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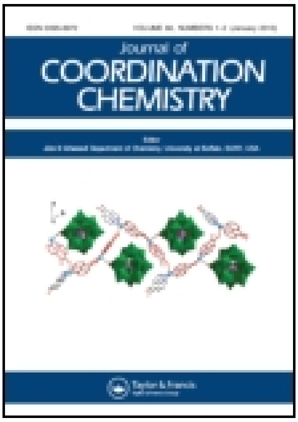

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Syntheses, structures, and luminescence of Co(II), Zn(II), Cd(II) complexes with 1-(2-furylmethylene)-2-(2-furyl)benzimidazole

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Syntheses, structures, and luminescence of Co(II), Zn(II), Cd(II) complexes with 1-(2-furylmethylene)-2-(2-furyl)benzimidazole

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The ligand 1-(2-furylmethylene)-2-(2-furyl)benzimidazole (**L**), CoL_2Cl_2 (**1**), $ZnL_2(NO_3)_2$ (**2**), $CdL_2(CH_3COO)_2$ (**3**), and $CoL_2(NO_3)_2$ (**4**) were synthesized in excellent yields using a simple and convenient method. These complexes were characterized by UV–vis spectra, IR spectra, elemental analysis, and Thermogravimetric analysis. Single crystals of **1–3** were obtained and their structures were determined by X-ray structure analysis. Luminescence of these complexes was also investigated.

Keywords: Benzimidazole; Furan heterocyclic; Crystal structures; π–π interaction; Luminescence properties

1. Introduction

Benzimidazole and its derivatives have wide applications in organic chemistry, biochemistry, and pharmacy. Benzimidazole derivatives exhibit significant activities, including antiviral, antihypertensive, antifungal, and anticancer [1–3]. In addition to their biological importance, stable complexes formed by benzimidazoles with various transition metals are an important subject in coordination chemistry, crystal engineering, medicine, and materials science owing to their structural motifs and properties, such as catalysis, luminescence, antivirus activity, and electrochemical properties [4–7]. Numerous compounds with furan ring also have interesting properties [8]. Herein, we synthesized 1-(2-furylmethylene)-2-(2-furyl)benzimidazole (**L**) in high yields by one-step condensation of *o*-phenylenediamine with furfural in ethanol at room temperature under stirring without using any catalyst. Metal complexes with Co(II) (**1**, **4**), Zn(II) (**2**), Cd(II) (**3**) were also synthesized and characterized. The crystal structures of **1**, **2**, and **3** have been established by X-ray diffraction. All the complexes were characterized by luminescence analysis. We found that metal complexes show efficient luminescence related to their structures.

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2. Experimental

2.1. *Materials and physical measurements*

All chemicals were of reagent grade and used without purification except furfural which was freshly distilled prior to use. C, H, and N contents were determined on an Elemental Vario EL III elemental analyzer. Infrared spectra were recorded using KBr from 4000–400 cm^{-1} employing a Bruker Equinox 55 spectrometer. Thermogravimetric analysis (TGA) was performed on a Netzsch STA 449C thermal analyzer instrument in flowing nitrogen at a heating rate of 10 °C min^{-1}. Fluorescence spectra were performed with a Hitachi F-4500 fluorescence spectrofluorometer. The UV–vis spectra were obtained with a Shimadzu UV-1800 spectrophotometer at 25 °C.

2.2. *Synthesis*

2.2.1. Ligand synthesis. A solution of o-phenylenediamine (0.5400 g, 5 mM) and furfural (0.89 mL, 10 mM) in ethanol (50 mL) was stirred for 2 h at room temperature. After the reaction, the volume was reduced to 10 cm^3 by rotary evaporation, then placed in the refrigerator at −10 °C. An yellow precipitate was obtained after 24 h which was collected by filtration, washed with absolute ethanol, and dried in vacuo. Yield: 88%. Anal. Calcd for $C_{16}N_2O_2H_{12}$ (%): C, 72.69; H, 4.48; N, 10.69. Found: C, 72.72; H, 4.58; N, 10.60. IR (KBr, cm^{-1}): 1623(s), 1507(s), 1473(m), 1392(m), 1276(m), 1149(m), 1015(s), 929(s), 883 (s), 742(vs), and 593(s).

