

苯二甲酸衍生物及含氮配体构筑的两个镍配位聚合物的合成、晶体结构与热稳定性研究

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摘要: 使用硝酸镍分别与 2 个含取代基的苯基二羧酸 3-硝基邻苯二甲酸(3-NPAH₂)和 5-溴间苯二甲酸(H₂BIPA)以及 2 个刚性含氮配体 4,4'-联吡啶(4,4'-bipy)、1,4'-二咪唑基二苯(L=1,4'-bis(imidazol-yl)benzene)在水热条件下合成了 2 个镍配位聚合物, 即, [Ni(3-NPA)(4,4'-bipy)(H₂O)]_n (**1**)和[Ni(BIPA)(L)]_n (**2**)。表征了 2 个化合物的红外光谱、元素分析、热重、X-射线单晶衍射以及 X-射线粉末衍射。化合物 **1** 是三斜晶系, $P\bar{1}$ 空间群, 含有[Ni(OCO)₄Ni]双核单元, 该双核单元再通过 4,4'-联吡啶连接而成一个二维单层结构。层与层之间通过羧酸氧原子与配位水分子之间产生氢键作用。化合物 **2** 是单斜晶系, $P2_1/c$ 空间群, 含有[Ni₂(COO)₂]双核单元, 该双核单元进一步通过 BIPA²⁻构成了一个双链结构, 该双链再通过 1,4'-二咪唑基二苯桥联配体连接形成了一个三维框架结构。此外, 热重分析结果显示化合物 **2** 具有较高的热稳定性。

关键词: 金属有机框架; 晶体结构; 刚性配体; 热稳定性

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Syntheses, Crystal Structures, Thermal Stabilities of Nickel(II) Coordination Polymers Constructed from the Substitutional Benzenedicarboxylic Acid and Auxiliary N-Donor Ligands

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Abstract: Two nickel(II) coordination polymers based on two substitutional benzenedicarboxylic acid ligands (3-NPAH₂=3-nitrophthalic acid, H₂BIPA=5-bromoisophthalic acid) and two different N-donor ligands (4,4'-bipy=4,4'-bipyridine, L=1,4'-bis(imidazol-yl)benzene) have been hydrothermally synthesized, namely, [Ni(3-NPA)(4,4'-bipy)(H₂O)]_n (**1**) and [Ni(BIPA)(L)]_n (**2**). The two compounds were characterized by IR spectrum, elemental analysis, powder X-ray diffraction (PXRD) and single-crystal X-ray diffraction, and thermal stabilities. Compound **1** crystallizes in the triclinic space group $P\bar{1}$. Compound **1** features a two-dimensional (2D) single layer structure based on a dimer unit [Ni(OCO)₄Ni] through four carboxylate groups from two 3-NPA²⁻ ligands, which is further linked by the 4,4'-bipy molecules. Hydrogen bond interactions are found among the carboxylate oxygen atoms and the coordinated water between the layers. Compound **2** crystallizes in a monoclinic space group $P2_1/c$. Compound **2** displays a three-dimensional (3D) framework structure based on the double chain structure containing the dimer [Ni₂(COO)₂] unit, which is further extended into the 3D framework structure by the L molecules. Furthermore, the thermal stabilities properties of compounds **1** and **2** were also investigated in detail. Compound **2** shows relatively high thermal stability property. CCDC: 986627, **1**; CCDC: 986628, **2**.

Key words: Metal-organic frameworks (MOFs); crystal structure; rigid ligand; thermal stability

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0 Introduction

During the last couple of decades, coordination polymers (CPs) or metal-organic-frameworks (MOFs) have been attracted considerable attention not only due to their potential applications as a class of functional materials in many fields, such as gas adsorption and storage, catalysis, chemical sensors, luminescence, magnetism but also for their intriguing structural diversities and fascinating topologies^[1-10]. MOFs can be constructed by metal ions or cluster and organic ligands, which be controlled by many factors, including reaction temperature, pH, metal/ligand stoichiometry, as well as the type of counterion used during their preparation process. The metal ions used for the construction of these inorganic-organic materials have been considerable attention in relation to their different coordinated geometries, such as transition metal ions, rare earth metal ions, and alkaline-earth metal ions. In numerous organic ligands, aromatic multicarboxylate have been extensively used to construct these materials owing to their various coordination modes^[11-14]. Among these ligands, aromatic multicarboxylate ligands containing substituents (halogen, nitro group) are rather limited in numbers at this stage^[15-19]. As a class of simple bidentate ligands, a variety of N-donor ligands have been used for the construction of MOFs, such as 4,4'-bipy, 1,4'-bis(imidazol-yl)benzene and its isomers, which have been prepared through the formation of connections between metal coordination sites and a linker^[20-21]. On the basis of above factors and as an extension of our previous work, herein we introduce two aromatic multicarboxylate ligands containing substituents and two N-donor ligands to construct two new nickel(II) ligated coordination polymers. We report the syntheses, crystal structures and thermal stabilities properties of two coordination polymers, namely, $[\text{Ni}(\text{3-NPA})(4,4'\text{-bipy})(\text{H}_2\text{O})]_n$ (**1**) and $[\text{Ni}(\text{BIPA})(\text{L})]_n$ (**2**).

