

## 基于吡啶基苯甲酸盐的两个银(I)配合物的合成、晶体结构及荧光性质

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**摘要:** 合成了 2 个含有吡啶基苯甲酸盐的银(I)配合物, 即  $[\text{Ag}_2(\text{PPh}_3)_2(4,4\text{-pybz})_2(\text{H}_2\text{O})_2]_n$  (**1**) 和  $[\text{Ag}_2(\text{PPh}_3)_2(4,3\text{-pybz})_2] \cdot 2\text{CH}_3\text{OH}$  (**2**) ( $\text{PPh}_3$ =三苯基膦, 4, 4-pybz=4-吡啶-4-基-苯甲酸根, 4,3-pybz=4-吡啶-3-基-苯甲酸根), 并通过红外光谱、元素分析和荧光光谱进行分析和表征, 它们的结构由 X 射线单晶衍射测定。在不同的溶剂下, 2 个配合物由  $\text{AgBF}_4$ 、 $\text{PPh}_3$  和不同的吡啶苯甲酸在氨水作用下, 以 1:1:1 的比例反应而成。在配合物 **1** 中, 所有的银原子由吡啶基苯甲酸桥连形成一维链状结构。在配合物 **2** 中, 2 个银原子通过 2 个 4-吡啶-3-基-苯甲酸根配体形成双核结构。在荧光光谱中, 在发射状态下所有的峰均来源于配体的  $\pi$ - $\pi^*$  跃迁。

**关键词:** 银; 三苯基膦; 4-吡啶-3-基-苯甲酸; 4-吡啶-4-基-苯甲酸

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## Syntheses, Crystal Structures and Fluorescence Properties of Two Silver(I) Complexes Derived from Pyridylbenzoate Ligands

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**Abstract:** Two silver (I) complexes with pyridylbenzoate ligands, namely  $[\text{Ag}_2(\text{PPh}_3)_2(4,4\text{-pybz})_2(\text{H}_2\text{O})_2]_n$  (**1**),  $[\text{Ag}_2(\text{PPh}_3)_2(4,3\text{-pybz})_2] \cdot 2\text{CH}_3\text{OH}$  (**2**) ( $\text{PPh}_3$ =triphenylphosphine, 4,4-pybz=4-pyridin-4-yl-benzoate, 4,3-pybz=4-pyridin-3-yl-benzoate) were synthesized and characterized by IR, elemental analysis and fluorescence spectra, and their structures were elucidated by single-crystal X-ray diffraction. They have been made by reacting  $\text{AgBF}_4$ ,  $\text{PPh}_3$ , different pyridylbenzoic acids in 1:1:1 molar ratio with ammonia water under different mixed solution conditions. In **1**, the Ag atoms are bridged by pyridylbenzoate-containing ligand to form infinite chain structure. In **2**, two Ag atoms are bridged by two 4-pyridin-3-yl-benzoate ligands to form binuclear complex. In fluorescence spectra, these emission peaks indicate that the origin of these emissions all involves emissive state derived from ligand-centered  $\pi$ - $\pi^*$  transition.

**Keywords:** silver; triphenylphosphine; 4-pyridin-3-yl-benzoic acid; 4-pyridin-4-yl-benzoic acid

## 0 Introduction

In recent year, the research of coordination

polymers have been of considerable interests due to important applications in gas storage<sup>[1-2]</sup>, heterogeneous catalysis<sup>[3-4]</sup>, sensing<sup>[5-6]</sup>, high photosensitivity material<sup>[7]</sup>

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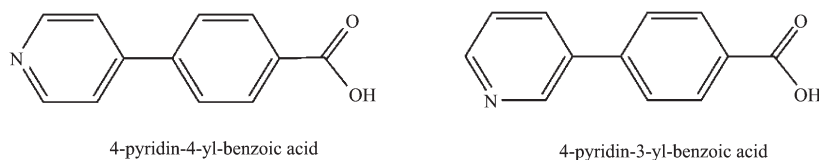
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and magnetic material<sup>[8]</sup>. The preparation of coordination polymers or complexes can be controlled by many factors, including temperature, solution, and concentration. Closed-shell  $d^{10}$  metal (Cu, Ag, Au) ions used for the construction of compound have also attracted attention on their coordination geometry, especially Ag (I) ions. Owing to their variable coordination number and flexible geometry and properties, they have been widely used<sup>[9]</sup>.