2.2.2. Synthesis of $Co(C_{16}N_2O_2H_{12})_2Cl_2$ (1). A solution of L (0.1320 g, 5 mM) in ethanol (25 mL) was added to a stirred solution of $CoCl_2{\cdot}6H_2O$ (0.5948 g, 2.5 mM) in ethanol (25 mL). The mixture was stirred at room temperature for 2 h. The precipitated complex was collected by filtration and washed with ethanol. Yield: 85%. Anal. Calcd for $C_{32}N_4O_4H_{24}CoCl_2$ (%): C, 58.33; H, 3.25; N, 8.47. Found (%): C, 58.37; H, 3.65; N, 8.51. IR (KBr, cm^{-1}): 1612(m), 1506(m), 1454(s), 1415(s), 1341(m), 1230(m), 1148(m), 1074(m), 1015(s), 909(w), 832(w), 752(vs), and 594(w). Single crystals suitable for X-ray analysis were obtained by slow evaporation in methanol at room temperature.

2.2.3. Synthesis of $Zn(C_{16}N_2O_2H_{12})_2(NO_3)_2$ (2). The procedure was the same as that for **1** except that $CoCl_2{\cdot}6H_2O$ was replaced by $Zn(NO_3)_2{\cdot}6H_2O$. Yield: 90%. Anal. Calcd for $C_{32}N_6O_{10}H_{24}Zn$ (%): C, 53.56; H, 3.29; N, 11.72. Found (%): C, 53.53; H, 3.35; N, 11.71. IR (KBr, cm^{-1}): 1615(m), 1470(s), 1417(m), 1358(m), 1307(vs), 1230(m), 1175 (m), 1149(m), 1074(m), 1019(s), 911(m), 884(w), 836(w), 753(vs), 593(w), 564(w), and 447(w). Crystals of **2** were obtained after recrystallization from acetonitrile.

2.2.4. Synthesis of $Cd(C_{16}N_2O_2H_{12})_2(CH_3COO)_2$ (3). Complex **3** was synthesized using the same method as for **1**, with substitution of $Cd(Ac)_2{\cdot}2H_2O$ for $CoCl_2{\cdot}6H_2O$. Yield: 80%. Anal. Calcd for $C_{36}N_4O_8H_{30}Cd$ (%): C, 56.99; H, 3.90; N, 6.93. Found (%): C, 56.96; H, 3.96; N, 7.38. IR (KBr, cm^{-1}): 3106(w), 1615(s), 1560(w), 1509(m), 1462(m), 1421(s), 1339(w), 1225(w), 1153(w), 1076(w), 1018(m), 919(w), 839(m), 747(s), 671(m), and 595(w). Single crystals suitable for X-ray structure analysis were grown by slow evaporation of the mother-liquor.

2.2.5. Synthesis of $Co(C_{16}N_2O_2H_{12})_2(NO_3)_2$ (4). A procedure similar to that described for the synthesis of **1** was carried out with **L** and $Co(NO_3)_2{\cdot}6H_2O$. Yield: 87%. Anal. Calcd for $C_{32}N_6O_{10}H_{24}Co$ (%): C, 53.93; H, 3.22; N, 11.79. Found: C, 54.01; H, 3.38; N, 11.82. IR (KBr, cm^{-1}): 3111(w), 1473(vs), 1384(vs), 1307(vs), 1298(vs), 1228(m), 1154(m), 1020(s), 904(m), 754(vs), and 592(m).

2.3. *X-ray crystal structure determinations*

Crystallographic data were obtained at 296 K with a Bruker Smart CCD diffractometer with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å). Absorption corrections were applied using SADABS. The structures were solved by direct methods and refined on F^2 using full-matrix least squares with SHELXTL-97. Details of the data collection and refinement for **1–3** are summarized in table 1. The selected bond lengths (Å) and angles (°) are listed in table 2.

Table 1. Crystal data and structure refinement details for **1–3**.