1 Experimental

1.1 Materials and methods

The ligand 1,4'-bis(imidazol-yl)benzene (L) was

synthesized according to the literature^[22]. All other starting reagents were of analytical grade and purchased from commercial sources without further purification. Elemental analyses for C, H, and N were performed on a Perkin-Elmer 240C analyzer. Infrared spectroscopy of a KBr pellet was recorded in the 400~4 000 cm^{-1} range with a Thermo Scientific Nicolet iS10 FT-IR spectrometer. Thermal analyses were performed on a Perkin-Elmer Pyris 1 instrument in the range of 30~800 $^{\circ}\text{C}$ at a heating rate of 10 $^{\circ}\text{C} \cdot \text{min}^{-1}$ under nitrogen flow. The powder X-ray diffraction (PXRD) data were collected on a Rigaku Ultima IV X-ray diffractometer with Cu $K\alpha$ radiation ($\lambda = 0.154\ 056\ \text{nm}$).

1.2 Synthesis of $[\text{Ni}(\text{3-NPA})(4,4'\text{-bipy})(\text{H}_2\text{O})]_n$ (**1**)

A mixture of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.1 mmol, 29.0 mg), 3-NPAH₂ (0.1 mmol, 21.0 mg), 4,4'-bipy (0.1 mmol, 15.6 mg), NaOH (0.1 mmol, 4.0 mg), and H₂O (10 mL) was stirred and then sealed in a 30 mL Teflon-lined autoclave, which was heated at 140 $^{\circ}\text{C}$ for 3 days. After slowly cooling to room temperature, aquamarine block-like crystals (45% yield based on Ni) were collected after washing with H₂O and drying in air. Anal. Calcd. for $\text{C}_{18}\text{H}_{13}\text{N}_3\text{NiO}_7$ (%): C 48.92, H 2.94, N 9.51; Found(%): C 48.78, H 3.01, N 9.47. IR spectrum (cm^{-1}): 3 448 (s), 1 608 (vs), 1 555 (s), 1 524 (s), 1 464 (m), 1 420 (s), 1 385 (vs), 1 347 (m), 1 294 (w), 1 223 (w), 1 130 (w), 1 070 (m), 1 045 (w), 928 (m), 819 (m), 792 (m), 756 (m), 717 (m), 688 (m), 637 (m).

1.3 Synthesis of $[\text{Ni}(\text{BIPA})(\text{L})]_n$ (**2**)

A mixture of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.1 mmol, 29.0 mg), H₂BIPA (0.1 mmol, 25.0 mg), L (0.1 mmol, 21.0 mg), NaOH (0.3 mmol, 12.0 mg), and H₂O (10 mL) was stirred and then sealed in a 30 mL Teflon-lined autoclave, which was heated at 160 $^{\circ}\text{C}$ for 3 days. After slowly cooling to room temperature, bright green block-like crystals (40% yield based on Ni) were collected after washing with H₂O and drying in air. Anal. Calcd. for $\text{C}_{20}\text{H}_{13}\text{BrN}_4\text{NiO}_4$ (%): C 46.92, H 2.54, N 10.94; Found (%): C 46.81, H 2.61, N 11.01. IR spectrum (cm^{-1}): 3 448 (m), 3 122 (m), 1 625 (vs), 1 539 (vs), 1 448 (s), 1 368 (s), 1 310 (m), 1 264 (m), 1 235 (m), 1 138 (w), 1 104 (w), 1 059 (m), 961 (s), 935 (m), 835

(m), 782 (m), 732 (s), 713 (s), 658 (m), 649 (m), 556 (w).