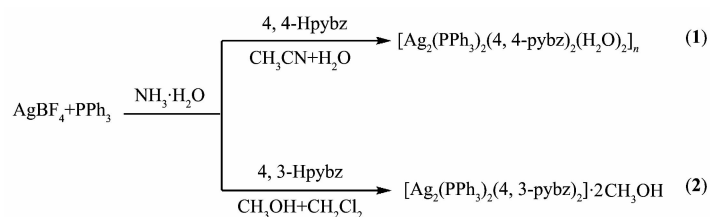
Pyridylbenzoate ligand is a useful kind of bridging ligand because it contains unsymmetrical bifunctional groups. In view of this character, it has a chance of generating chiral coordination polymers<sup>[10]</sup>. 4-pyridin-4-yl-benzoic acid (4,4-Hpybz) and 4-pyridin-3-yl-benzoic acid (4,3-Hpybz) are two rigid linear

kinds of ligands, which have the O and N donors on the opposite sides. In the previous studies, a number of group 11 or heterometallic organic frameworks with 1D, 2D and 3D have reported<sup>[11-14]</sup>.

Based on these considerations, we choose pyridylbenzoate ligand and report in this paper two coordination complexes derived from triphenylphosphine ligand and pyridylbenzoate ligands, namely  $[\text{Ag}_2(\text{PPh}_3)_2(4,4\text{-pybz})_2(\text{H}_2\text{O})_2]_n$  (**1**) and  $[\text{Ag}_2(\text{PPh}_3)_2(4,3\text{-pybz})_2] \cdot \text{CH}_3\text{OH}$  (**2**) (4,4-pybz = 4-pyridin-4-yl-benzoate, 4,3-pybz = 4-pyridin-3-yl-benzoate). They were synthesized and characterized by IR, elemental analysis and fluorescence spectrum, and their structures were elucidated by single-crystal X-ray diffraction.



Scheme 1 Ligands used in the present work



Scheme 2 Routine of synthesis for complexes **1** and **2**

## 1 Experimental

### 1.1 Materials and measurement

All chemical reagents are commercially available and used without furthermore treatment. FTIR spectra (KBr pellets) were measured on a Perkin-Elmer Infrared spectrometer. C, H and N elemental analysis were carried out on an ElementarVario MICRO CUBE (Germany) elemental analyzer.

### 1.2 Synthesis of $[\text{Ag}_2(\text{PPh}_3)_2(4,4\text{-pybz})_2(\text{H}_2\text{O})_2]_n$ (**1**)

A mixture of  $\text{AgBF}_4$  (0.2 mmol, 0.038 6 g), triphenylphosphine ( $\text{PPh}_3$ ) (0.2 mmol, 0.052 2 g) and 4-pyridin-4-yl-benzoic acid (4,4-Hpybz, 0.2 mmol,

0.040 0 g) with a little ammonia water were dissolved in a mixture of  $\text{CH}_3\text{CN}$  (5 mL) and  $\text{H}_2\text{O}$  (5 mL), stirred for 6 h and filtered. Colorless crystal **1** was obtained from the filtrate after standing at the room temperature for several days. Yield: 54%. Element analysis Calcd. for  $\text{C}_{60}\text{H}_{50}\text{Ag}_2\text{N}_2\text{O}_6\text{P}_2$  (%): C, 61.40; H, 4.26; N, 2.39; Found(%): C, 59.59; H, 4.16; N, 2.57. IR data ( $\text{cm}^{-1}$ , KBr pellets): 3 647w, 3 356m, 3 048m, 1 669w, 1 593s, 1 558s, 1 478m, 1 434m, 1 375s, 1 228w, 1 181w, 1 096w, 1 067w, 1 027w, 997w, 870w, 832m, 781s, 753s, 694s, 561w, 520m, 506m, 477m.

