(1)-N(1)#1 | 92.66(8)   |
| O(3)#1-Ni(1)-N(1)#1 | 88.76(10)  | O(3)-Ni(1)-N(1)#1   | 174.77(9)  |
| O(1)-Ni(1)-N(1)     | 92.66(8)   | O(1)#1-Ni(1)-N(1)   | 90.81(7)   |
| O(3)#1-Ni(1)-N(1)   | 174.77(9)  | O(3)-Ni(1)-N(1)     | 88.76(10)  |
| N(1)#1-Ni(1)-N(1)   | 86.02(13)  |                     |            |

Symmetry codes: for **1** #1 -x + 1, y, -z + 3/2; #2 x, -y + 1, z - 1/2; #3 x, -y + 1, z + 1/2; for **2** #1 -x + 1, y, -z + 3/2; #2 -x + 1, -y, -z + 2; #3 x, y - 1, z, #4 x, y + 1, z.



**Figure 1.** ORTEP view of 1 showing the coordination environment of the Ni center (50% probability level).



**Figure 2.** The neutral 1D zigzag polymeric chain spanning along the a-axis in 1.

pyridyl rings are oriented up- or down-side with respect to the Ni atom in the apexes of the zigzag chain, this orientation plays an important role in the packing into a higher network through hydrogen bonding interactions. It should be mentioned that adjacent chains recognize each other through the very short hydrogen bond between the oxygen atom of water molecule in the chain and the nitrogen of pyridyl ring in the adjacent chain (bond length is 2.77 Å and N–H...O angle is 152.67°) (**Figure 3(a)**), and C–H... $\pi$  interaction between the phenyl ring and the hydrogen of pyridyl ring. These interactions are of lengths vary between 3.2 - 3.6 Å. The angle between the coordinated water molecules (O(3)–Ni–O(3)) is 96.47°, so the extension via hydrogen bond interactions occurs in the b and c-axis converting the 1D zigzag chain into a three dimensional network with rectangular grid of dimensions = 12.06  $\times$  17.85 Å parallel to the ab-plane (**Figure 3(b)**). It is interesting to note that despite the presence of such large sized voids no interpenetration is observed.

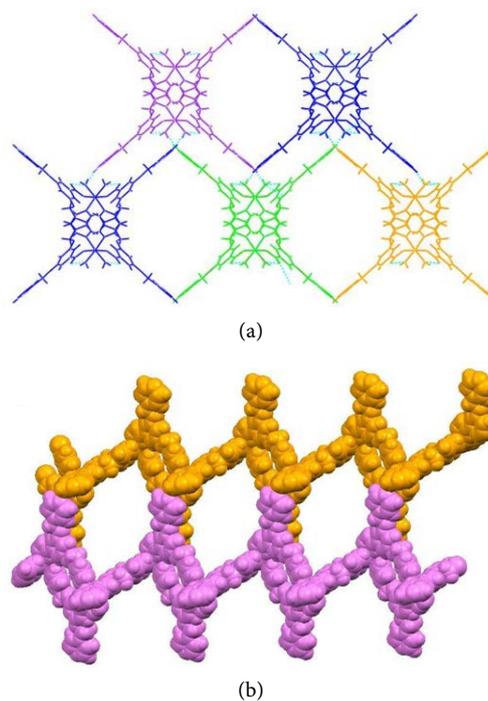
### 3.2. IR Spectra

The IR spectra of complex 1 show the absorption bands of the skeletal vibrations of the aromatic rings appear in the 1616 - 1382  $\text{cm}^{-1}$  region and strong broa-

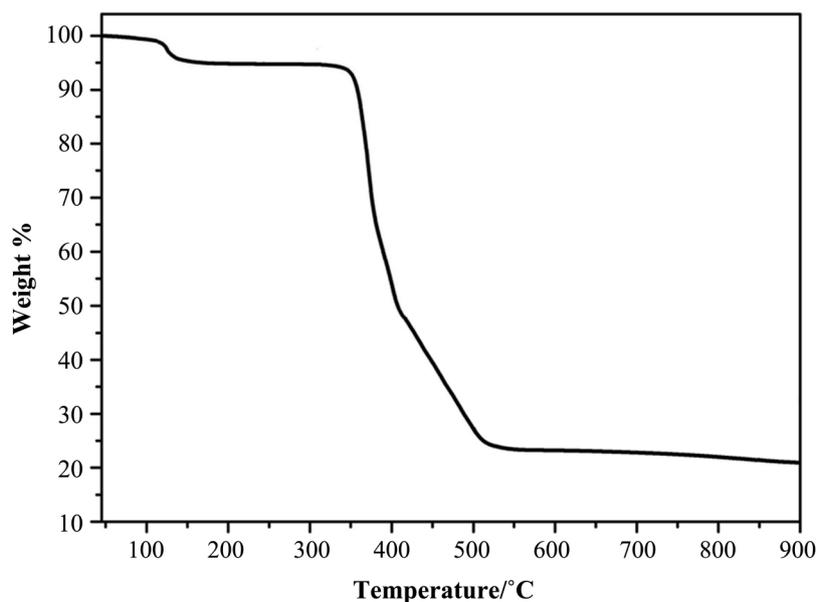
dened band at  $3414\text{ cm}^{-1}$  due to the presence of water ligands [35].

### 3.3. Compositional Stability of the New MOFs

To estimate the stability of the supramolecular architecture, thermogravimetric analyses experiments of complex 1 was carried out in the temperature range of  $50^\circ\text{C} - 900^\circ\text{C}$ . As shown in (Figure 4), the framework of complex 1 is stable up



**Figure 3.** (a) The extension in b and c direction via hydrogen bond shown as blue dots. (b) Rectangular void generated by hydrogen bonding and C–H... $\pi$  interactions.



**Figure 4.** TGA curve for comp.

to 358°C. A rapid weight loss can be detected from 358°C to 510°C. That is attributed to the complete decomposition of the organic ligands.

#### 4. Conclusion

In conclusion, novel MOF has been successfully synthesized and fully structurally characterized by using the flexible 3,5-bis(4-pyridylmethylenoxy)benzoate ligand and incorporating binary aromatic acid as co-ligands under the same hydrothermal conditions. Complex 1 exhibits 1D zigzag chain converted via hydrogen bond to 3D supramolecular framework with locked rectangle.

#### Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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