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Syntheses, crystal structure and luminescent properties of cadmium complexes based on 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine

Sheng-Gui Liu^{a,*}, Lin-Ping Zhang^b, Jian Liu^c, Wen-Yi Su^a, Xiao-Bo Shi^a

^a School of Chemistry Science and Technology, Zhanjiang Normal University, Zhanjiang 524048, People's Republic of China
^b Key Laboratory of Science & Technology of Eco-Textile (Ministry of Education), Donghua University, Shanghai 201620, People's Republic of China
^c School of New Energy Science and Engineering, Xinyu University, Xinyu 338004, People's Republic of China

HIGHLIGHTS

- Two new cadmium complexes based on 2,6-bis(1-phenylbenzimidazol-2yl)pyridine were synthesized.
- The two cadmium complexes were characterized by single X-ray analyses.
- The two cadmium complexes emit blue luminescence in solid state and in solution.
- ► The luminescence of the two cadmium complexes are predominantly from the ligand π^{*} → π transitions fluorescence.

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Two cadmium complexes based on 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine (bpbp), were synthesized and characterized by X-ray single crystal structure analyses. Both complexes emit blue luminescence in solid state and in solution.



ABSTRACT

Two cadmium complexes, Cd(bpbp)Cl₂ (complex **1**) and [Cd(bpbp)₂](ClO₄)₂ (complex **2**), based on 2,6bis(1-phenylbenzimidazol-2-yl)pyridine (bpbp), were synthesized and characterized by X-ray single crystal structure analyses. For complex **1**: crystal system, monoclinic, space group, C2/c, a = 27.427(3) Å, b = 13.4495(15) Å, c = 14.8381(17) Å, $\beta = 106.635(2)^{\circ}$, V = 5244.4(10) Å³, Z = 8. It is a neutral complex. The Cd(II) ion distorted trigonal bipyramidal geometry is five-coordinated by three nitrogen atoms from ligand (bpbp) and two chlorine ions. For complex **2**: crystal system, triclinic, space group, P-1, a = 13.4791(15) Å, b = 13.8506(16) Å, c = 16.5839(19) Å, $\alpha = 94.202(2)^{\circ}$, $\beta = 106.948(2)^{\circ}$, $\gamma = 94.872(2)^{\circ}$, V = 2935.3(6) Å³, Z = 2. It is an ionic complex. The Cd(II) ion octahedral geometry is six-coordinated by six nitrogen atoms from two ligand (bpbp). Both complexes emit blue luminescence with emission peaks at 420 and 430 nm in solid state and with emission peaks at 415 and 425 nm in DMF solution. In complex **1** absorption spectra, there is not only the free ligand absorption peak at 310 nm, but also shows strong Cd–Cl charge transfer peak at 350 nm in DMF solution.

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^{*} Corresponding author. Tel.: +86 759 3183176; fax: +86 759 3183510. *E-mail address:* lsgui@sohu.com (S.-G. Liu).

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Introduction

In recent years, luminescent metal complexes have attracted an increasing attention in many areas of chemistry, biology, medical and material science [1–9]. Their functions as emitter material in luminescence devices and luminescent chemosensor are particularly prominent. Luminescent chemosensor have the advantage of possessing high sensitivity and selectivity, as well as providing online and real-time analysis [10-12]. One of simple strategy for designing luminescent coordination compounds is to organize d^{10} electronic configuration metal ions with chromophore ligand. The origins of emission arise from the transitions of $\pi^* - \pi$ within ligand. This emission could be efficiently enhanced in coordination complex due to increasing of the rigidity of the ligand and reducing of energy loss by radiationless thermal vibrations [13,14]. The design and synthesis of desired structure luminescent complex is a challenge because the assembly of metal ion with ligand is sensitive to the delicate synthetic conditions [15].