### 1.3 Synthesis of $[\text{Ag}_2(\text{PPh}_3)_2(4,3\text{-pybz})_2] \cdot 2\text{CH}_3\text{OH}$ (**2**)

Complex **2** was prepared in a manner similar to

that described for **1**, using  $\text{AgBF}_4$  (0.2 mmol, 0.039 2 g),  $\text{PPh}_3$  (0.2 mmol, 0.052 4 g) and 4-pyridin-3-ylbenzoic acid (4,3-Hpybz, 0.2 mmol, 0.039 8 g) with a little ammonia water as starting materials in a mixture of  $\text{CH}_3\text{OH}$  (5mL) and  $\text{CH}_2\text{Cl}_2$  (5 mL). Yield: 49%. Element analysis Calcd. for  $\text{C}_{62}\text{H}_{54}\text{Ag}_2\text{N}_2\text{O}_6\text{P}_2$  (%): C, 61.96; H, 4.50; N, 2.33. Found (%): C, 62.14; H, 4.45; N, 2.21. IR data ( $\text{cm}^{-1}$ , KBr pellets): 3 324w, 3 053w, 2 918w, 2 811w, 1 585m, 1 535m, 1 477m, 1 433m, 1 388s, 1 332w, 1 309w, 1 179w, 1 152w, 1 095m, 1 068w, 1 040m, 1 007w, 870w, 844w, 813w, 786m, 747s, 695s, 559w, 520m, 507m, 495m, 463w.

#### 1.4 Structure determination

Single crystals of the title complexes were mounted on a Bruker Smart 1000 CCD diffractometer equipped with a graphite-monochromated  $\text{Mo } K\alpha$  ( $\lambda = 0.071\ 073\ \text{nm}$ ) radiation at 298 K. Semi-empirical absorption corrections were applied using SADABS

program<sup>[15a]</sup>. All the structures were solved by direct methods using SHELXS program of the SHELXS-97 package and refined with SHELXL-97<sup>[15b]</sup>. Metal atom centers were located from the  $E$ -maps and other non-hydrogen atoms were located in successive difference Fourier syntheses. The final refinements were performed by full matrix least-squares methods with anisotropic thermal parameters for non-hydrogen atoms on  $F^2$ . The hydrogen atoms were generated geometrically and refined with displacement parameters riding on the concerned atoms.

Crystallographic data and experimental details for structural analysis are summarized in Table 1, and selected bond lengths and angles of complexes **1~2** are summarized in Table 2. The hydrogen bonds of complexes **1~2** are listed in Table 3.

CCDC: 890642, **1**; 890650, **2**.

Table 1 Crystallographic data for complexes **1~2**

	<b>1</b>	<b>2</b>
Formula	$\text{C}_{66}\text{H}_{50}\text{Ag}_2\text{N}_2\text{O}_6\text{P}_2$	$\text{C}_{62}\text{H}_{54}\text{Ag}_2\text{N}_2\text{O}_6\text{P}_2$
Formula weight	1 172.70	1 200.75
Crystal system	Orthorhombic	Monoclinic
Space group	$P2_12_12_1$	$P2_1/c$
Crystal size / mm	0.38×0.35×0.18	0.43×0.40×0.35
$a$ / nm	1.437 52(13)	1.725 45(12)
$b$ / nm	1.803 98(17)	0.872 55(6)
$c$ / nm	2.012 7(2)	1.904 92(14)
$\beta$ / (°)	90.00	108.265 0(10)
$V$ / nm <sup>3</sup>	5.219 4(9)	2.723 4(3)
Reflections collected, unique	25 800, 9 212	9 404, 4 809
Data, restraints, parameters	9 212, 0, 649	4 809, 0, 335
$Z$	4	2
$\mu$ / mm <sup>-1</sup>	0.866	0.831
$F(000)$	2 384	1 224
Goodness of fit on $F^2$	1.087	1.056
$R_{\text{int}}$	0.043 1	0.022 2
$R_1[I > 2\sigma(I)]^a$	0.030 9	0.033 6
$wR_2[I > 2\sigma(I)]^b$	0.064 7	0.077 3
$R_1(\text{all data})^a$	0.046 5	0.045 5
$wR_2(\text{all data})^b$	0.073 4	0.084 0

<sup>a</sup>  $R = \sum (|F_o| - |F_c|) / \sum |F_o|$ ; <sup>b</sup>  $wR = [\sum w(|F_o|^2 - |F_c|^2)^2 / \sum w(F_o^2)]^{1/2}$

