Front. Chem. China 2007, 2(4): 407–413
DOI 10.1007/s11458-007-0077-6

RESEARCH ARTICLE

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Synthesis and photoluminescence properties of 8-hydroxyquinoline derivatives and their metallic complexes

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Abstract Three new 8-hydroxyquinoline derivatives, i.e. 5-[(4-styryl-benzylidene)-amino]-quinolin-8-ol (**1**), 5-[(4-bromo-2-fluoro-benzylidene)-amino]-quinoline-8-ol (**2**) and 2-[2-(9-ethyl-9H-carbazol-2yl)-vinyl]-quinolin-8-ol (**3**), and their metallic complexes were synthesized and identified by ultraviolet-visible (UV-Vis), ^{1}H nuclear magnetic resonance (^{1}H NMR), Fourier transform infrared spectrometer (FTIR), mass spectrometry (MS) spectra and elemental analyses. Their fluorescence properties were studied by photoluminescence, which indicated that the luminescence wavelength of 5- and 2-substitued-8-hydroxyquinoline derivatives shifted to red in comparison with that of 8-hydroxyquinoline. Meanwhile, the fluorescence lifetime of 2-[2-(9-ethyl-9H-carbazol-2yl)-vinyl]-quinolin-8-ol and its zinc complex showed long lifetime in benzene solution.

Keywords metallic complexes, fluorescence properties, 8-hydroxyquinoline derivates

1 Introduction

Since Tang and VanSlyke [1] initially employed vacuum deposition in 1987 for the formation of double organic light-emitting diodes (OLED) with diamine as the hole-transporting (HT) materials and 8-hydroxyquinoline aluminium (Alq_3) as the emitting and electron-transporting (ET) material, small organic molecules and polymer light-emitting materials have attracted extensive interest in this field [2−7].

In order to obtain organic materials being capable of emitting white and full color lights in high intensity, many researchers have attempted to choose different HT materials, ET materials, or change the configuration of the devices. To date, 8-hydroxyquinoline aluminium (Alq_3), an efficient green electroluminescent material, has been considered to be an excellent light-emitting material. However, it is still not satisfactory for the use of Alq_3 in view of its monocolor emission. Meanwhile, the fabrication device of Alq_3 is very difficult to make due to the low solubility of Alq_3 in general organic solvents. Therefore, the use of Alq_3 is hard to implement the needs of large area flat-panel display.

Recently, some results showed that different wavelength light-emitting could be obtained by modifying the structure of 8-hydroxyquinoline derivatives and changing the coordinated metal element. Carbazole was used as an important material in fabrication of the device due to its photo-physics property [8–9]. It was also found that some large conjugated substituents at 2- or 5-position of 8-hydroxyquinoline could tune luminescence wavelength. In view of these, a family of 8-hydroxyquinoline derivatives has been synthesized with substituents at the 5-position and 2-position of 8-hydroxyquinoline and their metallic complexes. The synthetic route is outlined in Scheme 1.

The structures of metallic complexes Al(**1**)$_3$, Al(**2**)$_3$, (**3**)$_2$Zn and (**3**)$_2$Cu are shown in Scheme 2.

2 Experimental

2.1 Reagents and instruments

Melting points were determined using an XT-4 microscope melting point apparatus and was uncorrected. Ultraviolet (UV)-visible spectra were recorded on a SHIMADZU UV-2550 spectrophotometer. Infrared (IR) spectra were recorded on a Perkin-Elmer Fourier transform infrared (FTIR) spectrometer and measured as KBr pellet. ^{1}H nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Bruker DRX-400 MHz spectrometer with TMS (Tetramethylsilane) as internal standard and DMSO (d_6)/$CDCl_3$ as solvent. Electron ionization mass spectrometry (EI-MS) spectra were performed with a FINNIGAN Trace DSQ mass spectrometer at 70 eV using a direct inlet system. Elemental analyses were recorded with a FINNIGAN C, H, N, S, O spectrometer. Fluorescence spectra were measured with a HITACHGI

Translated from *Chinese Journal of Organic Chemistry*, 2007, 27(3): 402–408 [译自: 有机化学]

