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基于 α,α-*L*-二芳基脯氨醇–吡啶衍生物的手性 Ag(I) 配位聚合物:圆二色谱、二次谐波响应和发光性质

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摘要:首次合成了 α,α-*L*-二芳基脯氨醇-吡啶衍生物:*S*-二(4-(吡啶-4-基)苯基)(吡咯烷-2-基)甲醇 (L),并用其与硝酸银自组装出具有梯形链结构的一维手性 Ag(I)配位聚合物{[Ag_4(L)$_4$](NO_3)$_4$·1.5CH_3OH·1.25H_2O}$_n$ (**1**);用 IR、XRD、TG、粉末 XRD 和单晶 XRD 对聚合物结构进行了表征。圆二色谱和二次谐波响应测试也证明了它具有结构上的手性。此外,还研究了其荧光性质。

关键词:手性配位聚合物;α,α-*L*-二芳基脯氨醇;圆二色谱;荧光性质
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A Chiral Ag(I) Coordination Polymer Based on an α,α-*L*-Diaryl Prolinol-Pyridine Derivative: Circular Dichroism, SHG Response and Luminescent Property

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Abstract: A chiral α,α-*L*-diaryl prolinol-pyridine derivative, (*S*)-bis(4-(pyridin-4-yl)phenyl)(pyrrolidin-2-yl)methanol (L), was synthesized and used to construct a chiral coordination polymer, {[Ag_4(L)$_4$](NO_3)$_4$·1.5CH_3OH·1.25H_2O}$_n$ (**1**), with Ag(I). The polymer displayed a one-dimensional ladder-like chain structure, which was characterized by single crystal XRD, PXRD, IR spectra, TGA and luminescent spectra. CD spectra and SHG response of the compounds confirmed that the bulk sample was of structural chirality. CCDC: 1938582, **1**.

Keywords: chiral coordination polymer; α,α-*L*-diaryl prolinol; CD spectra; luminescent property

0 Introduction

Chiral coordination polymers (CCPs), as a kind of functional crystal materials, have attracted comprehensive efforts to design and explore, because of their interesting topologies and structures[1-5] and their widely potential applications in heterogeneous catalysis[6-7], luminescence[8-9], gas storage[10-11], biomolecular loading[12-13], and so on. The potential properties and practical applications of such polymers are mainly dependent on the assembly of metal ions/clusters and organic linkers with accessible functional groups[14-15].

Proline is an important small chiral amino acid and one of the most well-known privileged chiral organic catalysts. Due to its unique properties, such as conformational rigidity, inexpensive, nontoxic and environmentally friendly, proline and its various derivatives have been used as the catalysts for

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asymmetric catalytic reactions[16-17]. The incorporation of chiral proline and metal-organic frameworks (MOFs) have been deeply investigated to constructed heterogeneous catalysts via direct grafting to the linker[18-19], post-synthetic modification of the achiral linker backbone[20-21], or coordination to the open metal sites of the cluster[22-23]. α,α-*L*-diaryl prolinols, as a famous branch of *L*-proline derivatives, have been applied in a wide range of asymmetric organocatalysis catalysis[24]. However, to our best knowledge, only one chiral coordination polymer/MOF, based on α,α-*L*-diaryl prolinols, has been reported, in which a chiral Cu-MOF was constructed by chiral proline carboxylic acid derivative and applied in enantioselective aldol addition[25]. Herein, we synthesized a new chiral α,α-*L*-diaryl prolinol-pyridine ligand and constructed a chiral coordination polymer with silver ions under mild condition. The polymer displayed a one-dimensional (1D) ladder-like chain structure, which has been characterized by single crystal X-ray diffraction, powdered X-ray diffraction (PXRD), infrared (IR) spectra, thermogravimetric analysis (TGA), circular dichroism (CD) spectra, second-harmonic generation (SHG) and luminescent spectra.

1 Experimental

1.1 Materials and method

All chemicals were purchased and used without further purification. The ligand (L) synthesis was described in the following experimental section.