**Table 2** Selected bond distances(nm) and bond angles(°) for complexes **1~2**

<b>1</b>					
Ag(1)-N(2)	0.232 5(3)	Ag(1)-P(1)	0.236 86(10)	Ag(1)-O(1)	0.234 6(3)
Ag(1)-O(5)	0.251 2(2)	Ag(2)-N(1)	0.225 3(3)	Ag(2)-P(2)	0.235 99(9)
Ag(2)-O(3)	0.233 2(3)	Ag(2)-O(6)	0.286 4(4)		
N(2)-Ag(1)-O(1)	95.32(11)	N(1)-Ag(2)-O(3)	95.56(11)	N(2)-Ag(1)-P(1)	129.17(9)
N(1)-Ag(2)-P(2)	135.13(9)	O(1)-Ag(1)-P(1)	132.87(8)	O(3)-Ag(2)-P(2)	128.68(8)
N(2)-Ag(1)-O(5)	90.02(11)	N(1)-Ag(2)-O(6)	98.50(12)	O(1)-Ag(1)-O(5)	86.87(10)
O(3)-Ag(2)-O(6)	86.84(10)	P(1)-Ag(1)-O(5)	105.34(7)	P(2)-Ag(2)-O(6)	91.87(8)
<b>2</b>					
Ag(1)-N(1)	0.234 2(2)	Ag(1)-O(1)	0.239 6(2)	Ag(1)-P(1)	0.235 50(7)
Ag(1)-O(2)	0.251 5(2)				
N(1)-Ag(1)-P(1)	136.69(6)	N(1)-Ag(1)-O(2)	88.70(8)	N(1)-Ag(1)-O(1)	92.17(8)
P(1)-Ag(1)-O(2)	121.76(6)	P(1)-Ag(1)-O(1)	130.14(5)	O(1)-Ag(1)-O(2)	53.38(8)

**Table 3** Hydrogen bonds of complexes **1~2**

D-H...A	<i>d</i> (D-H) / nm	<i>d</i> (H...A) / nm	<i>d</i> (D...A) / nm	∠DHA / (°)
<b>1</b>				
O5-H5B...O2	0.085	0.198	0.267 5(4)	137.8
<b>2</b>				
O3-H3...O2	0.082	0.189	0.270 2(5)	168.6

## 2 Results and discussion

### 2.1 Synthesis of the complex

As is known to all, ligand and solvent are factors of influencing the structures of the compounds. In the preparation of title complexes, the ligands 4-pyridin-4-yl-benzoic acid(4,4-Hpybz) and 4-pyridin-3-yl-benzoic acid (4,3-Hpybz) influence the coordination modes of the silver atom. Complex **1** is obtained by the reaction of AgBF<sub>4</sub>, PPh<sub>3</sub> and 4,4-Hpybz with ammonia water in 1 : 1 : 1 molar ratio in mixed solvent (CH<sub>3</sub>CN/H<sub>2</sub>O) generating an infinite chain structure. Complex **2** is obtained by the reaction of AgBF<sub>4</sub>, PPh<sub>3</sub> and 4,3-Hpybz with ammonia water in 1 : 1 : 1 molar ratio in mixed solvent (CH<sub>3</sub>OH/CH<sub>2</sub>Cl<sub>2</sub>) generating a binuclear structure. In the synthesis process, NH<sub>3</sub>·H<sub>2</sub>O is used as a critical material of deprotonation, which makes 4,4-Hpybz and 4,3-Hpybz transformed into 4,4-pybz and 4,3-pybz.

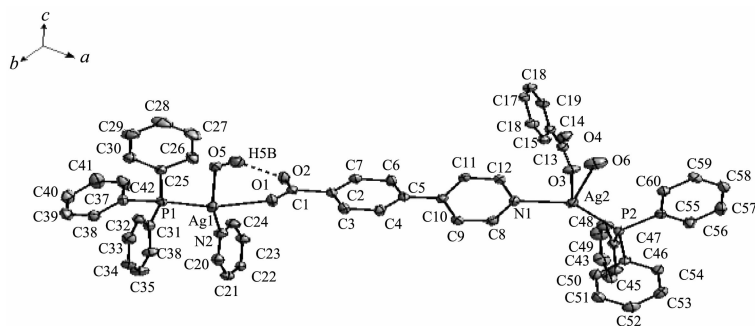
### 2.2 Infrared spectroscopy

The infrared spectra of complex **1~2** show the

absorptions around 1 434 cm<sup>-1</sup> are due to C-C stretch vibration of the phenyl rings and the middle absorptions around 3 048 cm<sup>-1</sup> are caused by C-H vibration of the phenyl rings. The absorptions of the COO<sup>-</sup> stretch vibration are around 1 388 cm<sup>-1</sup>.