2,6-Bis(benzimidazol-2-yl)pyridine usually functions as tridentate ligand, but the five N atoms all can coordinate to metal center ion, even if with the same central ion, different counter-anion involved, different pH value, will result in different complex [16.17]. The author investigated the syntheses, structures and their blue luminescent properties of Zn(II). Cd(II). In(III) dichloride complexes based on 2.6-bis(benzimidazolyl)pyridine [18,19], 2.6-Bis-(benzimidazol-2-yl)pyridine can easily be tailored at NH group [20]. The substitution of the N-H bond of 2,6-bis(benzimidazolyl)pyridine will change their many properties (e.g. acid-base degree, solubility and photophysical properties). The luminescence analysis is favorable to trace analysis for such environmental toxic ions as cadmium. It is necessary to investigate that how cadmium ions coordinate to luminescent chromophore so as to set up luminescence analysis method of cadmium. As part of our continuing studies, herein we synthesized two cadmium complexes based on 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine (bpbp) to investigate their luminescent properties.

Experimental

General

o-Phenylenediamine, pyridine-2,6-dicaboxyl acid and bromobenzene were purchased from Shanghai Aladdin Reagent Company. All the chemicals and solvents used were analytically pure and without further purification. The analyses (C, H and N) were made on a Perkin–Elmer 240C elemental analyzer. ¹H NMR spectroscopic measurements were carried out on a Bruker AM-300 NMR spectrometer, using TMS (SiMe₄) as an internal reference. The solid infared spectra (IR) were obtained from a Bruker IFS66V vacuumtype FT-IR spectrophotometer by using KBr pellets. The UV absorption spectra were recorded on a model UV-240 spectrophotometer (Shimadzu, Japan). Fluorescence measurements were performed on a Model RF-5 spectrofluorimeter (Shimadzu, Japan). 2,6-bis(1phenylbenzimidazol-2-vl)pyridine was synthesized according to the literature [21,22]. This ligand structure was confirmed by element analysis IR, and ¹H NMR. Element analysis for C₃₁H₂₁N₅, found: C, 80.22; H, 4.67; N, 15.10, calculated: C, 80.32; H, 4.57; N, 15.11. Selected IR data (KBr, cm⁻¹): 3417, 1674, 1588, 1503, 1439, 1392, 1336, 1250, 1147, 1072, 1002, 933, 874, 830, 744, 685, 575, 541, 502, 421 and ¹H NMR: (CDCl₃): 6.99 (d, 4H), 7.184–7.367(m, 12H), 7.876 (t, 3H), 8.08 (d, 2H).

Synthesis of [Cd(bpbp)Cl₂] (1)

To an alcohol solution (10 ml) of CdCl₂ (367 mg, 2.0 mmol) was added an alcohol solution (20 ml) of bpbp (927 mg, 2.0 mmol). The reaction mixture was stirred for 30 min at room temperature. White crystals were obtained from room evaporation of alcohol, yield 993 mg (76%). Anal. Calcd. for $C_{31}H_{21}C_{12}N_5Cd$ ([Cd(bpbp)Cl₂]): C, 57.56%; H, 3.27%; N, 10.83%. Found: C, 57.29%; H, 3.41%; N, 10.73%. Selected IR data (KBr, cm⁻¹): 3448, 1658, 1495, 1443, 1378, 1255, 1106, 1061, 1002, 877, 808, 755, 679.

Synthesis of $[Cd(bpbp)_2](ClO_4)_2 \cdot CH_3CH_2OH(2)$

To an alcohol solution (10 ml) of Cd(ClO₄)- $6H_2O$ (420 mg, 2.0 mmol) was added an alcohol solution (20 ml) of bpbp (993 mg, 2.0 mmol). The reaction mixture was stirred for 30 min at room temperature. On recrystallization from ethanol, white crystals were obtained. Yield: 987 mg (79%). Anal. Calcd. for C₆₄H₄₈N₁₀Cl₂O₉Cd ([Cd(bpbp)₂](ClO₄)₂·CH₃CH₂OH): C, 59.85, H, 3.77% N, 10.90%: C, 59.49%, 3.81, N, 10.60%. Selected IR data (KBr, cm⁻¹): 3473, 3065, 1594, 1502, 1460, 1411, 1378, 1336, 1293, 1200, 1094, 1002, 877, 818, 750, 696, 617.