^{1}H NMR spectra were obtained on a Bruker 600 MHz ADVANCE spectrometer. Mass spectra were afforded on Agilent 1260-6224. IR spectra (KBr Pellets) were collected on a Nicolet FT-IR 200 spectrophotometer Infrared in the mid-IR region. Circular dichroism (CD) spectra were recorded with JASCO J-810 spectrophotometer at room temperature under air. Powder X-ray diffraction (PXRD) patterns were collected on an Rigaku, D/max-2500 X-ray diffractometer operating at 36 kV and 30 mA using Mo *Kα* radiation (λ=0.071 073 nm) within the 2θ range of 5°~50° and the scan rate was 5°·min^{-1}. Thermogravimetric analysis (TGA) was performed on a Netzsch STA 449 F3 Jupiter in a temperature range of 30~900 ℃ in N_2 and a heating rate of 10 ℃·min^{-1}. Kurtz powder method was used to test the second-harmonic generation (SHG) efficiency of **1**.

1.2 Synthesis of ligand L

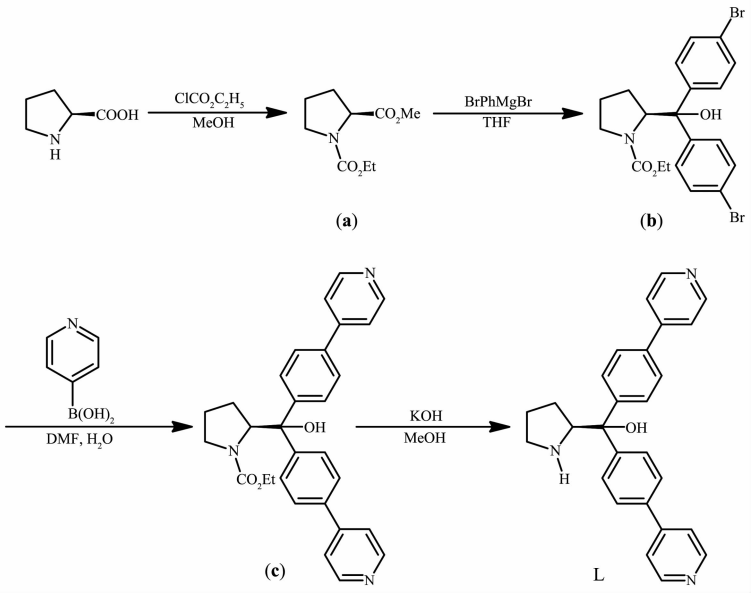
1.2.1 Syntheses of (*S*)-1-ethyl 2-methyl pyrrolidine-1,2-dicarboxylate (**a**) and (*S*)-ethyl 2-(bis(4-bromophenyl)(hydroxy)methyl)pyrrolidine-1-carboxylate (**b**)

Compounds **a** and **b** were prepared in the same manner as reported in the literature[26].

The product **b** was a white solid (10.602 g, Yield: 25.25%). ^{1}H NMR (600 MHz, $CDCl_3$, 25 ℃, TMS): 7.43~7.41 (m, 4H), 7.27~7.22 (m, 4H), 4.82 (dd, *J*=9.0, 4.0 Hz, 1H), 4.13~4.10 (m, 2H), 3.44~3.43 (m, 1H), 2.96 (m, 1H), 2.11~2.03 (m, 1H), 2.08~1.81 (m, 1H), 1.52~1.50 (m, 1H), 1.24~1.21 (t, *J*=7.0 Hz, 3H), 0.91~0.87 (m, 1H). ESI MS (*m/z*): Calcd. for $C_{20}H_{21}Br_2NO_3$ $[M-H]^+$: 481.979 3, Found: 481.979 3.

1.2.2 Synthesis of (*S*)-ethyl 2-(hydroxybis(4-(pyridin-4-yl)phenyl)methyl)pyrrolidine-1-carboxylate (**c**)

Under N_2 atmosphere, a mixture of **b** (10.612 g, 0.022 mol), pyridine-4-boronic acid (11.802 g, 0.066 mol), anhydrous K_2CO_3 (18.812 g, 0.11 mol), THF (100 mL) and H_2O (100 mL) was stirred for 10 min. Before the addition of $Pd(PPh_3)_4$ catalyst, O_2 was removed by a N_2 purge by vacuuming, and then the reaction was heated to reflux for 4 hours, and was monitored by TLC. After the reaction was completed, it was concentrated, followed by stirring with 100 mL water, extracting three times with 100 mL ethyl acetate, drying over anhydrous magnesium sulfate overnight, filtering and purifying by column chromatography on silica gel. The organic phase was combined, then washed with saturated $NaHCO_3$ solution and purified by column on silica gel to afford product **c** (5.612 g, Yield: 43.04%). ^{1}H NMR (600 MHz, $CDCl_3$, 25 ℃, TMS): 8.11~8.10 (d, 4H), 7.70~7.66 (d, 4H), 7.61~7.59 (m, 4H), 7.54~7.52 (m, 4H), 4.17~4.10 (m, 2H), 3.97~3.94 (m, 6H), 3.48~3.37 (m, 1H), 3.02 (m, 1H), 2.19~2.15 (m, 1H), 2.04 (m, 1H), 1.57~1.55 (m, 1H), 1.28~1.24 (d, 4H), 0.97~0.96 (m, 1H); ESI MS (*m/z*): Calcd. for $C_{30}H_{29}N_3O_3$ $[M+1]^+$: 480.207 4, Found: 480.227 4.