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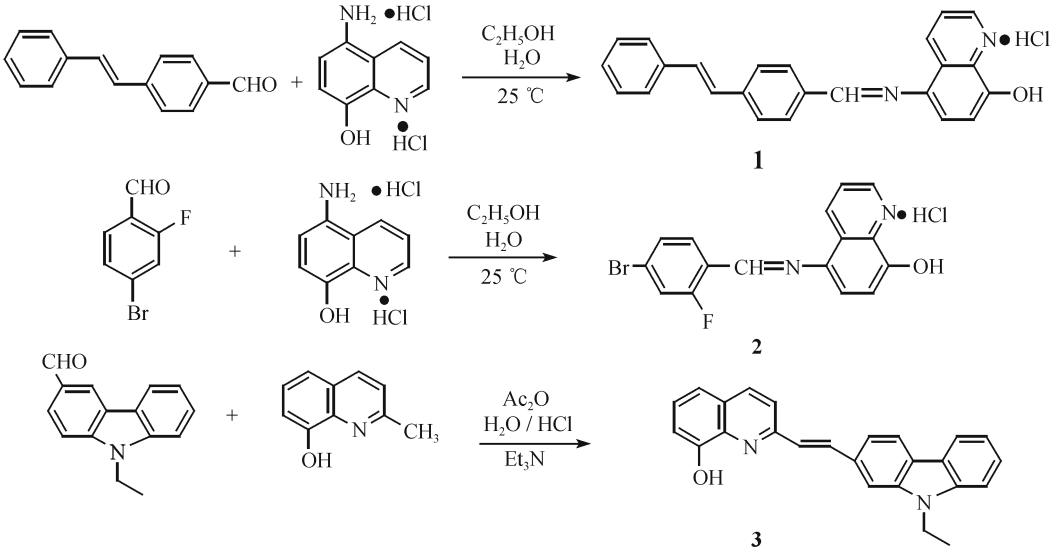
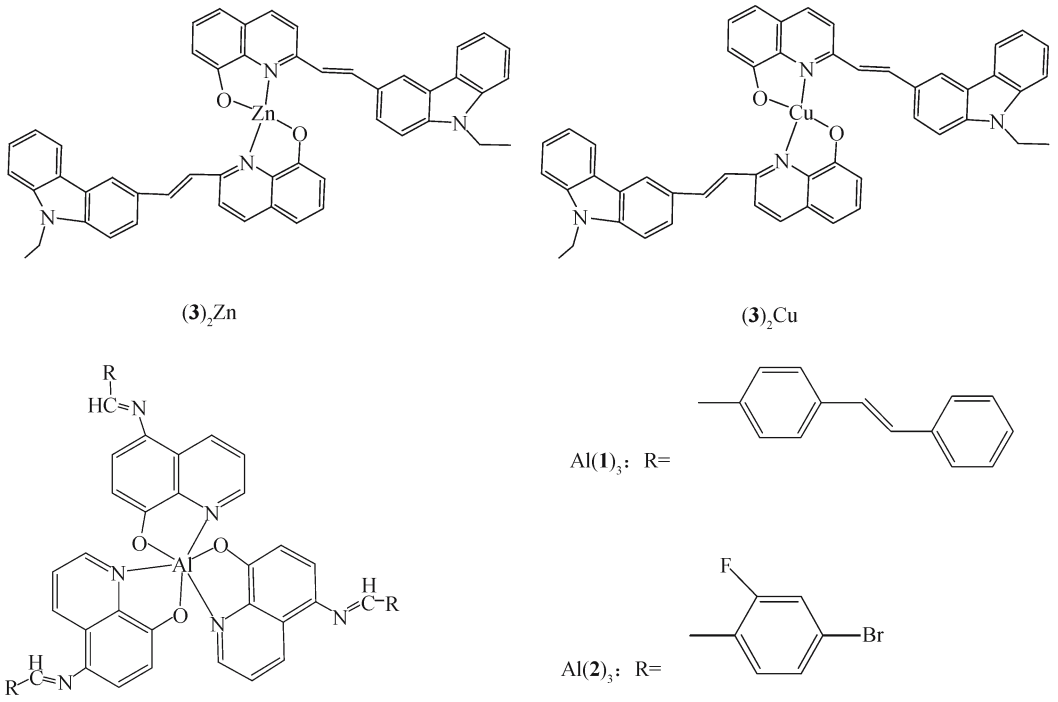


Scheme 1



Scheme 2

F-2500 Fluorophotometer. Thermogravimetric analyzer (TGA) was determined on a Thermal Analyzer (TGA2050, TA Instruments, USA). The decay curves were measured by a FLS920-Combined Fluorescence Lifetime and Steady State Spectrometer (EDINBURGH INSTRUMENTS). All the measurements were carried out at room temperature. 2-Methyl-8-hydroxyquinoline, 5-amino-8-hydroxyquinoline dihydrochloride, 4-styryl-benzaldehyde, 4-bromo-2-fluoro-benzaldehyde and 9-ethyl-9H-carbazole-3-carbaldehyde were purchased from Tokyo Kasei Kogyo Co., all solvents were purchased from Guangzhou Chemical Company in China. All chemicals were of the highest grade available and were used without further purification. The experimental course was monitored by TLC (Thin layer Chromatography). Column chromatography was carried out on silica gel (Wako C-300).