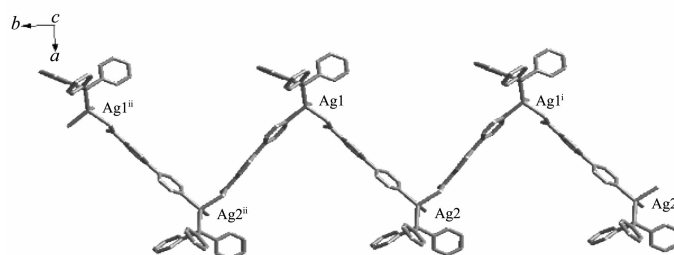
### 2.3 Description of the crystal structure

Single-crystal X-ray diffraction analysis reveals that **1** crystallizes in the Orthorhombic system with space group *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>. The asymmetric unit (Fig.1) is comprised of two Ag(I) ions, two PPh<sub>3</sub> ligands, two water ligands and two 4-pyridin-4-yl-benzoate (4,4-pybz) ligands, which is further linked by 4,4-pybz to generate a 1D coordination polymer (Fig.2). The metal which adopts four-coordinated mode, is uniquely bonded to one of oxygen atoms of a carboxyl fragment and a N atom from the pyridine ring fragment of the 4,4-pybz ligand establishing a distorted tetrahedral geometry about the metal, just as in the complex {[Ag(PPh<sub>3</sub>)<sub>2</sub>(bpp)](BF<sub>4</sub>)<sub>n</sub>}<sup>[16]</sup>. Ag-N (0.2325(3), 0.2253(3) nm) and Ag-P bond lengths(0.2368 6(10), 0.235 99(9)



A part of hydrogen atoms are omitted for clarity; Thermal ellipsoids drawn at the 30% probability level

Fig.1 Perspective view of complex **1**



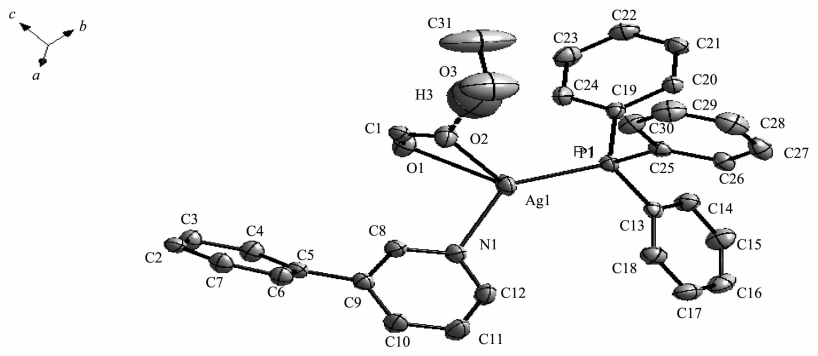
Hydrogen atoms are omitted for clarity; Symmetry code: <sup>i</sup>  $x, -1+y, z$ ; <sup>ii</sup>  $x, 1+y, z$

Fig.2 Linear chain of **1**

nm) are typical Ag(I)-N<sub>py</sub> and Ag(I)-P distances, respectively<sup>[17]</sup>. Ag(I)-N<sub>py</sub> bond distances are longer than that observed in [Ag(pycz)(H<sub>2</sub>O)]·3H<sub>2</sub>O (pycz=4-(4-pyridyl)benzoate) (0.214 5(3) nm)<sup>[18]</sup>. Moreover, the intramolecular O-H···O hydrogen bond is observed (O···O 0.267 5(4) nm, O-H···O 137.8°) in the complex **1**.

Complex **2** is a binuclear heteroleptic complex formed with distinctly soft Ag(I), PPh<sub>3</sub> and 4,3-pybz in 1:1:1 molar ratio. Each Ag is four-coordinated, surrounded by a P atom from a PPh<sub>3</sub> ligands and two chelating O atoms and a N atom from two 4,3-pybz