Complex	**1**	**2**	**3**
Empirical formula	$C_{32}H_{24}N_4O_4Cl_2Co$	$C_{32}H_{24}N_6O_{10}Zn$	$C_{36}H_{30}N_4O_8Cd$
Formula weight	658.38	717.94	759.04
Temperature (K)	296(2)	296(2)	296(2)
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	*P*21/*c*	*C*2/*c*	*C*2/*c*
Unit cell dimensions (Å, °)			
a	17.453(2)	18.353(2)	16.924(3)
b	9.7735(11)	10.6297(13)	11.287(2)
c	18.139(2)	17.908(2)	17.693(3)
β	106.511(3)	117.406(2)	99.281(4)
Volume ($Å^3$), *Z*	2966.5(6), 4	3101.5(6), 4	3335.5(10), 4
Calculated density ($Mg\,m^{-3}$)	1.474	1.538	1.512
Absorption coefficient (mm^{-1})	0.803	0.862	0.714
F(000)	1348	1472	1544
θ range (°)	1.22−25.10	2.29−25.10	2.18−26.00
Limiting indices	$-11 \leqslant h \leqslant 20$	$-20 \leqslant h \leqslant 21$	$-20 \leqslant h \leqslant 16$
	$-11 \leqslant k \leqslant 11$	$-12 \leqslant k \leqslant 10$	$-13 \leqslant k \leqslant 13$
	$-21 \leqslant l \leqslant 21$	$-19 \leqslant l \leqslant 21$	$-12 \leqslant l \leqslant 21$
Reflections collected/unique	14,662/5279	7673/2762	8605/3249
R_{int}	0.0721	0.0417	0.0518
Data/restraints/parameters	5279/20/388	2762/3/222	3249/40/222
Goodness-of-fit on F^2	1.060	1.030	1.035
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0706$, $wR_2 = 0.1668$	$R_1 = 0.0450$, $wR_2 = 0.1050$	$R_1 = 0.0749$, $wR_2 = 0.1902$
R indices (all data)	$R_1 = 0.1213$, $wR_2 = 0.1947$	$R_1 = 0.0758$, $wR_2 = 0.1234$	$R_1 = 0.1103$, $wR_2 = 0.2144$
Largest diff. peak and hole ($e\,Å^{-3}$)	0.977 and −0.500	0.299 and −0.281	0.931 and −0.742

Table 2. Selected bond lengths (Å) and angles (°) for **1–3**.

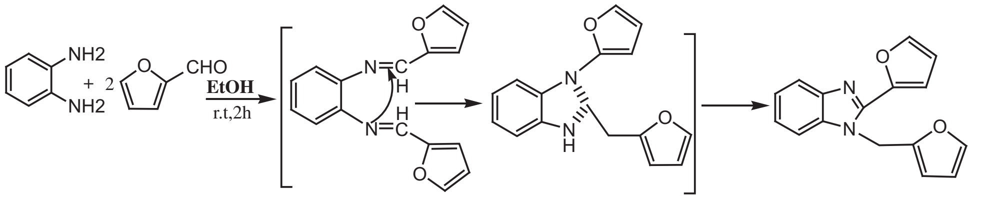
1			
Co(1)–N(2)	2.041(4)	Co(1)–N(1)	2.042(5)
Co(1)–Cl(2)	2.2648(19)	Co(1)–Cl(1)	2.2668(17)
N(2)–Co(1)–N(1)	108.78(19)	N(2)–Co(1)–Cl(2)	106.32(14)
N(1)–Co(1)–Cl(2)	114.40(14)	N(2)–Co(1)–Cl(1)	115.25(14)
N(1)–Co(1)–Cl(1)	106.52(14)	Cl(2)–Co(1)–Cl(1)	105.79(7)
2			
Zn(1)–O(1)#1	1.997(2)	Zn(1)–O(1)	1.997(2)
Zn(1)–N(1)	2.014(3)	Zn(1)–N(1)#1	2.014(3)
O(1)#1–Zn(1)–O(1)	122.72(15)	O(1)#1–Zn(1)–N(1)	107.02(11)
O(1)–Zn(1)–N(1)	98.81(11)	O(1)#1–Zn(1)–N(1)#1	98.81(11)
O(1)–Zn(1)–N(1)#1	107.02(11)	N(1)–Zn(1)–N(1)#1	124.56(15)
3			
Cd(1)–N(1)	2.279(6)	Cd(1)–N(1)#1	2.279(6)
Cd(1)–O(4)#1	2.313(6)	Cd(1)–O(4)	2.313(6)
Cd(1)–O(3)	2.439(6)	Cd(1)–O(3)#1	2.439(6)
N(1)–Cd(1)–N(1)#1	105.0(3)	N(1)–Cd(1)–O(4)#1	106.5(2)
O(4)#1–Cd(1)–O(4)	135.7(4)	O(4)#1–Cd(1)–O(3)	54.4(2)
N(1)–Cd(1)–O(3)#1	154.3(2)	O(4)#1–Cd(1)–O(3)#1	93.5(2)
O(4)–Cd(1)–O(3)#1	54.4(2)	O(3)–Cd(1)–O(3)#1	92.0(3)