2.2 Synthesis of compound **1**

A 250 mL flask was charged with a mixture of 5-amino-8-hydroxyquinoline dihydrochloride (0.218 g, 0.937 mmol) and H_2O (10 mL) under nitrogen atmosphere, then a solution of 4-styryl-benzaldehyde (0.198 g, 0.953 mmol) in C_2H_5OH (25 mL) was added dropwise at room temperature within 1.5 h under stirring. Small platelets formed on the surface of

the solution almost immediately after addition of the benzaldehyde, and then yellow precipitates appeared. At the end of the reaction, the precipitated yellow solid was filtered off, washed with anhydrous ethanol 2−3 times and dried in vacuum. A purified sample was obtained by recrystallization from DMF (Dimethylformamide). Yield 0.217 g, 65%. m.p. 242°C−244°C; FT-IR (KBr) υ: 3,440.94, 1,618.82, 1,596.89, 1,583.07, 1,500.69, 1,471.32, 1,246.01, 961.21 cm^{-1}; UV-vis (λ_{max}): 267, 333, 385.5 nm; MS (EI) *m/z* (%): 350.1 (M^+); ^{1}H-NMR (DMSO-d_6, 400 MHz) δ: 9.12 (d, J = 7.80 Hz, 1H, Ar), 8.94 (s, 1H, OH), 8.79 (s, 1H, N=CH), 8.05 (d, J = 8.20 Hz, 2H, Ar), 7.79 (d, J = 8.00 Hz, 2H, Ar), 7.65 (d, J = 7.80 Hz, 3H, Ar), 7.45 (d, J = 15.2 Hz, 2H, CH=CH), 7.28−7.38 (m, 7H, Ar), 7.17 (d, 2H, J = 8.00 Hz). Anal. calcd for $C_{24}H_{18}N_2$: C 82.29, H 5.18, N 8.00; found C 82.15, H 5.17, N 8.30.

2.3 Synthesis of compound **2**

The yellow compound 2 was synthesized in the same manner as described for compound 1. Yield 0.212 g, 80%. m.p. 190°C−192°C, FT-IR (KBr) υ: 3,430.00 (O−H), 1,626.97 (C=N), 1,602.10, 1,551.38, 1,481.94 (Ar), 1,307.26 (C−F), 575.20 (C−Br), 1,263.69 (Ar−O) cm^{-1}; UV-vis (λ_{max}): 269, 381 nm; MS (EI) *m/z* (%): 344, 346; ^{1}H-NMR (DMSO-d_6, 400 MHz) δ: 9.27 (d, J = 8.00 Hz, 1H, Ar), 9.06 (s, 1H, N=CH), 8.93 (s, 1H, OH), 8.23−8.27 (m, 1H, Ar), 7.92 (d, J = 8.10 Hz, 1H, Ar), 7.62−7.80 (m, 3H, Ar), 7.39 (d, J = 8.00 Hz, 1H, Ar), 7.23 (d, J = 8.00 Hz, 1H, Ar). Anal. calcd for $C_{16}H_{10}N_2BrF$: C 55.65, H 2.90, N 8.12; found C 55.24, H 3.04, N 8.07.