ligands (Fig.3). The ligand 4,3-pybz acts as a typical multiple dentate ligand to join Ag(I) atoms, just as in the complex [La<sub>2</sub>Cu<sub>2</sub>(ox)<sub>2</sub>L<sub>6</sub>]·4HL (HL=4-pyridin-4-ylbenzoic acid)<sup>[19]</sup>. The angles around Ag(I) ranging from 88.70(8)° to 136.69(6)° indicate that the geometry around Ag atom is distortedly tetrahedral. In complex **2**, Ag(I)-N<sub>py</sub> bond distance (0.234 2(2) nm) is longer than those in the complex [Ag<sub>3</sub>(CH<sub>3</sub>CN)<sub>3</sub>(L1)<sub>2</sub>Cl](BF<sub>4</sub>)<sub>2</sub>·3CH<sub>3</sub>CN (0.218 7(18) and 0.232 0(5) nm)<sup>[20]</sup>. The main structure of **2** links free CH<sub>3</sub>OH by hydrogen bonding interactions. The O-H···O hydrogen bond to link free



A part of hydrogen atoms are omitted for clarity; Thermal ellipsoids drawn at the 30% probability level

Fig.3 Asymmetric unit of complex **2**

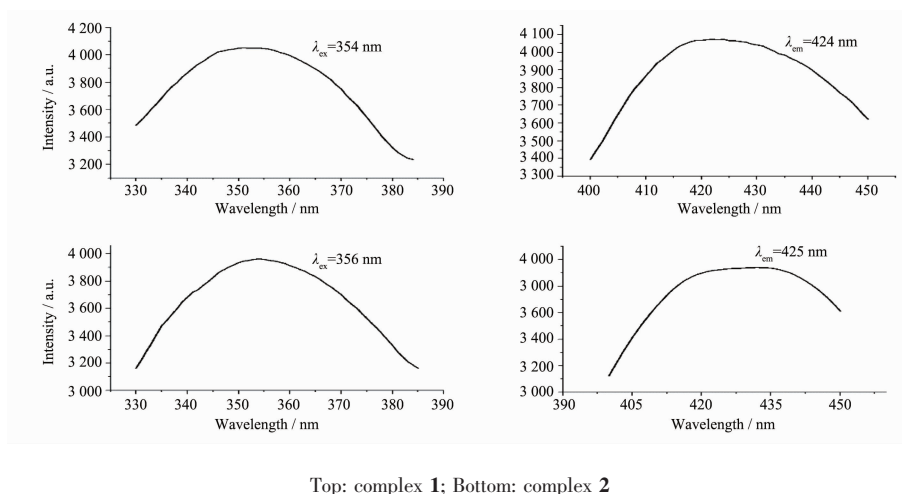


Fig.4 Solid-state excitation and emission spectra of **1~2** at 298 K

CH<sub>3</sub>OH is observed (O...O 0.270 2(5) nm, O-H...O 168.6°) in the complex **2**.

## 2.4 Fluorescence Spectrum

The luminescent excitation and emission spectra of complexes **1~2** and 4,4-Hpybz, 4,3-Hpybz ligand in the solid state at room temperature are obtained. The emission peak of PPh<sub>3</sub> is at 402 nm ( $\lambda_{\text{ex}}=372$  nm)<sup>[21]</sup>. In the fluorescence emission spectra of 4,4-Hpybz ligand, the emission peaks are found at 395 nm ( $\lambda_{\text{ex}}=322$  nm). The 4,3-Hpybz ligand exhibits fluorescence signal centered at 417 nm with an excitation maximum at 341 nm. When excited at 354 nm, a fluorescence emission peak of complex **1** is found at 424 nm. The complex **2** exhibits fluorescence signal centered at 356 nm with an excitation maximum at 425 nm (Fig.4). The red-shift of emission peaks of **1~2** are derived from ligand-centered  $\pi-\pi^*$  transition.

## 3 Conclusions

Two new Ag(I) complexes of phosphine-containing ligands, namely [Ag<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>(4,4-pybz)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>]<sub>n</sub> (**1**) and [Ag<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>(4,3-pybz)<sub>2</sub>]·2CH<sub>3</sub>OH (**2**), have been synthesized and characterized by elemental analysis, IR, X-ray diffraction and fluorescence spectra. Structure analyses show that, the Ag atoms are bridged by pyridylbenzoate-containing ligand to form infinite chain structure in **1**. Two Ag atoms are bridged by two 4-pyridin-3-yl-benzoate ligands to form binuclear complex in **2**. The luminescent spectra show

that the origin of these emissions all involves emissive state derived from ligand centered  $\pi-\pi^*$  transition. We hope our results could offer new strategy for the characterization and design of coordination polymers.

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