Symmetry code: (#1) $-x$, y, $-z+1/2$.

3. Results and discussion

3.1. *Synthesis*

Traditionally, 1-(2-furylmethylene)-2-(2-furyl)benzimidazole has been synthesized via one-pot condensation of *o*-phenylenediamines with furfural in the presence of different catalysts, such as bismuth triflate [9], trifluoroacetic acid [10], Amberlite IR-120 [11], Al_2O_3–Fe_2O_3 nanocrystals [12], and nano In_2O_3 [13], under harsh reaction conditions; some of the catalysts are expensive and heterogeneous. Cui *et al.* [14] synthesized it with stirring at 30–40 °C for 10 h under nitrogen in the presence of *p*-toluenesulfonic acid (TsOH). Radatz *et al.* [15] obtained it using glycerol as a solvent at 90 °C. Herein, we report an efficient procedure for the synthesis of 1-(2-furylmethylene)-2-(2-furyl)benzimidazole. The reaction procedures are operationally straightforward, mild, environmentally benign, and use ethanol as a solvent and no catalyst [16]. Four metal complexes of **L** have been prepared and characterized. The IR spectra, elemental analyses, and thermal analyses are in agreement with the crystal structure. A plausible mechanism (scheme 1) of the reaction has been proposed on the basis of formation of a Schiff base as an intermediate,



Scheme 1. A possible mechanism for synthesis of **L**.

followed by a nucleophilic attack. The intermediate proceeded with cyclization, followed by rearrangement to form the product [17,18].

3.2. *Crystal structure analysis of 1–3*

3.2.1. Co($C_{16}N_2O_2H_{12}$)$_2$$Cl_2$ (1). Co(II) is four-coordinate with two chlorides (Cl1, Cl2) and two nitrogen atoms (N1, N2) from two benzimidazoles (figure 1). The coordination geometry is a distorted tetrahedral [19] with the N(1)–Co(1)–Cl(2) angle of 114.40(14)° significantly larger than the Cl(2)–Co(1)–Cl(1) angle 105.79(7)°, and the N(2)–Co(1)–N(1) angle 108.78(19)°. The bond lengths, Co–N 2.042(5) and 2.041(4) Å, Co–Cl 2.2648(19) and 2.2668(17) Å, are within normal ranges for similar complexes [20–23]. As shown in figure 2, intramolecular face-to-face $\pi-\pi$ stacking interactions exist between the furan and benzimidazole rings with a centroid–centroid distance of 3.632 Å. The dihedral angle between the two planes is 26.895°. Crystal packing exhibits intermolecular $\pi-\pi$ stacking (edge-to-face interaction, $D = 3.596$ Å) between the O4–C25 bond in the furan ring and the benzimidazole ring.