2.4 Synthesis of compound **3**

A mixture of 2-methyl-8-hydroxyquinoline (3.97 g, 25 mmol), 9-ethyl-9H-carbazole-3-carbaldehyde (5.58 g, 25 mmol) and acetic anhydride (25 mL) was stirred and heated at 125°C–135°C for 40 h under nitrogen atmosphere. After cooling, the mixture was poured into ice water (100 mL) and stirred 4–5 h, and a brown black solid was obtained by filtration. Then the solid (12 mmol) was dissolved in DMF (30 mL) by heating at 120°C, aqueous hydrochloric acid (36%–38%, 7 mL) was added to the solution, and the mixture was heated at 125°C–135°C for 2 h. The precipitated orange solid was filtered off, washed with water and dried in vacuum. A flask was charged with a mixture of the orange solid (3.982 g, 9 mmol) and DMF (25 mL). The mixture was heated under reflux, then triethylamine (2.1 g, ~20 mmol) was added, and immediately a clear solution was obtained. After completion of the reaction, the mixture was poured into ice water (100 mL), the obtained yellow solid was filtered off, then washed with water and dried to afford crude compound **3**. The product was purified by silica gel (100–200 mesh) column chromatography using ethyl acetate/petroleum ether as an eluent. Yield 3.282 g, 82.42%. m.p. 140°C–141°C; FT-IR (KBr) ν (cm^{-1}): 3,392.59, 3,037.03, 2,972.83, 2,923.45, 1,618.04, 1,593.40, 1,561.36, 1,507.15, 1,489.90, 1,477.58, 1,332.19, 1,245.94, 1,231.16, 962.55, 748.17, 587.99. ^{1}H-NMR (DMSO) δ: 9.48 (s, 1H), 8.53 (s, 1H), 8.30 (d, 1H, J = 16.08 Hz), 8.27 (d, 1H, J = 8.53 Hz), 8.21 (d, 1H, J = 7.66 Hz), 7.87 (d, 1H, J = 8.55 Hz), 7.79 (d, 1H, J = 8.58 Hz), 7.68 (d, 1H, J = 8.56 Hz), 7.63 (d, 1H, J = 8.56 Hz), 7.50 (d, 1H, J = 16.11 Hz), 7.49 (t, 1H, J = 8.14 Hz), 7.37 (q, 2H, J = 7.18 Hz), 7.26 (t, 1H, J = 7.60 Hz), 7.10 (d, 1H, J = 6.48 Hz), 4.47 (q, 2H, J = 7.20 Hz), 1.35 (t, 3H, J = 7.15 Hz). EI-Ms m/z (M^+): 364. Anal. Calcd for $C_{25}H_{20}N_2O$: C 82.42; H 5.49; N 7.69. Found C 82.31; H 5.81; N 7.55.

2.5 Synthesis of metallic complex Al(**1**)$_3$

A flask was charged with a mixture of ligand **1** (0.210 g, 0.6 mmol) and DMF (10 mL), then $AlCl_3 \cdot 2H_2O$ (0.048 3 g/10 mL H_2O, 0.2 mmol) was added dropwise at room temperature within 1.5 h. The mixture was heated under reflux for 4 h, then cooled and neutralized to pH 8 with $NH_3 \cdot H_2O$ (5 mol/L). Yellow precipitates were obtained when $NH_3 \cdot H_2O$ was added, then the precipitates were filtered off, washed with methanol or ethanol 3–5 times and dried in vacuum. Yield, 0.162 g, 75%, m.p. >300°C, UV-vis (λ_{max}): 269, 334.5, 440.5 nm; FT-IR (KBr) υ: 3,400.44, 1,614.92, 1,602.98, 1,571.78, 1,499.57, 1,328.44, 1,102.40, 960.41, 500.70, 454.79 cm^{-1}; Anal. calcd for $C_{72}H_{51}N_6Al$: C 80.47, H 4.75, N 7.82; found C 80.13, H 4.53, N 8.29.

2.6 Synthesis of metallic complex Al(**2**)$_3$

The metallic complex Al(**2**)$_3$ was prepared following the above general procedure for Al(**1**)$_3$. Yield 0.175 g, 70%. m. p. >300°C; FT-IR (KBr) υ: 3,392.27, 1,614.85, 1,601.47, 1,572.09, 1,499.86, 1,326.95, 1,101.33, 542.94, 460.14 cm^{-1}, UV-vis (λ_{max}): 267, 433 nm; Anal. calcd for $C_{48}H_{27}N_6Br_3F_3Al$: C 54.39, H 2.55, N 7.93; found C 54.88, H 2.57, N 7.73.