X-ray crystallography

Single crystal structure determination complexes were performed on a Bruker SMART APEX CCD diffractometer equipped with a normal focus, 3 kW sealed tube X-ray source and graphite monochromated Mo–K $_{\alpha}$ radiation (λ = 0.71073 Å) at 173 K, operating at 50 kV and 30 mA. The structures were solved by direct methods by using program SHELXTL. Absorption correction adopted semi-empirical from equivalents. Fourier difference techniques, and refined by full-matrix least-squares. All non-hydrogen atoms in both structures were refined anisotropic displacement parameters. All hydrogen atoms were theoretically added. The crystal data are summarized in Table 1. Selected bond lengths and angles for complex **1** are listed in Table 2, complex **2** listed in Table 3.

Table 1				
Crystallographic data	for comp	olex 1 an	d complex	2.

	Complex 1	Complex 2
Formula	C ₃₁ H ₂₁ CdCl ₂ N ₅	C ₆₄ H ₄₇ CdCl ₂ N ₁₀ O ₉
Formula weight	646.83	1283.42
Crystal system	Monoclinic	Triclinic
Space group	C2/c	P-1
a/Å	27.427(3)	13.4791(15)
b/Å	13.4495(15)	13.8506(16)
c/Å	14.8381(17	16.5839(19)
α/°	90	94.202(2)
βĮ°	106.635(2)	106.948(2)
$\gamma/^{\circ}$	90	94.872(2)
V/Å ³	5244.4(10)	2935.3(6)
Ζ	8	2
Density calc.mg.m ⁻³	1.638	1.452
Absorption coefficient (mm ⁻¹)	1.068	0.531
F(000)	2592	1332.0
Crystal size	$0.12 \times 0.17 \times 0.47$	$0.12 \times 0.15 \times 0.18$
Theta range for data collection	1.5, 27.0	1.59, 27.08
Index ranges	$-24 \leqslant h \leqslant 35$	$-17 \leqslant h \leqslant 17$
	$-14 \leqslant k \leqslant 17$	$-17 \leqslant k \leqslant 17$
	$-17 \leqslant l \leqslant 18$	$-21 \leqslant l \leqslant 21$
Nref, Npar	5763, 352	10333, 829
Tot., Uniq. Data, <i>R</i> (int)	15379, 5763, 0.032	21182, 10218, 0.025
GOF on F ²	1.09	1.08
Max. and min. transmission	0.6336, 0.8825	0.909, 0.938
Observed data $[I > 2.0 \text{ sigma}(I)]$	4407	8524
R indices (all, data) R ₁ , wR ₂	$R_1 = 0.0327$	$R_1 = 0.0269$
	$wR_2 = 0.0867$	$wR_2 = 0.2873$
Largest diff. peak and hole $e.Å^{-3}$	-0.46, 0.66	-0.99, 1.29

Table 2

Selected bond lengths (Å) and angles (°) for complex **1**.

		1	
Cd1–Cl2	2.4412(8)	Cd1-Cl1	2.4635(8)
Cd1-N1	2.332(2)	Cd1-N3	2.415(2)
Cd1-N4	2.311(3)	N1-C1	1.382(4)
N1-C13	1.327(4)	N3-C14	1.342(4)
N3-C18	1.337(4)	N4-C19	1.321(4)
N4-C31	1.386(4)		
Cl2-Cd1-Cl1	112.59(3)	Cl2-Cd1-N1	110.63(6)
Cl2-Cd1-N3	107.20(6)	Cl2-Cd1-N4	99.46(6)
Cl1-Cd1-N1	99.81(6)	Cl1-Cd1-N3	140.09(6)
Cl1-Cd1-N4	102.28(6)	N1-Cd1-N3	67.98(8)
N1-Cd1-N4	131.77(8)	N3-Cd1-N4	67.54(8)

Table 3

Selected bond lengths (Å) and angles (°) for complex 2.