1.4 Crystallographic studies

Crystallographic data collections for compounds **1** and **2** were carried out on a Bruker SMART APEX CCD diffractometer using graphite-monochromated Mo $K\alpha$ ($\lambda=0.071\ 073\ \text{nm}$) radiation at 296 K. The data were collected in the θ range 2.15° to 26.00° for **1**, 2.44° to 25.99° for **2**, using a narrow-frame method with scan widths of 0.30° in ω and an exposure time of 10 s per frame. The data were integrated by using the SAINT program^[23], which also used for the intensity corrections for Lorentz and polarization effects. Absorptions corrections were applied. The structures were solved by direct methods using the program

SHELXS-97^[24] and all non-hydrogen atoms were refined anisotropically on F^2 by the full-matrix least-squares technique using the program SHELXL-97^[25] crystallographic software package. The hydrogen atoms of water molecules were found from the Fourier maps. All hydrogen atoms were refined isotropically with the isotropic vibration parameters related to the non-hydrogen atoms to which they bonded. The details of the crystal parameters, data collection and refinement for the compounds **1** and **2** are listed in Table 1. Selected bond lengths and angles are given in Tables 2 and 3.

CCDC: 986627, **1**; CCDC: 986628, **2**.

Table 1 Crystal data and structure refinement for **1** and **2**

	1	2
Formula	$\text{C}_{18}\text{H}_{13}\text{N}_3\text{NiO}_7$	$\text{C}_{20}\text{H}_{13}\text{BrN}_4\text{NiO}_4$
Formula weight	442.02	511.96
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/c$
a / nm	0.859 4(3)	1.265 4(2)
b / nm	1.092 0(4)	1.017 32(16)
c / nm	1.146 4(7)	1.584 3(3)
$\alpha / (^\circ)$	105.994(9)	90
$\beta / (^\circ)$	108.043(9)	112.389(2)
$\gamma / (^\circ)$	107.775(6)	90
V / nm^3	0.889 0(7)	1.885 8(5)
Z	2	4
$D_c / (\text{g} \cdot \text{cm}^{-3})$	1.651	1.803
μ / mm^{-1}	1.141	3.185
$F(000)$	452	1 024
GOF on F^2	1.013	1.040
$R_1, wR_2^a [I > 2\sigma(I)]$	0.046 8, 0.136 8	0.024 3, 0.061 6
R_1, wR_2^a (all data)	0.055 8, 0.145 0	0.028 1, 0.062 9
$(\Delta\rho)_{\text{max}}, (\Delta\rho)_{\text{min}} / (\text{e} \cdot \text{nm}^{-3})$	788, -488	533, -731

$$^a R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|; wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)]^{1/2}$$

Table 2 Selected bond lengths (nm) angles ($^\circ$) for **1**^a

Ni(1)-N(2)	0.208 2(3)	Ni(1)-O(3)C	0.203 6(3)	C(8)-O(3)	0.125 2(5)
Ni(1)-N(3)	0.207 1(3)	Ni(1)-O(1W)	0.205 2(3)	C(8)-O(4)	0.123 5(5)
Ni(1)-O(1)	0.215 3(3)	C(7)-O(1)	0.127 8(4)		
Ni(1)-O(2)	0.213 2(3)	C(7)-O(2)	0.123 5(4)		
O(1W)-Ni(1)-O(2)	85.15(11)	O(1W)-Ni(1)-N(2)	93.94(12)	O(3)C-Ni(1)-O(1)	165.65(10)
O(3)C-Ni(1)-O(1W)	92.73(11)	N(3)-Ni(1)-N(2)	96.43(13)	O(1W)-Ni(1)-O(1)	88.41(12)

Continued Table 2

O(3)C-Ni(1)-N(3)	86.46(12)	O(3)C-Ni(1)-O(2)	104.64(10)	N(3)-Ni(1)-O(1)	89.86(12)
O(1W)-Ni(1)-N(3)	169.63(11)	N(3)-Ni(1)-O(2)	85.06(12)	N(2)-Ni(1)-O(1)	99.69(11)
O(3)C-Ni(1)-N(2)	94.50(12)	N(2)-Ni(1)-O(2)	160.86(11)	O(2)-Ni(1)-O(1)	61.19(9)

^aSymmetry transformation used to generate equivalent atoms: A: $-x+2, -y+2, -z$; B: $-x, -y+1, -z$; C: $-x+1, -y+2, -z+1$.