2.7 Synthesis of metallic complex Zn(**3**)$_2$

The complex Zn(**3**)$_2$ was prepared from compound **3** and $Zn(CH_3COO)_2$ in methanol. Thus, $Zn(CH_3COO)_2$ (0.092 g, 0.5 mmol) was dissolved in anhydrous methanol (15 mL). The obtained Zn(II) solution was added dropwise to the solution of compound **3** (0.364 g, 1 mmol) in anhydrous methanol (50 mL) under stirring, and then the mixture was stirred for 24 h. Yellow precipitates were obtained, then filtered off, washed with methanol and DMF 3–5 times respectively, and dried in vacuum. Yield 0.385 g, 87%. Elemental analysis: found C 74.31, H 4.94, N 6.73 (%) calculated for $(EtCzHQ)_2Zn \cdot H_2O$: C 74.17, H 4.94, N 6.92 (%), IR (KBr) 3,048, 2,969, 2,929, 1,616, 1,586, 1,502, 1,493, 1,477, 1,435, 1,386, 1,297, 1,231, 1,101, 829, 745, 472.

2.8 Synthesis of metallic complex Cu(**3**)$_2$

The complex Cu(**3**)$_2$ was prepared following the above general procedure for Zn(**3**)$_3$. Yield 0.167 g, 67%, m.p.

286°C−288°C. FT-IR (KBr) υ: 3,049, 2,975, 1,620, 1,592, 1,552, 1,504, 1,490, 1,434, 1,380, 1,285, 1,232, 1,105, 831, 746, 521 cm^{-1}; Anal. calcd for $(EtCzHQ)_2Cu{\cdot}H_2O$: C 74.26, H 4.95, N 6.93; found C 74.72, H 4.86, N 6.77.

3 Results and discussion

3.1 Properties of compounds **1, 2** and **3** and their metallic complexes

As shown in Table 1, these compounds have good thermal stability with high melting points, and element analyses identified the structure of the compounds.

3.2 Characteristics of spectra

Compounds **1** and **2** exhibited IR absorption bands at 3,400, 1,629–1,600 and 1,400–1,600 cm^{-1} due to hydroxyl function, "C = N" and phenyl ring function, respectively. An absorption peak at 360–400 cm^{-1} appeared in the UV-vis spectra of **1** and **2** confirming the formation of bond "C = N". The EI-MS spectrum of **1** showed a molecular-ion peak at *m/e* 350.1, while *m/e* 344 and *m/e* 346 molecular-ion peaks appeared in the EI-MS spectrum of **2** due to the isotope effect of bromine element. The two singlets at δ 8.80 and 8.25 in the ^{1}H-NMR spectra of compounds **1** and **2** were designated to the hydrogen of "N = CH".

The IR spectra of Al(**1**)$_3$, Al(**2**)$_3$, (**3**)$_2$Zn and (**3**)$_2$Zn were similar to those of their parent compounds **1**, **2** and **3** except several peaks changed. New absorptions at 480–525 cm^{-1} due to metal-nitrogen coordination, and at 420–460 cm^{-1} due to metal-oxygen coordination appeared in the complex spectra [10,11]. Meanwhile, the intensity of hydroxyl peaks was weakened in Al(**1**)$_3$ and Al(**2**)$_3$ compared with their parent compounds due to metal-oxygen coordination. Meanwhile, the broad absorption peaks appeared owing to water molecule existence in (**3**)$_2$Zn and (**3**)$_2$Cu.

3.3 UV absorption spectra of metallic complexes

The UV-vis absorption spectra are shown in Fig. 1. As indicated in the absorption spectra, the ligands **1**, **2** and **3** showed a common broad band at 370–385 nm assigned to an intramolecular π-π* charge-transfer band in the quinoline ring. In contrast, this band was much weaker in the complexes due to strong conjugation action. Large red shifting bands at 380–440 and 265–333 nm were observed in the complexes. The absorption figure of Al(**1**)$_3$ was similar with that of Al(**2**)$_3$. The luminescence property of ligand **3** has been reported in the literature [8]. Compared with the spectrum of **3**, its complexes (**3**)$_2$Zn and (**3**)$_2$Cu gave several new absorption peaks at 300–380 nm. Meanwhile, the absorption spectra of Zn and Cu complexes were similar to that of Alq_3.

3.4 Fluorescence properties of these compounds

Fluorescence spectra were measured on a HITACHGI F-2500 Fluorophotometer at 10^{-4} mol/L concentration of the compounds in DMF. The fluorescence spectra of ligands and complexes are shown in Fig. 2.