Cd1-N9	2.290(6)	Cd1-N4	2.305(6)
Cd1–N8	2.339(5)	Cd1-N3	2.361(6)
Cd1-N6	2.385(6)	Cd1-N1	2.398(6)
N9-Cd1-N4	121.4(2)	N9-Cd1-N8	69.9(2)
N4-Cd1-N8	116.04(19)	N9-Cd1-N3	111.6(2)
N4-Cd1-N3	69.9(2)	N8-Cd1-N3.	172.5(2)
N9-Cd1-N6	138.8(2)	N4-Cd1-N6	82.5(2)
N8-Cd1-N6	69.38(19)	N3-Cd1-N6	107.99(19)
N9-Cd1-N1	80.0(2)	N4-Cd1-N1	138.5(2)
N8-Cd1-N1	104.4(2)	N3-Cd1-N1	69.1(2)
N6-Cd1-N1	104.5(2)		

Result and discussion

Synthesis, IR for complexes

Reaction of 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine (bpbp) with CdCl₂ in alcohol solution resulted in complex **1**. Reaction of 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine with Cd(ClO₄)₂ in ethanol produce complex **2**. It is important to choose anion of metal salt in the formation of complex in this coordination reaction. Different anions inorganic metal salt can produce different complex. When ligand (bpbp) react with CdCl₂, it will result in neutral complex Cd(bpbp)Cl₂, although bpbp is two equivalent of CdCl₂. If bpbp react with Cd(ClO₄)₂, it will result in ionic complex $[Cd(bpbp)_2](ClO_4)_2$ since ClO_4^- act as counter anions. However, 2,6-bis(benzimidazol-2-yl)pyridine (containing two NH groups) react with Cd(ClO_4)_2, if in the present of triethylamine glycerol solution, 2,6-bis(benzimidazol-2-yl)pyridine will deprotonate and result in neutral Cd(II) complex Cd(bbp)_2, whereas, it will form ionic complex $[Cd(bbp)_2](ClO_4)_2$, if in alcohol solution absence of triethylamine [23,24]. In the IR spectra of free ligand, there are two peaks at 1678 and 1588 cm⁻¹, but there is only a strong peak at 1653 cm⁻¹ in complex **1** and 1592 cm⁻¹ in complex **2**, which shows that coordinated Cd(II) will obviously affect skeletal vibration of the aromatic rings. In addition, in complex **2**, there is a strong peak at 1090 cm⁻¹, which shows ClO₄⁻ in this complex.

Crystal structures

The ORTEP drawing of complex **1** is shown in Fig. 1. The central Cd(II) ion is five-coordinated and surrounded by a N₃Cl₂ environment, adopting a distorted trigonal bipyramidal geometry. The ligand (bpbp) affords three N atoms to coordinate to Cd(II) and two chloride ions lie each side of the planar pyridine ring. The bond length of Cd1-N4, Cd1-N1, Cd1-N3 are respectively 2.311(2), 2.332(2), 2.415(2) Å. The average bond length of Cd-N is 2.359 Å. The bond length of Cd_1-Cl_1 and Cd_1-Cl_2 are 2.4634(8), 2.4412(8) Å, respectively. The average bond length of Cd–Cl is 2.4513 Å. The bond angles of N_3 -Cd₁- N_4 , N_3 -Cd₁- N_1 , N_3 -Cd₁-Cl₁, N₃-Cd₁-Cl₂ are 67.54(8), 67.98(8)°, 140.10(6), 107.20(6)°. The three atoms N₁, N₃, N₄ chelate center metal cadmium to result in the formation of a coplanar of two benzimidazole rings and the pyridine ring of the ligand, but steric strain make the center cadmium ion deviate from this plane (mean deviation = 0.4841°). The two substitute phenyl rings are inclined with the two benzimidazole rings, respectively. The dihedral angle between substituted phenyl ring and benzimidazole ring are 56.7° for phenyl ring C₇-C₁₂, 58.9° for phenyl ring C₂₀-C₂₅. The crystal packing of complex 1 is shown in Fig. 2. Two adjacent molecules form a dimer by two point-to-face C–H... π weak interactions between substituted benzene protons and substituted benzene ring (the distance of hydrogen atom to the center of substituted benzene is 2.997 Å), substituted benzene protons and benzimidazole benzene ring (the distance of hydrogen atom to the center of benzimidazole benzene