Scheme 1　Synthetic route of ligand L

1.2.3　Synthesis of (*S*)-bis(4-(pyridin-4-yl)phenyl) (pyrrolidin-2-yl)methanol (L)

A mixture of **c** (5.612 g, 0.012 mol), KOH (5.6 g, 0.1 mol) in methanol (50 mL) and H_2O (3 mL) was stirred and heated at reflux overnight. After the reaction was finished, the mixture was cooled to room temperature. Then it was concentrated in vacuum to remove a portion of methanol, followed by stirring with 100 mL water, extracting three times with 100 mL ethyl acetate, drying over anhydrous magnesium sulfate overnight, evaporating to afford the product L (3.212 g, Yield: 65.56%). IR (KBr) cm^{-1}: 3 285(s), 3 071(s), 3 031(m), 2 963(w), 2 863(m), 2 436(m), 1 933 (m), 1 739(m), 1 645(s), 1 592(m), 1 532(m), 1 472(m), 1 398(m), 1 324(m), 1 284(m), 1 224(s), 1 170(m), 1 084 (m), 1 024(m), 983(s), 909 (m), 849(m), 803(m), 736 (m), 629(m). ^1H NMR (600 MHz, d_6-DMSO, 25 ℃, TMS): 8.62~8.60 (d, 4H), 7.77~7.66 (m, 12H), 4.37 (s, 1H), 3.46~3.37 (m, 2H), 2.90~2.83 (d, 2H), 1.68~1.58 (m, 2H), 1.52~1.44 (m, 2H); ESI MS (*m*/*z*): Calcd. for $C_{27}H_{25}N_3O[M+1]^+$: 408.207 4, Found: 408.207 4.

1.3　Synthesis of polymer 1

1.3.1　Synthesis of $\{[Ag_4(L)_4](NO_3)_4\cdot 1.5CH_3OH\cdot 1.25H_2O\}_n$ (**1**)

A mixture of L (40.7 mg, 0.1 mmol), $AgNO_3$ (17.1 mg, 0.1 mmol), H_2O (10 mL) and MeOH (10 mL) were stirred for half an hour at room temperature, then filtered. The filtrate was evaporated in the dark for 20 days. Colorless transparent block crystals of **1** were obtained and dried in air (18 mg, Yield: 30.3% based on $AgNO_3$). Anal. Calcd. for $C_{109.5}H_{106.5}Ag_4N_{16}O_{18.75}$(%): C 55.30, H 4.51, N 9.42; Found(%): C 54.63, H 4.80, N 9.37. IR (KBr) cm^{-1}: 3 406(s), 3 225(s), 2 957(m), 2 877 (w), 1 920(m), 1 606(m), 1 545(s), 1 485(m), 1 378(m), 1 327(m), 1 224(m), 1 097(m), 1 070(m), 996(s), 809 (m), 756(m).

1.4　X-ray diffraction analysis

A suitable single crystal of **1** with approximate dimensions was mounted on a Bruker D8 Venture diffractometer. The diffraction data were collected using a graphite monochromated Ga *Kα* radiation (λ = 0.134 138 nm) at 193(2) K. Absorption corrections were applied using SADABS[27]. The structure was solved by using the SHELXS-2018/3 program package[28]. All non-hydrogen atoms were isotropically refined initially and subsequently treated anisotropically (with the exception of the disordered atoms). The organic hydrogen atoms were generated geometrically. The assignment of the absolute structures for **1** was confirmed by the refinement of the Flack enantiopole parameter to values of 0.068(16)[29]. One nitrate in **1** was badly disordered. N-O bond lengths and O-N-O bond angles of the disordered nitrate were restrained to chemically reasonable values using the DFIX

commands in SHELXS-2018/3[28]. Crystal data as well as details of the data collection and refinements for the complexes were summarized in Table 1. Selected bond distances and bond angles were listed in Table 2.