The fluorescence spectra of ligand **1** and Al(**1**)$_3$ were shown in Fig. 2(a). The maximal emission peak of ligand **1** occurred at 406 nm with an excitation wavelength (λ_{max} = 340 nm), indicating that the ligand **1** itself had low luminescence property. As for the complex Al(**1**)$_3$, a broad peak appeared at 450–600 nm. The maximal fluorescence emission peak occurred at 517 nm with the maximal excitation wavelength (λ_{max} = 380 nm), and the fluorescence intensity of Al(**1**)$_3$ was higher than that of ligand **1**. The color of the fluorescence of Al(**1**)$_3$ was between blue and green.

The fluorescence spectra of ligand **2** and Al(**2**)$_3$ were shown in Fig. 2(b). The maximal emission peak of ligand **2** occurred at 452 nm with an excitation wavelength (λ_{max} = 340 nm), indicating that the ligand **2** itself had certain luminescence property. As for the complex Al(**2**)$_3$, two broad peaks appeared at 460–600 nm. The maximal emission peaks occurred at 494 and 541 nm with the maximal excitation wavelength (λ_{max} = 380 nm), and the fluorescence intensity of Al(**2**)$_3$ was higher than that of ligand **2**. The color of the fluorescence of Al(**2**)$_3$ was between green and orange. Therefore, multi-function and intensive fluorescence can be obtained by coordination of a metal ion with an appropriate ligand.

The fluorescence spectra of ligand **3** and (**3**)$_2$Zn were shown in Fig. 2(c). The maximal emission peak of ligand **3**

Table 1 Elemental analyses for compounds of **1, 2** and **3**, and their complexes

Compd	m.p		Elemental analysis: found (cacld)		
	Color	°C	C	H	N
1	Buff	242–244	82.15 (82.29)	5.17 (5.18)	8.30 (8.00)
2	Yellow	190–192	55.24 (55.65)	3.04 (2.90)	8.07 (8.12)
3	Shallow yellow	189–190	82.32 (82.42)	5.70 (5.49)	7.55 (7.69)
Al(**1**)$_3$	Yellow	>300	80.13 (80.47)	4.53 (4.75)	8.29 (7.82)
Al(**2**)$_3$	Yellow	>300	54.88 (54.39)	2.57 (2.55)	7.73 (7.93)
(**3**)$_2$Zn	Orange	244–246	74.31 (74.17)	4.94 (4.94)	6.73 (6.92)
(**3**)$_2$Cu	Green	286–288	74.72 (74.26)	4.86 (4.95)	6.77 (6.93)