Fig. 1. Molecular structure of complex 1. Hydrogen atoms have been omitted for clarity (ORTEP, 50% ellipsoids).



Fig. 2. The crystal packing of complex 1 (the broken line stand for weak interaction).



Fig. 3. Molecular structure of $[Cd(bpbp)_2]^{2*}$ cation. All hydrogen atoms of bpbp, alcohol solvent molecule and counter anions have been omitted for clarity (ORTEP, 50% ellipsoids).

is 3.176 Å), and C–H···Cl weak interaction (the distance of H···Cl is 2.832 Å). These dimers form a chain through benzimidazole benzene ring π - π weak interactions along *a* axis (the distance of two benzene ring center is 3.541 Å).

The ORTEP drawing for complex **2** with atom numbering is shown in Fig. 3. The center Cd(II) ion has N_6 coordination sphere, being bound by two bpbp ligand. In every ligand, the two benz-imidazole rings and center pyridine ring form a good planar (mean deviation = 0.2364° , 1.38°), the dihedron between the two ligand

planar is 14.0°, which show Cd(II) atom in a greatly distorted octahedral geometry. The dihedral angle between substituted phenyl ring and benzimidazole ring are 62.8° for phenyl ring C₈-C₁₃, 87.9° for phenyl ring C_{22} - C_{26} , 44.2° for phenyl ring C_{40} - C_{46} , 59.1° for phenyl ring C_{55} - C_{60} . The bond length of Cd_1 - N_9 , 2.290(6), Cd1-N4 2.305(6), Cd1-N8, 2.339(5), Cd1-N3 2.361(6), Cd1-N6, 2.385(6), Cd_1-N_1 2.398(6). The average bond length of Cd-N is 2346 Å. which fall into the range of the reported Cd-N distances [25]. The crystal packing of complex 2 is shown in Fig. 4, $ClO_4^$ counter-ions are included in the crystal lattice not only by electrostatic interaction between perchlorate ions and coordinated cations $[Cd(bpbp)_2]^{2+}$, but also by C-H···O weak hydrogen bonds. (The distance: $C_9-H_{9A}\cdots O5^i = 3.257$, $C_{16}-H_{16A}\cdots O_7 = 3.32$, $C_{45} H_{45A} \cdots O_1^{ii} = 3.348, C_{51} - H_{51A} \cdots O_{11}^{iii} = 3.26, C_{60} - H_{60A} \cdots O_{13} = 3.219,$ $C_{64}-H_{64A}\cdots O_{10}^{iv} = 3.50 \text{ Å}$, Symmetry codes: i: 3 - x; 3 - y, 3 - z; ii: 2 - x; 1 - y, 2 - z; iii: 2 - x, 2 - y, 3 - z; iv: 2 - x, 3 - y, 3 - z).