Table 3 Selected bond lengths (nm) angles ($^{\circ}$) for **2**^a

Ni(1)-N(1)	0.208 45(17)	Ni(1)-O(2)	0.210 10(14)	Ni(1)-O(3)A	0.205 60(14)
Ni(1)-N(3)	0.205 23(18)	Ni(1)-O(1)D	0.201 63(14)	Ni(1)-O(4)A	0.236 20(15)
O(1)D-Ni(1)-N(3)	109.76(7)	O(3)A-Ni(1)-N(1)	91.32(6)	O(1)D-Ni(1)-O(4)A	99.96(6)
O(1)D-Ni(1)-O(3)A	159.34(6)	O(1)D-Ni(1)-O(2)	89.49(6)	N(3)-Ni(1)-O(4)A	150.06(6)
N(3)-Ni(1)-O(3)A	90.72(6)	N(3)-Ni(1)-O(2)	93.78(6)	O(3)A-Ni(1)-O(4)A	59.44(5)
O(1)D-Ni(1)-N(1)	84.50(6)	O(3)A-Ni(1)-O(2)	91.83(6)	N(1)-Ni(1)-O(4)A	84.16(6)
N(3)-Ni(1)-N(1)	94.95(7)	N(1)-Ni(1)-O(2)	170.68(6)	O(2)-Ni(1)-O(4)A	89.90(5)

^aSymmetry transformation used to generate equivalent atoms: A: $-x+1, -y+2, -z+1$; B: $-x, -y+1, -z$; C: $-x+2, -y+1, -z+1$;

D: $-x+1, -y+1, -z+1$.

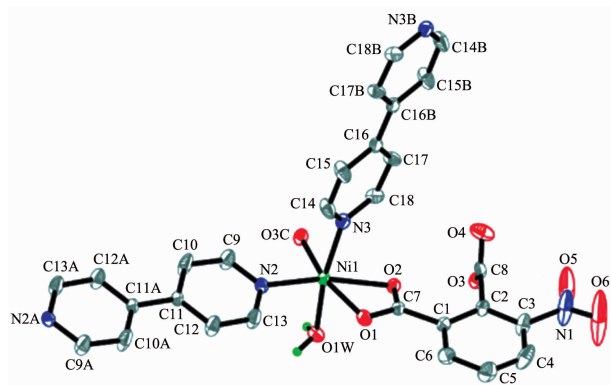
2 Results and discussion

2.1 Crystal and molecules structures

2.1.1 Crystal structure description of

$[\text{Ni}(\text{3-NPA})(4,4'\text{-bipy})(\text{H}_2\text{O})]_n$ (**1**)

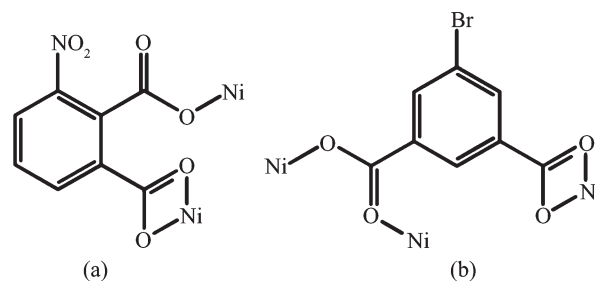
X-ray crystallography determination reveals that **1** crystallizes in the triclinic space group $P\bar{1}$. As shown in Fig.1, the asymmetric unit consists of one Ni(II) ion, one 3-NPA²⁻, one 4,4'-bipy molecule, and one coordination water molecule. The Ni1 center ion is six-coordinated with distorted octahedral coordination geometry, the six positions are occupied by three carboxylate oxygen atoms (O1, O2, O3C) from two equivalent 3-NPA²⁻ ligands and two nitrogen atoms



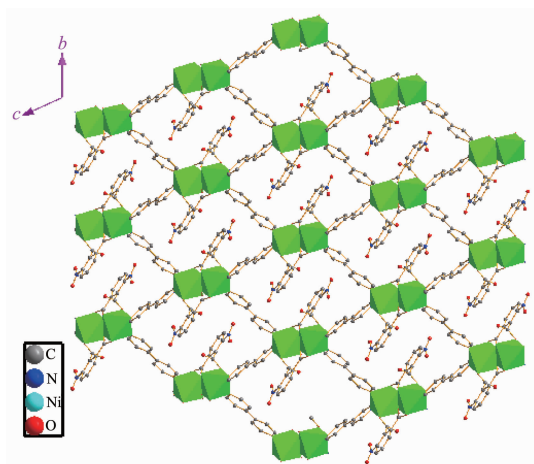
All hydrogen atoms are omitted for clarity; Symmetry codes: A: $-x+2, -y+2, -z$; B: $-x, -y+1, -z$; C: $-x+1, -y+2, -z+1$