3.2.2. Zn($C_{16}N_2O_2H_{12}$)$_2$(NO_3)$_2$ (2). Zn(II) is coordinated with two nitrogens (N1, N1A) from two ligands and two oxygens (O1, O1A) of nitrate, giving a distorted tetrahedral geometry (figure 3). Bond angles of N(1A)–Zn(1)–N(1), O(1A)–Zn(1)–N(1), and O(1)–Zn(1)–O(1A) are 124.56(15)°, 107.02(11)°, and 122.72(15)°, respectively. The bond distances are 2.014(3) Å for Zn(1)–N(1) and 1.997(2) Å for Zn(1)–O(1), shorter than those reported in Zn(II) coordination complexes [24,25]. There exist $\pi-\pi$ stacking intermolecular interactions between the furan ring and the benzimidazole of neighboring

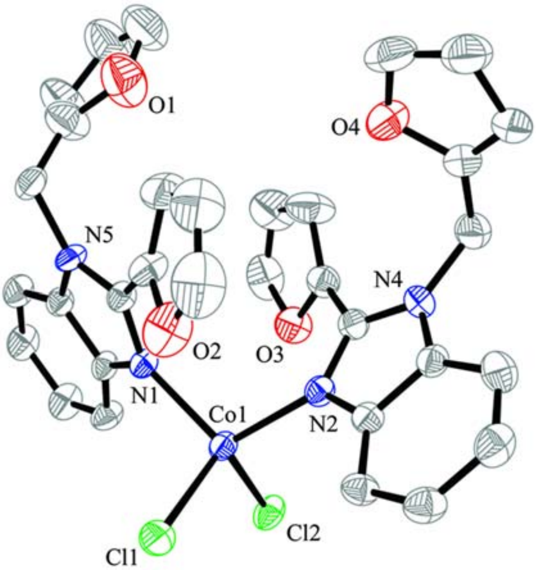


Figure 1. Coordination environment of Co(II) in **1** (30% ellipsoid probability). Hydrogens are omitted for clarity.

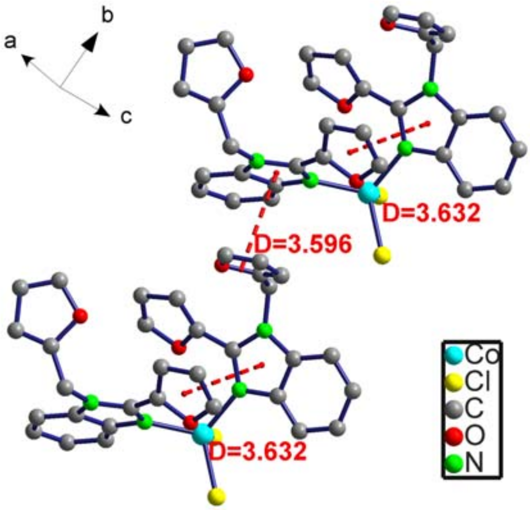


Figure 2. Interaction of **1**.

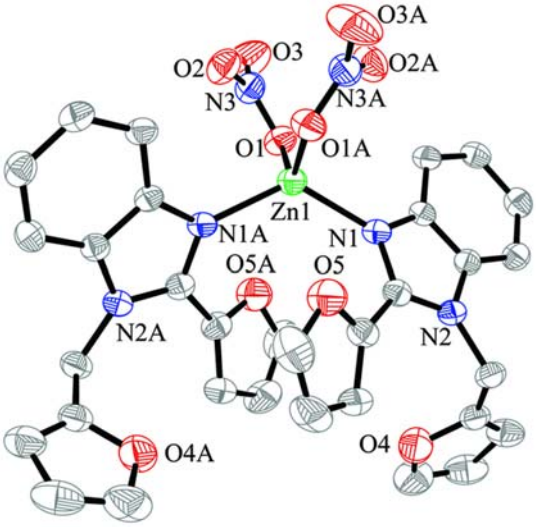


Figure 3. Coordination environment of Zn(II) in **2** (30% ellipsoid probability). Hydrogens are omitted for clarity.

molecules with centroid–centroid distance of 3.622 Å. The dihedral angle between the two planes is 1.850° (figure 4). Weaker $\pi-\pi$ stacking intramolecular interaction exists between two furan rings with a centroid distance of 3.747 Å.