CCDC: 1938582, **1**.

Table 1 Crystal data and structure refinement parameters of 1

Formula	$C_{438}H_{426}Ag_{16}N_{64}O_{75}$	$F(000)$	4 854
Formula weight	9 512.32	Reflection	57 913
Crystal system	Monoclinic	Unique	17 283
Space group	$C2$	Observed data [$I>2\sigma(I)$]	11 746
a / nm	3.234 4(1)	R_{int}	0.078
b / nm	2.349 7(1)	μ / mm^{-1}	4.526
c / nm	1.445 9(1)	Flack x	0.068(16)
β / (°)	111.016(2)	R_1[a] [$I>2\sigma(I)$]	0.073
V / nm^3	10.257 3(5)	wR_2[b] (all data)	0.207 4
Z	1	GOF	0.99
D_c / (g·cm^{-3})	1.54	Largest diff. peak and hole / (e·nm^{-3})	1 020, −760

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

Table 2 Selected bond distances (nm) and bond angles (°) of 1

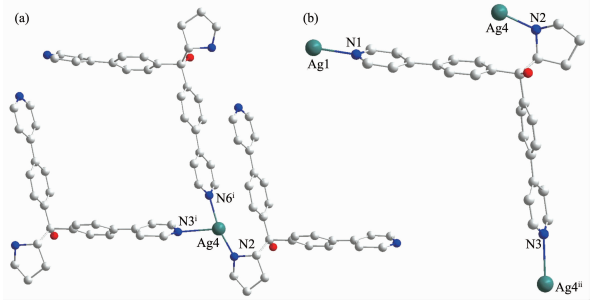
Ag1-N5^{i}	0.221 5(11)	Ag1-N1	0.225 3(12)	Ag1-N4	0.243 1(13)
Ag2-N8	0.222 4(11)	Ag2-N12	0.219 0(14)	Ag2-N9^{ii}	0.259 9(11)
Ag3-N7^{ii}	0.220 7(17)	Ag3-N10	0.253 8(13)	Ag3-N11^{ii}	0.222 1(13)
Ag4-N2	0.219 7(11)	Ag4-N3^{i}	0.267 7(15)	Ag4-N6i	0.222 3(13)
N5^{i}-Ag1-N1	150.4(4)	N1-Ag1-N4	84.7(5)	N5^{i}-Ag1-N4	115.7(4)
N12-Ag2-N9^{ii}	94.8(4)	N12-Ag2-N8	159.8(4)	N8-Ag2-N9^{ii}	103.6(4)
N7^{ii}-Ag3-N11^{ii}	157.4(5)	N11^{ii}-Ag3-N10	107.9(4)	N7^{ii}-Ag3-N10	88.6(5)
N6^{i}-Ag4-N3^{i}	77.98(16)	N6^{i}-Ag4-N2	162.1(4)	N2-Ag4-N3^{i}	113.75(17)

Symmetry codes: ^{i} x, y, $-1+z$; ^{ii} x, y, $1+z$.

2 Results and discussion

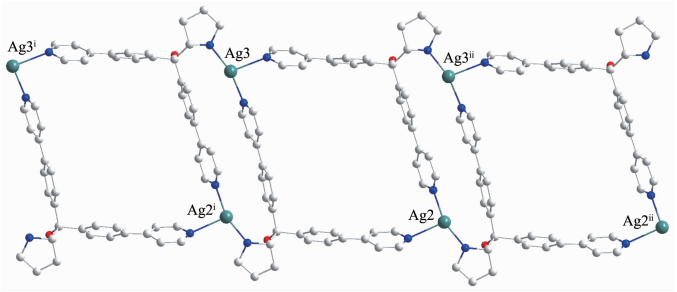
2.1 Crystal structure of 1

Single-crystal XRD study reveals that **1** crystallizes in the monoclinic system with chiral space group $C2$, consisting of four Ag(I) ions, four chiral L ligands, four nitrates, one and a half free methanol molecules, one and a quarter lattice water molecules in an a symmetric unit. Four crystallographically independent Ag(I) ions contain the same coordination mode with similar bond lengths and bond angles, and display an approximately trigonal planar geometry, surrounded by two nitrogen atoms of two pyridine ring and one nitrogen atom of pyrrolidine ring, respectively, from three adjacent organic ligands (Fig.1a). Meanwhile,