Fig.1 ORTEP drawing of the asymmetric unit of **1** with 30% probability of thermal ellipsoids

(N2, N3) from two 4,4'-bipy molecules as well as one water molecule (O1W). The Ni-N/O bond lengths are in the range of 0.203 6(3)~0.215 3(3) nm. The 3-NPA²⁻ acts as bidentate ligand by using its three carboxylate oxygen atoms to link to two Ni(II) ions (Scheme 1a). A dimer unit $[\text{Ni}(\text{OCO})_4\text{Ni}]$ is formed by two Ni(II) ions bridged by four carboxylate groups from two 3-NPA²⁻ ligands. The Ni \cdots Ni distance is 0.518 9(2) nm in the unit. The dimer units are further connected by the 4,4'-bipy molecules to form a single layer framework structure along two different directions (Fig.2). The layer can be viewed as a regular parallelogram topology in which the dimer units are linked through the 4,4'-bipy bridges. The size of the side lengths can be estimated from the distance of the dimer units, thus affording a quadrangular grid with the large window of $1.171 \times 1.732 \text{ nm}^2$. The hydrogen bond interactions are found between the coordination water



Scheme 1 Coordination modes of the 3-NPA²⁻ and BIPA²⁻ ligands in **1** and **2**

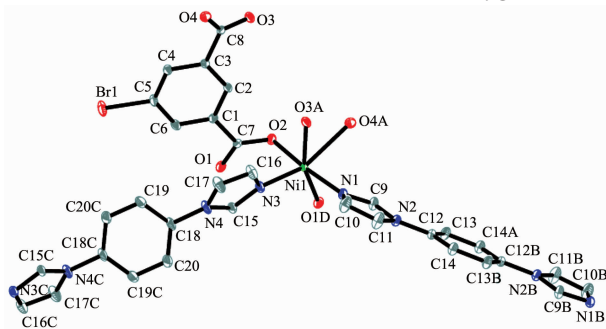


All hydrogen atoms are omitted for clarity; Color code: $[\text{NiO}_4\text{N}_2]$, green octahedron

Fig.2 One single layer structure of **1** in the bc -plane and the carboxylate oxygen atoms ($\text{O1W}\cdots\text{O4}^a$, 0.255 2 nm; $\text{O1W}\cdots\text{O1}^b$, 0.269 6 nm. Symmetry code: ^a $-x+1, -y+2, -z+1$; ^b $-x+2, -y+2, -z+1$) in the layer.

2.1.2 Crystal structure description of $[\text{Ni}(\text{BIPA})(\text{L})]_n$ (**2**)

Compound **2** crystallizes in a monoclinic space group $P2_1/c$. The asymmetric unit contains one Ni(II) ion, one BIPA^{2-} and one L molecule. The Ni(II) ion is coordinated by carboxylate oxygen atoms (O2 , O1D , O3A , O4A) from three BIPA^{2-} and two nitrogen atoms (N1 , N3) from two L molecules to give a $[\text{NiO}_4\text{N}_2]$ distorted octahedral geometry (Fig.3). The Ni-N/O bond lengths are in the of 0.201 63(14)~0.236 2(15) nm. The BIPA^{2-} serves as a tridentate ligand through its four carboxylate oxygen atoms, two of which chelate a Ni (II) ion, each of two oxygen atoms



1,4'-bis(imidazol-yl)benzene) have been introduced the reaction system, and the d^8 Ni(II) prefers an octahedral geometry, while the Zn(II) usually shows a tetrahedral coordination environment in above compounds. Obviously, the difference could result in the different coordination modes of 3-NPAH₂ and H₂BIPA ligands, and hence the final structures.

2.2 PXRD patterns and thermal stabilities of compounds **1** and **2**

To characterize the thermal stabilities of compounds **1** and **2**, thermal analyses were performed for both compounds **1** and **2**. As shown in Fig.6, compound **1** is stable up to about 150 °C, and a weight loss of 2.60% in the temperature range 150~200 °C is close to the calculated value for the removal of one coordinated water molecule (Calcd. 4.00%). About 200 °C, another plateau is found until the burning of the compound occurs above *ca.* 290 °C. Between 290 and 400 °C, a quick weight loss of **1** is observed. Above 400 up to 800 °C, slowly weight loss is also found. In the case of compound **2**, a plateau appears between 30 and 400 °C, above which a rapid weight loss is observed owing to the burn of the organic groups and the collapse of structure. Since the plateaus are distinct in the temperature range 200~290 °C for **1**, 30~400 °C for **2**, dehydrated product **1'** and heated product **2'** were obtained by heating **1** and **2** at 280 °C and 380 °C, respectively, under a nitrogen flow at a rate of 5 °C·min⁻¹. From the powder XRD patterns, it is clear that the original structures of compounds **1** and **2** remain after heating (Fig.7 and Fig.8). The dehydration process is accompanied by a

