Synthesis and Structural Characterization of a Ni(II) Coordination Polymer Based on 4,4'-Bipyridine and Acetato

Synthesis and Structural Characterization of a Ni(II) Coordination Polymer Based on 4,4'-Bipyridine and Acetato

Lihua Wang[1, *], DanDan Lin[2], Qian Liang[2]

[1]College of Information and Control Engineering, Weifang University, Weifang 261061, China
[2]College of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, China

*Corresponding author e-mail: taixs@wfu.edu.cn

Abstract. A new coordination polymer, $[Ni(bpy)(acetato)_2]_n$ (bpy = 4,4'-bipyridine), was synthesized and characterized by X-ray single-crystal structural analysis. For the complex: $C_{14}H_{14}N_2NiO_4$, M_r = 332.98, triclinic, space group *P*-1, a = 8.0538(16) Å, b = 9.1680(18) Å, c = 10.659(2) Å, α = 109.80(3)º, β = 99.90(3)º, γ = 101.81(3)º, V= 699.5(2) Å^3, Z = 2, Dc = 1.581g·cm^{-3}, μ = 1.403mm^{-1}, F(000) = 344, and final R_1 = 0.0603, ωR_2 = 0.1730. The Ni(II) ion adopts a distorted octahedral geometry coordinated by two N atoms and four O atoms. The Ni(II) complex forms 1D chain structure 2D layered structure by the bridge of 4,4'-bipyridine and acetato.

1. Introduction

In recent years, metal coordination polymer materials have been explored because of their novel structures and excellent properties such as antitumor activities [1-2], catalytic properties [3-5], luminescent properties [6-8], and magnetic properties [9, 10]. So the studies on synthesis, structure and property of metal coordination polymer materials are very important. Based on the above investigation, in this work, a new coordination polymer, $[Ni(bpy)(acetato)_2]_n$ (bpy = 4,4'-bipyridine), was synthesized and characterized by X-ray single-crystal structural analysis.

2. Experimental Section

2.1. Materials and Instrumentation

4, 4'-bipyridine, Ni $(CH_3COO)_2$ $4H_2O$ were purchased from Xiya reagent Company. The analyses of C, H and N were carried out with an Elementar Vario EL III elemental analyzer. The single-crystal diffraction data were collected on a Bruker Smart-1000 CCD diffractometer.

2.2. Synthesis of $[Ni(bpy)(acetato)_2]_n$

0.1560g 4,4'-bipyridine (1.0mmol) was dissolved in 10mL ethanol/water (v : v = 3 : 1) with stirring, then 0.2488g $Ni(CH_3COO)_2$ $4H_2O$ (1.0mmol) solid was added to the above solution. The above mixture was heated at 70 ℃ and kept at this temperature for 5h with stirring. The light green crystals of $[Ni(bpy)(acetato)_2]_n$ were obtained by slow evaporation of the filtrate solution after 20 days.

Elemental analysis calc. for $C_{14}H_{14}N_2NiO_4$: C, 50.45, H, 4.20, N, 8.41(%); Found: C, 50.59, H, 4.62, N, 8.13(%).

2.3. Crystal Data and Structure Determination

A single crystal of $[Ni(bpy)(acetato)_2]_n$ with dimensions of 0.26mm × 0.22mm × 0.18mm was mounted on a Bruker Smart-1000 CCD diffractometer equipped with a graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) at 293(2) K. 11208 total reflections were collected in the $2.94 \leq \theta \leq 25.00$ °, 5488 were independent with *R*int = 0.0448. The structure was solved by direct method using SHELXL-97 [11] and refined with SHELXTL-97 [12]. The crystal data of $[Ni(bpy)(acetato)_2]_n$ was listed in Table 1.

Table 1. Crystal data for $[Ni(bpy)(acetato)_2]_n$.

Formula	**$C_{14}H_{14}N_2NiO_4$**
Formula weight	332.98
Crystal system	triclinic
Space group	*P*-1
a [Å]	8.0538(16)
b [Å]	9.1680(18)
c [Å]	10.659(2)
α [ ]	109.80(3)
β [ ]	99.90(3)
γ [ ]	101.81(3)
Z	2
F(000)	344
Temperature [K]	293(2)
V [Å^3]	699.5(2)
Calculated density [g cm^{-3}]	1.581
Crystal size [mm^3]	0.26×0.22×0.18
μ [mm^{-1}]	1.403
S	1.322
Limiting indices	$-9 \leq h \leq 9$, $-10 \leq k \leq 10$, $-12 \leq l \leq 12$
Reflections collected	5488
Unique reflections	2460
Parameters	191
Restraints	0
R_{int}	0.0448
R_1, wR_2 [all data]	0.0655, 0.1785
R_1, wR_2 [$I>2\sigma(I)$]	0.0603, 0.1730
Largest diff.peak and hole [e ·Å^{-3}]	1.689, -0.509

3. Results and Discussion

3.1. Characterization

The result of elemental analyses shows that the Ni(II) coordination polymer, $[Ni(bpy)(acetato)_2]_n$, has been synthesized successfully.