Photophysical properties

The UV–Vis absorption and liquid luminescence spectra were recorded at a concentration of 1.0×10^{-5} mol/L in DMF at room temperature. The UV–Vis absorption spectra of free ligand and complexes **1** and **2** are shown in Fig. 5. The ligand and both complexes all have absorption in the range of 270–390 nm. The maximal absorption are all at about 310 nm, which are blue shift by 20 nm than cadmium 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine dichloride complex, which can be assigned to intraligand $\pi \rightarrow \pi^*$ transitions. There is one more peak observed at 350 nm for complex **1**, which probably arise from metal-chloride charge transfer since the ligand and complex **2** do not show this peak, this behavior were also observed in cadmium, indium 2, 6-bis(benzimidazol-2-yl)pyridine dichloride complexes [19].

The free ligand and complexes **1** and **2** all display blue luminescence in solid state (Fig. 6). The free ligand 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine is also a blue luminescent compound similar to 2,6-bis(benzimidazol-2-yl)pyridine[19]. The complexes all show broad luminescent bands since their luminescence are predominantly from the ligand $\pi^* \rightarrow \pi$ transitions fluorescence. Under excitation of 370 nm wavelength light, the complexes **1** and **2** exhi-



Fig. 4. The packing diagram of the complex 2 viewing along b axis (the broken lines showing C-H ·· O weak hydrogen bonds).



Fig. 5. UV-visible absorption spectra of bpbp, complexes ${\bf 1}$ and ${\bf 2}$ in DMF solution.



Fig. 6. Fluorescence spectra of the ligand, complexes 1 and 2 in solid state $(\lambda_{ex}=370~\text{nm}).$

bit blue luminescent with the maximal peaks at 420 and 430 nm which is 20, 10 nm red-shift compared to the free ligand (440 nm) respectively. Notably, it is observed that the luminescent intensity of two complexes increase than free ligand because of effective increase of rigidity of ligand after coordination to cadmium ion. The luminescent intensity of complex **2** is stronger than complex **1** is due to that there are two luminescent ligand (bpbp) molecules in complex **2** and one in complex **1**. The red shift of complex **1** and **2** than free ligand (bpbp) may be caused by the differences of the coplanar of two benzimidazole rings and center pyridine rings of ligands and the dihedral angle between substituted phenyl rings and benzimidazole rings in the two complexes in solid state. In DMF solution, under excitation of 330 nm wavelength light, the free

ligand, complexes **1** and **2** also exhibit luminescence with the maximal peaks at 380, 415 and 425 nm, respectively (Fig. 7). In comparison with the solid state, there are only slight 5 nm blue-shifts for the complexes **1** and **2** but 60 nm blue-shift for free ligand, which show DMF solvent affect luminescence of free ligand much greatly than complexes **1** and **2** (see Fig. 7).

Conclusion

Two cadmium complexes, Cd(bpbp)Cl₂ and [Cd(bpbp)₂](ClO₄)₂, based on 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine were synthesized and characterized by X-ray single crystal structure analy-



Fig. 7. Fluorescence spectra of the ligand, complexes 1 and 2 in DMF solution $(\lambda_{ex}$ = 330 nm).

ses. When CdCl₂ was chosen as cadmium source, it will result in neutral complex Cd(bpbp)Cl₂, but Cd(ClO₄)₂ instead of CdCl₂ as cadmium source, it will produce ionic complex, [Cd(bpbp)₂](ClO₄)₂. In complex Cd(bpbp)Cl₂, Cd(II) ion distorted trigonal bipyramidal geometry is coordinated by three nitrogen atoms from 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine and two chlorine ions. In complex [Cd(bpbp)₂](ClO₄)₂, the Cd(II) ion octahedral geometry is coordinated by six nitrogen atoms from two 2,6-bis(1-phenylbenzimidazol-2-yl)pyridine molecules. In complex Cd(bpbp)Cl₂ absorption spectra, there is not only the free ligand absorption peak at 310 nm, but also shows strong Cd–Cl charge transfer peak at 350 nm. The two complexes exhibit blue luminescence in the solid state and solution.

Acknowledgments

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Appendix A

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 873909 for complex **1** and no. 873910 for complex **2**. Copies of this information may be obtained from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (www: http://www.ccdc. cam.ac.uk).

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