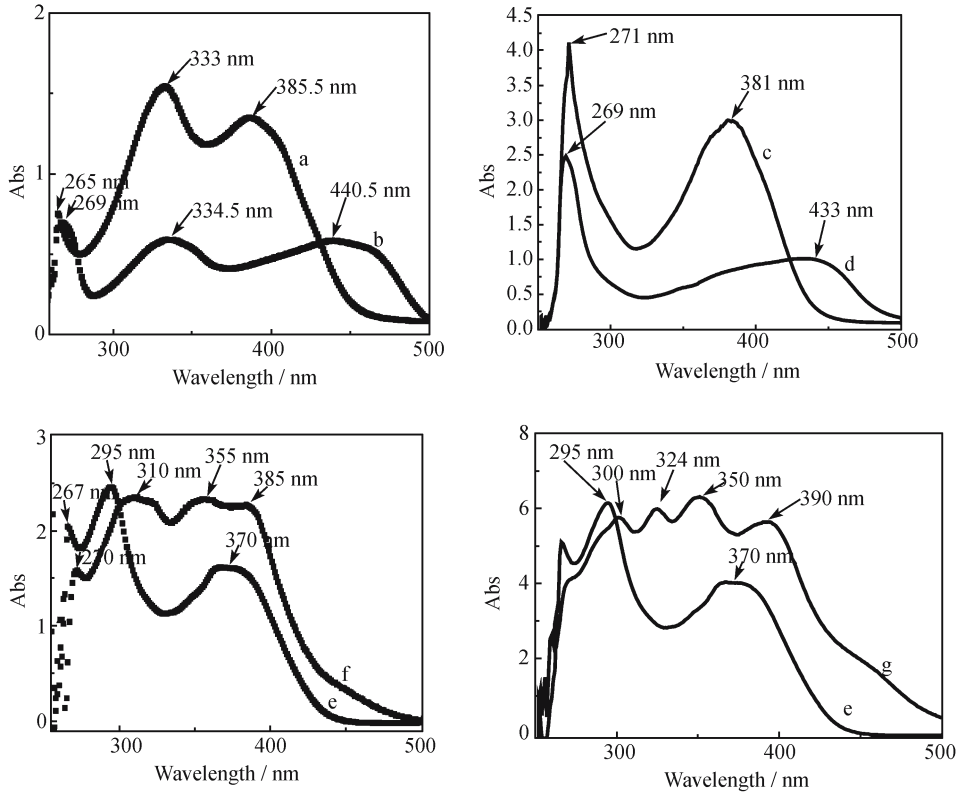


Fig. 1 Absorption spectra of quinoline derivatives and their metal complexes: **1** (a), Al(**1**)$_3$ (b), **2** (c), Al(**2**)$_3$ (d), **3** (e), Zn(**3**)$_3$ (f) and Cu(**3**)$_3$ (g)

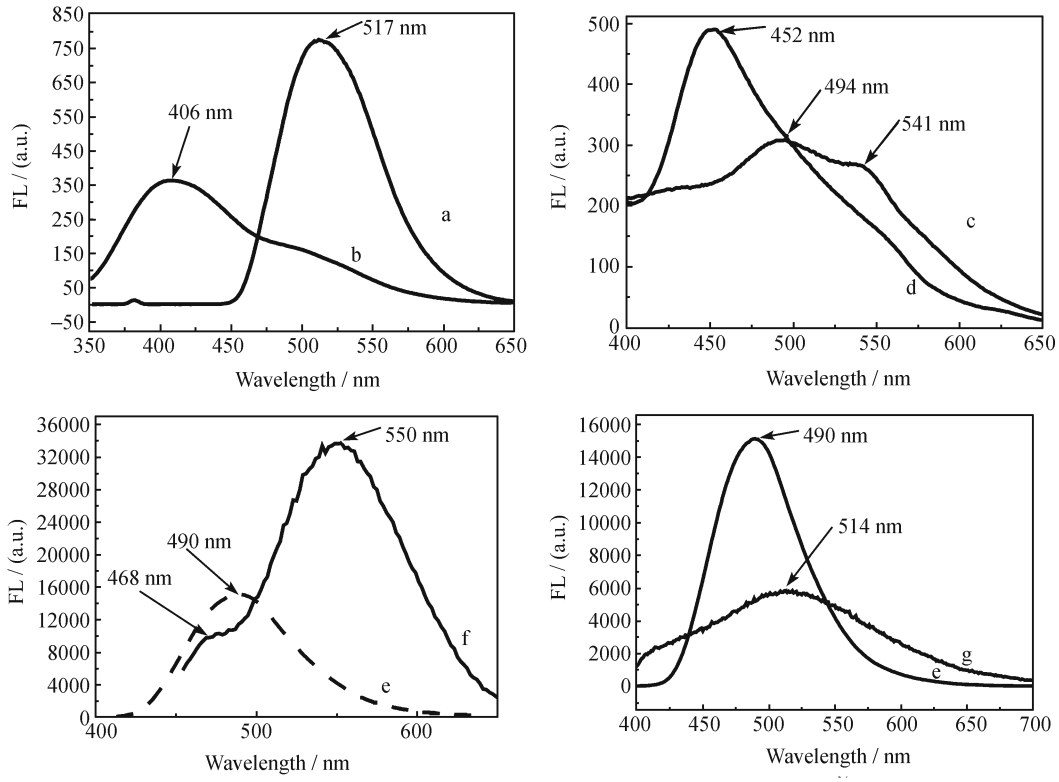


Fig. 2 Fluorescence emission spectra of quinoline derivatives and their metal complexes: **1** (b), Al(**1**)$_3$ (a), **2** (d), Al(**2**)$_3$ (c), **3** (e), (**3**)$_2$Zn (f) and (**3**)$_2$Cu (g)

occurred at 490 nm with an excitation wavelength (λ_{max} = 370 nm), indicating that the ligand **3** itself had strong luminescence property. As for the complex $(\mathbf{3})_2$Zn, a broad peak appeared at 500–600 nm. The maximal emission peak occurred at 550 nm with the maximal excitation wavelength (λ_{max} = 385 nm), and the fluorescence intensity of $(\mathbf{3})_2$Zn was higher than that of ligand **3**. The color of the fluorescence of $(\mathbf{3})_2$Zn was between blue and green.