3.2. Structural Description of [Ni(bpy)(acetato)$_2$]$_n$

The coordination environment of Ni(II) ions in [Ni(bpy)(acetato)$_2$]$_n$ is shown in Figure 1. The 1D and 2D structure of [Ni(bpy)(acetato)$_2$]$_n$ is given in Figure 2. The crystal structure analysis indicates that the symmetric unit is made up of one Ni(II) atom, one 4,4'-bipyridine and two acetatos ligand. In Figure 1, Ni(II) is six-coordinated with four oxygen atoms (O1, O2, O3, O4) of coordinated acetato and two nitrogen atoms (N1, N2) from two different the 4,4'-bipyridine ligands and exhibits a distorted octahedron coordination geometry. The acetato groups adopt two coordination modes (monodentate and bidentate). The sum of bond angle around Ni1 is 359.92 °, indicating that O1, O2, O3 and O4 locate at the equatorial plane, and N1 and N2 locate at the axial position. In [Ni(bpy)(acetato)$_2$]$_n$, 4,4'-bipyridine ligand acts as bridging ligands to link Ni(II) ions by N atoms to form an infinite 1D chained structure (Figure 2). The 1D chain are further interacted by bridging acetato ligands and π-π stacking of 4,4'-bipyridine ligands to form a 2D layered structure (Figure 2).

Selected bonds: Ni1-O1 2.011(3) Å; Ni1-O2 2.021(3) Å; Ni1-O3 2.287(5) Å; Ni1-O4 2.173(4) Å; Ni1-N1 2.169(4) Å; Ni1-N2 2.171(4) Å; C1-N2 1.335(6) Å; C2-N2 1.345(6) Å; C10-N1 1.331(6) Å; C8-N1 1.345(6) Å; C11-O2 1.243(6) Å; C11-O1 1.252(6) Å; C13-O3 1.232(6) Å; C13-O4 1.248(6) Å; C1-C3 1.385(7) Å; C3-C5 1.390(6) Å;

Selected angles: O1-Ni1-O2 123.47(16) °; O1-Ni1-O4 147.56(17) °; O2-Ni1-O4 88.80(15) °; O1-Ni1-N1 91.79(13) °; N1-Ni1-O2 91.94(14) °; O4-Ni1-N1 90.17(15) °; N2-Ni1-O1 85.87(13) °; O2-Ni1-N2 87.36(14) °; O4-Ni1-N2 93.10(15) °; N2-Ni1-N1 176.65(13) °; O1-Ni1-O3 90.90(16) °; O2-Ni1-O3 145.53(16) °; O3-Ni1-O4 56.75(14) °; O3-Ni1-N1 89.35(16) °; O3-Ni1-N2 93.09(16) °; N2-C1-C3 124.4(4) °; N2-C2-C4 124.2(4) °; O1-C11-O2 126.9(5) °; O1-C11-C12 115.9(5) °; O2-C11-C12 117.2(5) °; O3-C13-O4 117.6(5) °; O3-C13-C14 123.0(6) °; C10-N1-C8 115.2(4) °; C1-N2-C2 115.0(4) °;

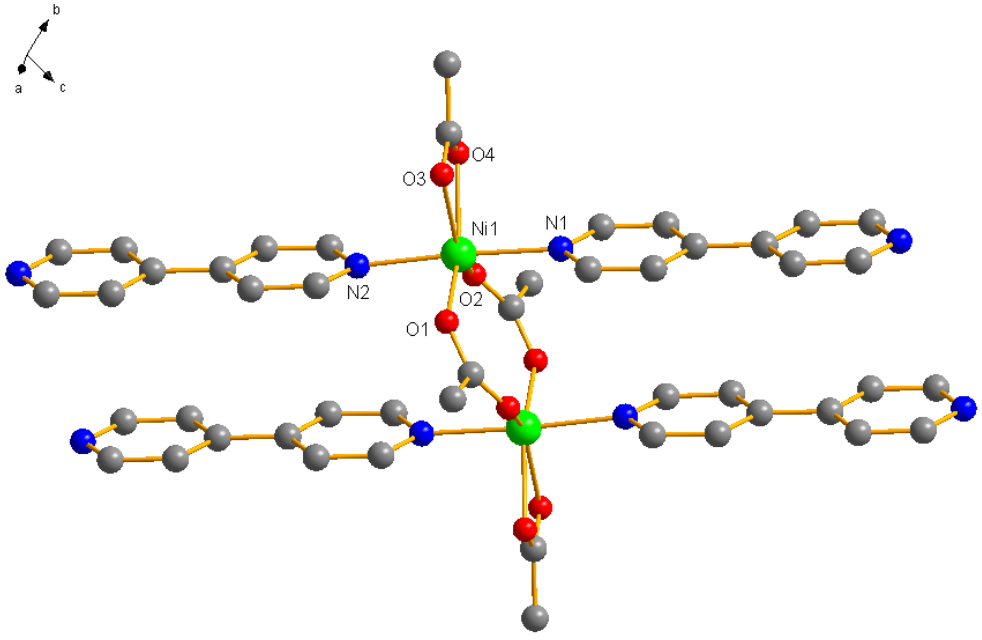

Figure 1. The coordination environment of Ni(II) ions in [Ni(bpy)(acetato)$_2$]$_n$.