Symmetry codes: ^{i} x, y, $-1+z$; ^{ii} x, y, $1+z$

Fig.1 Coordination environment of Ag(I) (a) and coordination mode of L (b)



Symmetry code: ⁱ x, y, $-1+z$; ⁱⁱ x, y, $1+z$

Fig.2　One dimensional ladder-like chain structure in **1**

each chiral organic ligand in **1** acts in a tetradentate mode (Fig.1b) and bridges four adjacent Ag(I) ions to build a 1D ladder-like chain structure (Fig.2).

2.2　Infrared spectra and powdered X-ray diffraction

The IR bands, being most useful for defining the coordination mode of L and **1**, were $\nu(C{=}N)_{pyridine}$ and $\nu(C\text{-}N)_{pyrrole}$ vibrations[30]. As shown in Fig.3, such two bands of the ligand L were at 1 645 and 1 284 cm^{-1}; however, they shifted to 1 606 and 1 224 cm^{-1}, respectively, in **1**, which indicates that the N atoms on the pyridine and pyrrole participated in the coordination. In addition, the infrared spectrum of complex **1** had absorption peaks at 1 485, 1 378, and 1 327 cm^{-1}, indicating the presence of nitrate[31-32], which is consistent with the resolved crystal structure.

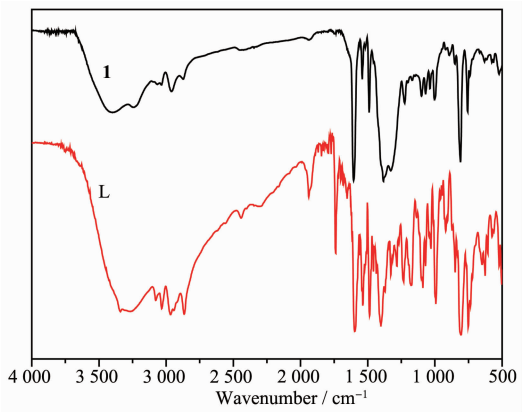


Fig.3　Infrared spectra of **1** and L

The simulated and experimental PXRD patterns of coordination polymer **1** were given in Fig.4. The results show that the crystal structures are truly representative of the bulk materials[33]. The differences in intensity are due to the preferred orientation of the powder samples[33].

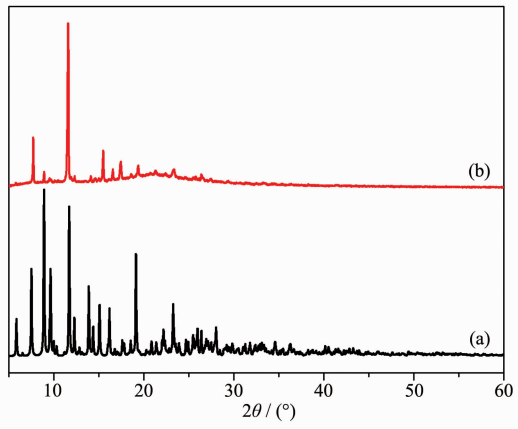


Fig.4　PXRD patterns of polymer **1**: (a) simulated from X-ray single crystal data; (b) as newly synthesized

2.3　TG analysis

To confirm the stability and structural integrity of **1** at elevated temperatures, the powdered sample was examined by TGA[34]. The test conditions ranged from 30 to 900 ℃ in a N_2 atmosphere at a heating rate of 10 ℃·min^{-1}. The sample was first dried in vacuum to remove any residual solvent. From the thermogravimetric curve shown in Fig.5, there were two obvious and separate thermal weight-loss processes due to the presence of lattice water and methanol molecules. The

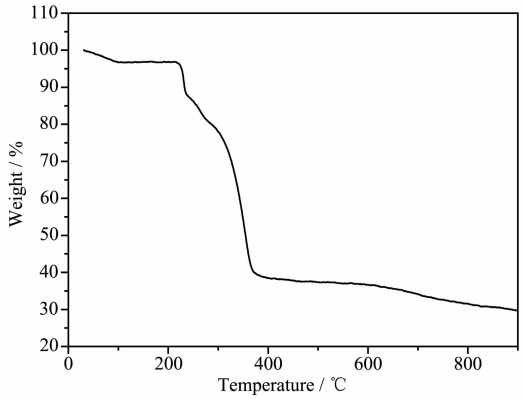


Fig.5　TG analysis of **1**

first weight loss stage began at 30 ℃ and ended at 108 ℃, which is due to the release of all the water and methanol molecules (Obsd. 3.2%, Calcd. 3.0%). The second stage corresponding to the removal of the ligands was observed between 108 and 400 ℃, indicating the decomposition of the whole structure.