3.2.3. $Cd(C_{16}N_2O_2H_{12})_2(CH_3COO)_2$ (3). The Cd(II) is six-coordinate with two nitrogens (N1A, N1) of two L and four oxygens (O4A, O4, O3A, O3) from two acetates to complete a distorted octahedral geometry [26] (figure 5). The bond distances are 2.439(6) Å for Cd(1)–O(3), 2.313(6) Å for Cd(1)–O(4), and 2.279(6) Å for Cd(1)–N(1), comparable

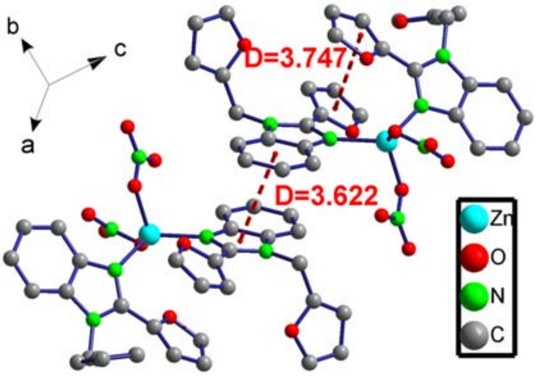


Figure 4. Interaction of **2**.

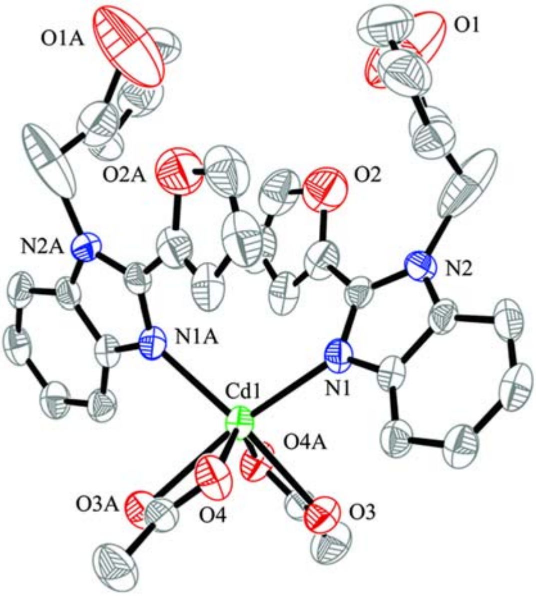


Figure 5. Coordination environment of Cd(II) in **3** (30% ellipsoid probability). Hydrogens are omitted for clarity.

with reported examples [27]. The centroid–centroid distance between the C33−C46 bond in the furan ring and the adjacent benzimidazole ring is 3.502 Å, indicating a strong edge-to-face π−π interaction (figure 6). There are two kinds of π−π stacking interactions between **1** and **2**, and intramolecular π−π interactions among **1–3** are different. The reason may be that Cd(II) is six-coordinate, but Co(II) and Zn(II) are four-coordinate.

3.3. *Characterization of the complexes*

3.3.1. UV–vis Spectra. UV–vis spectra of free ligand and their metal complexes were determined in N,N-Dimethylformamide (DMF) (Supplementary material). The free ligand has maxima at 275 and 308 nm, attributed to π−π^* transitions [28]. In the complexes,

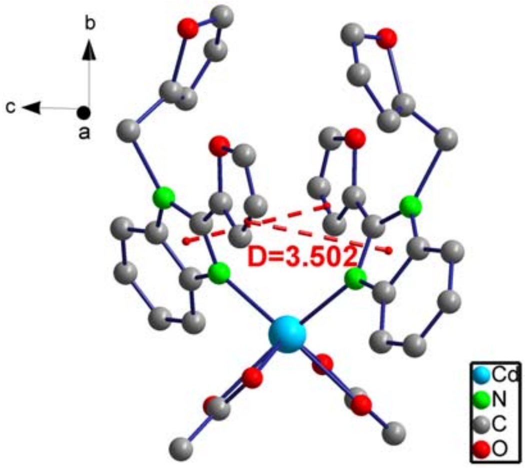


Figure 6. Interaction of **3**.

these peaks shift to longer wavelengths. The red shift reveals that the ligand is coordinated, agreeing with X-ray diffraction. The low-energy absorption at 325 nm can be assigned to metal-to-ligand charge-transfer transitions.