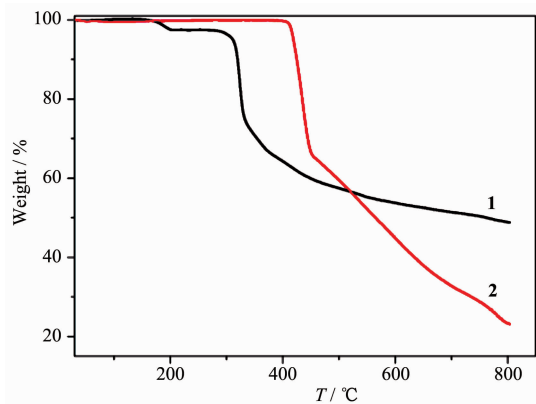


Fig.6 TGA curves for compounds **1** and **2**

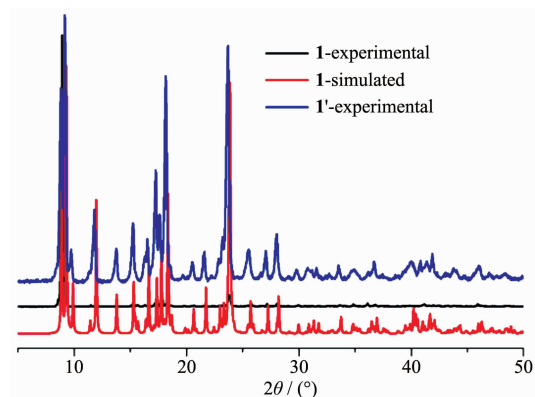


Fig.7 PXRD patterns for compound **1** and its dehydrated product **1'**

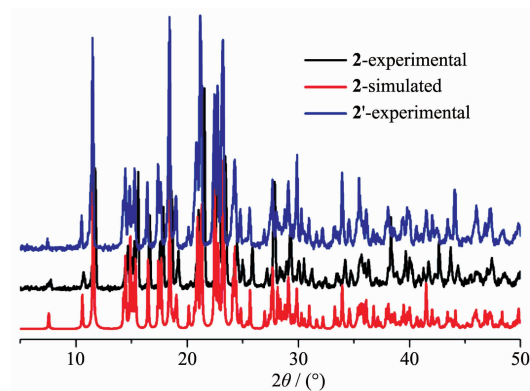


Fig.8 PXRD patterns for compound **2** and its heated product **2'**

color change from aquamarine to yellow for **1**. In order to understand the fact that the color change of **1** after releasing one coordinated water molecule, the IR of dehydrated products **1'** and **2'** have been measured (Fig.S1, Supporting informatin). The results show that characteristic bands of the carboxylate stretching vibrations at 1 600~1 300 cm⁻¹ do not change obviously and hence the final structure. The above result show that compound **2** displays relatively high thermal stabilities containing the rigid substitutional benzenedicarboxylic acid ligands (H₂BIPA) and the rigid N-donor ligands (1,4'-bis(imidazol-yl)benzene).

3 Conclusion

This paper reports two nickel (II) coordination polymer, namely, [Ni(3-NPA)(4,4'-bipy)(H₂O)]_n (**1**) and [Ni(BIPA)(L)]_n (**2**), based on the rigid 3-nitrophthalic acid (3-NPAH₂), 5-bromoisophthalic acid (H₂BIPA) as well as two N-donor ligands 4,4'-bipyridine(4,4'-bipy), 1,4'-bis(imidazol-yl)benzene (L). Compound **1** shows a

two-dimensional (2D) single layer structure. Compound **2** features a double chain structure containing a dimer unit $[\text{Ni}_2(\text{COO})_2]$, which is further extended into a 3D framework structure by the L molecules. Moreover, the thermal stabilities properties of compounds **1** and **2** have been investigated in detailed. The result of compound **2** shows the incorporation of the inorganic metal layers or chains within the structures of coordination polymers could significantly increase its thermal stabilities of the hybrid material.

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