The fluorescence spectra of ligand **3** and $(\mathbf{3})_2$Cu were shown in Fig. 2(d). For the complex $(\mathbf{3})_2$Cu, a broad peak appeared at 500–600 nm. The maximal emission peak occurred at 514 nm with the maximal excitation wavelength (λ_{max} = 390 nm), and the fluorescence intensity of $(\mathbf{3})_2$Cu was weaker than that of ligand **3**. This result could be explained by the paramagnetic properties of Cu (II) (d^9) ions, having unpaired d-electrons which effectively quench both the singlets and triplets of ligand **3**. Quenching the fulorescence by paramagnetic maetal ions has been a well-known phenomenon.

3.5 PL Lifetime of $(\mathbf{3})_2$Zn

The PL lifetime τ of $(\mathbf{3})_2$Zn in various solvents was measured on a FLS920-Combined Fluorescence Lifetime and Steady State Spectrometer with a diode laser (pulse width of 10 ns, repetition rate of 10 Hz) at wavelength 308 nm. Laser excitations were performed at wavelength 400 nm so that the lowest energy states involving excitation in intramolecular charge-transfer band were excited. Here, much attention was paid to the emission in the red and orange-red regions that corresponded to the emission from the lowest-energy band. In this energy region, the emission curves displayed single-exponential decay. The curves are shown in Fig. 3, and the measured lifetime decreased with the increase in solvent polarity; for instance, $(\mathbf{3})_2$Zn had a lifetime of 7.20 ns in benzene and 1.34 ns in DMF. These data indicated that the excited molecules were quenched efficiently by polar solvents and the quenching should be due to the dipole-dipole interaction between solvent and $(\mathbf{3})_2$Zn.

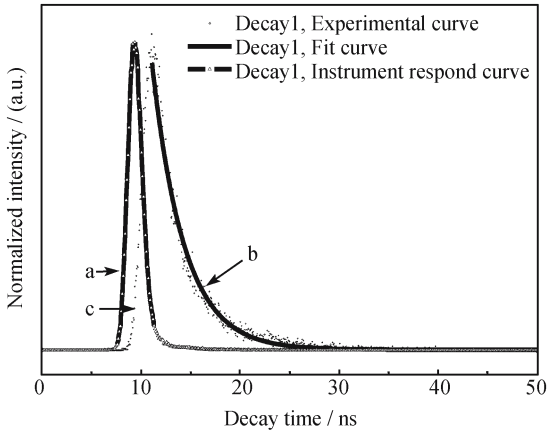


Fig. 3 Fluorescence decay curves of $(3)_2$Zn in benzene

3.6 Thermal stabilities of metallic complexes

The thermal stabilities of these metallic complexes were examined by differential scanning calorimetry (DSC) and thermogravimetic analysis (TGA) at a rate of 10 K/min. It was observed that thermal decomposition occurred in two steps for the complexes $(\mathbf{3})_2$Cu and $(\mathbf{3})_2$Zn. When $(\mathbf{3})_2$Cu and $(\mathbf{3})_2$Zn were heated, the first weight loss peaks of $(\mathbf{3})_2$Cu and $(\mathbf{3})_2$Zn were observed at 122.3°C and 117.5°C, respectively. As the temperature increased, there was an intense mass loss in the range of 280°C–420°C for the compounds $(\mathbf{3})_2$Cu (total weight loss 89.952%) and $(\mathbf{3})_2$Zn (total weight loss 87.606%), and the final weights obtained after the decomposition of $(\mathbf{3})_2$Cu and $(\mathbf{3})_2$Zn were in accordance with the mass of CuO and ZnO, respectively. The transition temperatures (T_g) of $(\mathbf{3})_2$Cu and $(\mathbf{3})_2$Zn are shown Table 2. In the meantime, the results of element analyses (in Table 1) were in accordance with that of the thermogravimetric analyses.

Table 2 T_g of $(\mathbf{3})_2$Cu and $(\mathbf{3})_2$Zn

Compound	T_g
$(\mathbf{3})_2$Cu	287°C
$(\mathbf{3})_2$Zn	246°C

4 Conclusions

Three new 8-hydroxyquinoline derivatives and their metallic complexes were synthesized and characterized with their photoluminescent properties. The photoluminescence color was found to shift to red for the 2- and 5-substituted 8-hydroxyquinoline derivatives, providing a general way to tune the emission color of this class of compounds. Moreover, these complexes displayed good thermal stability. The lifetime of the complexes was long in non-polar solvent.

Acknowledgements The authors acknowledge financial support from the National Natural Science Foundation of China (Grant Nos. 20231020, 20471020) and Natural Science Foundation of Guangdong Province.

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