3.3.2. IR Spectra. The $\upsilon_{\text{Ar}-\text{H}}$ absorptions of the benzimidazole moieties at 3100−3000, 1625−1410, and 760−740 cm^{-1} ($\delta_{=\text{CH}}$) are observed in all complexes. Absorptions of fural rings in the benzimidazole moieties of **1–4** are observed at 1310−1020 cm^{-1} ($\upsilon^{as}{}_{\text{C}-\text{O}-\text{C}}$) and 1075−1020 cm^{-1} ($\upsilon^{s}{}_{\text{C}-\text{O}-\text{C}}$). The IR stretch of C−C of benzene appears between 1392 and 1623 cm^{-1} [29]. The strong and single bands at 1385 and 1384 cm^{-1} for **2** and **4** indicate the presence of nitrate. In all the complexes, C−N is shifted in relation to free ligand, indicative of the coordination through benzimidazole nitrogen. These spectroscopic features of **1–3** were further confirmed by the corresponding crystal structure determinations.

3.3.3. Thermal analysis. The TGA curves of **1–4** show very similar thermal behavior (Supplementary Material). The first weight loss corresponds to the loss of one ligand (30−300 °C for **1**; 30−280 °C for **2**; 30−220 °C for **3**; 30−300 °C for **4**), the second weight loss is attributable to the residual ligand. The higher decomposition temperature indicates that **2** has better thermal stability than **3** confirmed by the shorter M−N bond length in **2**. The thermal decomposition behaviors of these complexes reflected that they have good thermal stability.

3.3.4. Luminescence. The luminescence spectra of L and **1–4** in DMF were investigated at room temperature (figure 7). The free ligand exhibits a broad luminescence emission band with a maximum at 365 nm upon excitation at 310 nm; such broad emission bands may be tentatively assigned to intraligand (π–π^{*}) transitions. In the same conditions, **1–4** exhibit more intense luminescence with emission maxima at 355 and 365 nm, respectively.

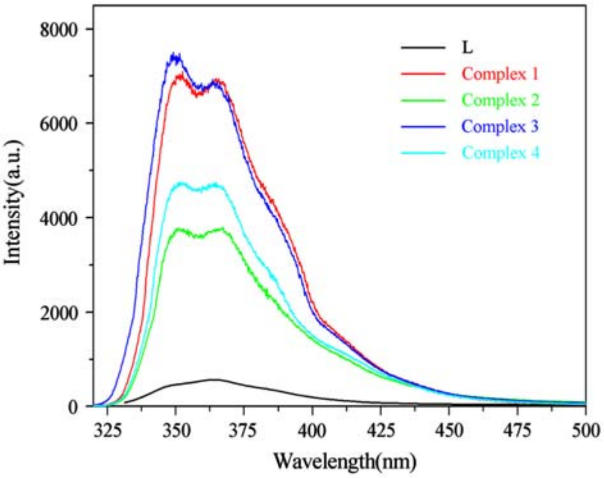


Figure 7. Emission spectra of **L** and **1**–**4**.

By comparing the emission spectra of **1–4**, the luminescence intensity of 3 is stronger than that of 1, 2, and 4, probably due to the differences of metal ions and the coordination environment, because the luminescence behavior is closely associated with the metal ions and the ligands coordinated around them [30–32]. Similar emission of **2** and **4** may be due to the similar coordination environments.

4. Conclusion

The 1-(2-furylmethylene)-2-(2-furyl)benzimidazole (**L**) and its Co(II) (**1**, **4**), Zn(II) (**2**), Cd(II) (**3**) complexes have been prepared in an efficient and environmentally friendly method. Structural analysis shows that Cd(II) is six-coordinate, but Co(II) and Zn(II) are four-coordinate. Luminescent properties of the complexes are closely related with the structure and metal ions.

Supplementary materials

CCDC 847749, 947750 for **2**, **3** contain the Supplementary crystallographic data. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK [Fax:+44 (0)-1223-336033; E-mail: deposit@ccdc.cam.ac.uk].

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