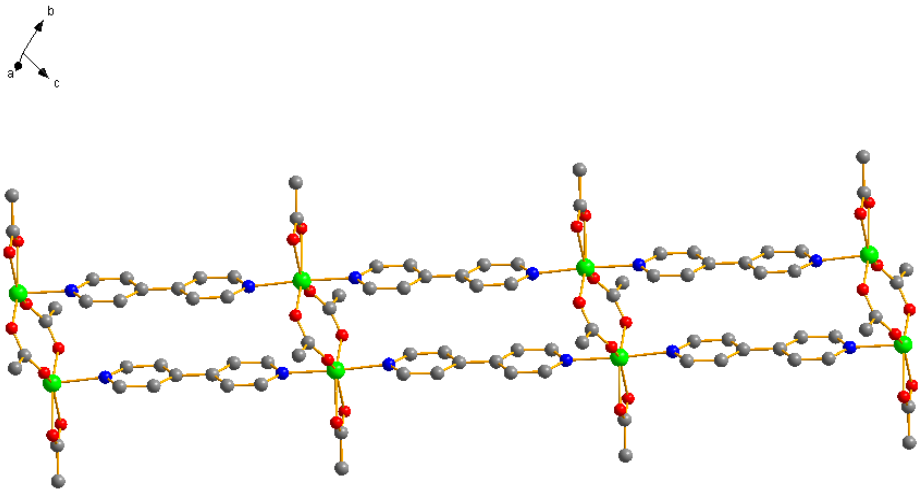

Figure 2. 1D and 2D structure of $[Ni(bpy)(acetato)_2]_n$.

References

[1] X.S. Tai, X. Wang, Synthesis, structural characterization and antitumor activity of a Ca(II) coordination polymer based on 4-formyl-1,3-benzenedisulfonate-2-furoic acid hydrazide ligands, Crystallogr. Rep. 62(2017) 242-245.

[2] X.S. Tai, W.H. Zhao, Synthesis, crystal structure and antitumor activity of Ca(II) coordination polymer based on 1,5-naphthalenedisulfonate, J. Inorg. Organomet. Polym. 23(2013) 1354-1357.

[3] Y. Yun, G.F. Xu, S, Ren, Synthesis and catalytic property of a rare earth coordination polymer, Oxid. Commun. 39(2016)2513-2518.

[4] X.L. Wang, X.T. Sha, G.C. Liu, A.X. Tian, H.Y. Lin, Structural influencing factors on ZnII/CdII coordination polymers based on a "V"-like bis-pyridyl-bis-amide derivative: construction, fluorescent sensing and photocatalysis properties, Inorg. Chim. Acta 440(2016)94-101.

[5] W.L. Leong, J.J. Vittal, One-dimensional coordination polymers: complexity and diversity in structures, properties, and applications, Chem. Rev. 111(2011)688-764.

[6] Y.R. Huang, L.L. Gao, X.Q. Wang, L.M. Fan, T.P. Hu, Syntheses, structures and luminescent properties of two novel Zn (II) coordination polymers, J. Solid State Chem. 258(2018)854-858.

[7] J. Liu, F. Wang, J. Zhang, Synthesis, structure and luminescent of Ag based homochiral metal tetrazolate coordination polymers, Inorg. Chem. Commun. 89(2018)41-45.

[8] X.S. Tai, H.Y. You, A new 1D chained coordination polymer: synthesis, crystal structure, antitumor activity and luminescent property, Crystals 5(2015)608-616.

[9] W.T. Huang, H. Zhou, Coordination polymers of transition metal diphosphonates: synthesis, crystal structure and magnetic behaviour, Polyhedron 154(2018)420-429.

[10] Y.L. Liu, P. Liu, K.B. Li, C.S. Zhou, K.F. Yue, Two new coordination polymers based on a flexible bib ligand: structures and magnetic properties, J. Mol. Struct. 1147(2017)192-196.

[11] G.M. Sheldrick, SHELXL-97, Program for Crystal Structure Solution; University of Göttingen: Göttingen, Germany, 1997.

[12] G.M. Sheldrick, SHELXTL-97, Program for Crystal Structure Refinement; University of Göttingen: Göttingen, Germany, 1997.