2.4 Circular dichroism spectra and second-harmonic generation

CD spectrum has been proved to be a useful tool to analyze the enantiomeric optical activity of the bulk crystals[35-39]. Powdered bulk samples of the ligand L and polymer **1** were used to confirm their chiral nature in a KBr matrix between 180 and 400 nm at room temperature, as shown in Fig.6. The ligand L and **1** presented similar dichroic signals in CD spectrum, with two positive Cotton effects at 267 and 282 nm, as well as 274 and 310 nm frequencies, and two negative Cotton effects at 261 and 274 nm, as well as 270 and 297 nm frequencies, respectively, which confirms the chirality of the bulk samples.

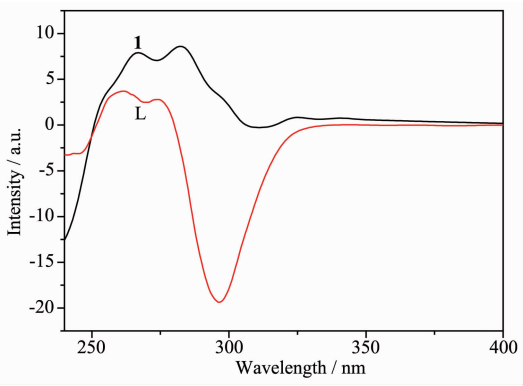


Fig.6 CD spectra of complex **1** and L in the solid state at room temperature

Second harmonic generation (SHG) was an effective method to analyze the asymmetry of the crystals. Polymer **1** is worthwhile to test the NLO properties because it is a chiral coordination polymer with chiral *C*2 space group[35-40]. The preliminary test results of the powdered sample suggest that **1** has SHG efficiency, approximately 1.2 times as big as that of KDP, which indicated that it can be applied to second-order nonlinear materials.

2.5 Luminescent property

It is well-known that d^{10} complexes always exhibited excellent luminescent properties[41]. As illustrated in Fig.7, the luminescent properties of the ligand L and **1** in a methanol solution were investigated at room temperature. The ligand L exhibited an intense UV radiation with λ_{max} at 366 nm upon excitation at 300 nm, which may be attributed to the π-π* transition. The emission spectra of **1** showed a purple luminescence emission at 384 nm upon excitation at 300 nm. Compared to the ligand, a red shift of 18 nm occurred, which may be assigned to charge transfer between the ligand and the metal ions in **1**[32].

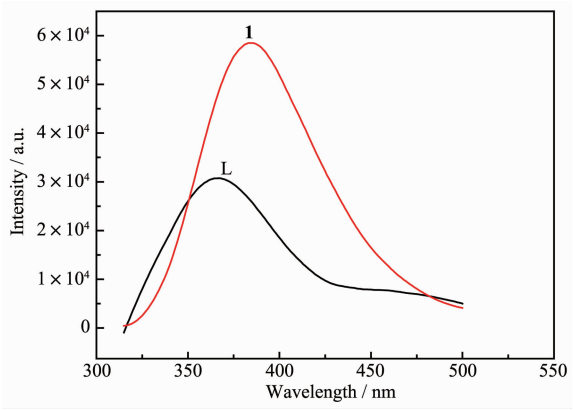


Fig.7 Luminescent spectra of complex **1** and ligand L in a methanol solution

3 Conclusions

In summary, chiral *L*-proline was used as a starting material to synthesize a new pyridine-functionalized α,α-*L*-diaryl prolinol ligand. The structure of the ligand was characterized by NMR, MS and IR, and successfully applied to construct a chiral Ag(I) coordination polymer with 1D ladder-like chain structure. CD spectrum and SHG response indicate that the bulk sample of **1** is chiral. Moreover, the luminescent properties of the ligand and polymer were also studied.

Supporting information is available at http://www.wjhxxb.cn

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