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Synthesis of light-emmting materials bis-[2′-2″-(9*H*-fluoren-2-yl)-vinyl-8-hydroxyquinoline] zinc(II) and bis-[2′-4″-(4,5-diphenyl-1*H*-imidazol-2-yl)styryl-8-hydroxyquinoline] zinc(II)

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ABSTRACT

Two novel light-emitting materials bis-[2′-2″-(9*H*-fluoren-2-yl)-vinyl-8-hydroxyquinoline] zinc(II) (**3**) and bis-[2′-4″-(4,5-diphenyl-1*H*-imidazol-2-yl)styryl-8-hydroxyquinoline] zinc(II) (**4**) containting 8-hydroxyquinoline and fluorene or imidazole moieties have been synthesized. The optical properties of these complexes were influenced by the styryl substituents, and exhibited orange-emission. They have higher fluorescence quantum yields than Alq$_3$, and good stabilities with thermal decomposition temperatures 395 °C and 435 °C. The single-layer OLED fabricated by **3** emitted lemon-yellow, and exhibited good device performance with a maximum luminance of 489 cd m^{-2}, and luminance efficiency of up to 0.41 cd A^{-1}. The single-layer OLED fabricated by **4** emitted yellow-green, and exhibited good device performance with a maximum luminance of 323 cd m^{-2}, and luminance efficiency of up to 0.54 cd A^{-1}.

1. Introduction

To have an efficient and practical OLED device, the requirements for the materials include low ionization potentials, highly thermal stability, ease of forming amorphous glass and high quantum yield of photoluminescence.[1,2] Undoubtedly, approaches for improving the thermal stability of organic molecules without reducing fluorescence are needed. Imidazole derivatives show unique chemical and physical properties because they contain imidazole heterocycle which has better thermal stability, and benzene rings can increase the degree of conjugation of the organic molecule. Fluorene derivatives also show high quantum yield of photoluminescence and high thermal stability,[3] as well as their high hole mobility which have been extensively used as hole-transporting materials in OLEDs.[4] In addition, research suggests that the zinc analog of light-emitting materials AlQ$_3$,[5,6] zinc bis(8-hydroxyquinoline) (ZnQ$_2$) has a better injection efficiency, a lower operating voltage, and higher quantum yields than the commonly used AlQ$_3$.[7] Inspired by these results, we designed and synthesized the following two new 8-hydroxyquinoline derivatives containing imidazole or fluorene unit and 8-hydroxyquinoline with pi-conjugated bonds. It is expected that a combination of fluorine or imidazole and 8-hydroxyquinoline could not only remain the thermal and morphological stability but also improve the quantum efficiency. On the other hand, complicated configurations of an organic light-emitting diode, multi-layers,[8,9] doped[10,11] or phosphorescent[12,13] OLEDs showed their industrial limits in terms of costly and time consuming fabrication process and durability due to interfacial or phase separation problems.[14–16] Thus, there are many attempts to fabricate a promising single-layer device.[17–22]

We report herein the first synthesis of light-emmting molecules bis-[2′-2″-(9*H*-fluoren-2-yl)-vinyl-8-hydroxyquinoline] zinc(II) (**3**) and bis-[2′-4″-(4,5-diphenyl-1*H*-imidazol-2-yl)styryl-8-hydroxyquinoline] zinc(II) (**4**) which containing the fluorene or imidazole moiety and 8-hydroxyquinoline derivative. The synthetic strategies for fluorene- and imidazole- based light-emmting molecules **3** and **4** are outlined in Scheme 1. We also investigated their thermal stabilities, fluorescence properties and use in single-layer OLEDs, where the results indicated that the organic materials have better thermal stability with luminescent properties for promising single-layer OLEDs.

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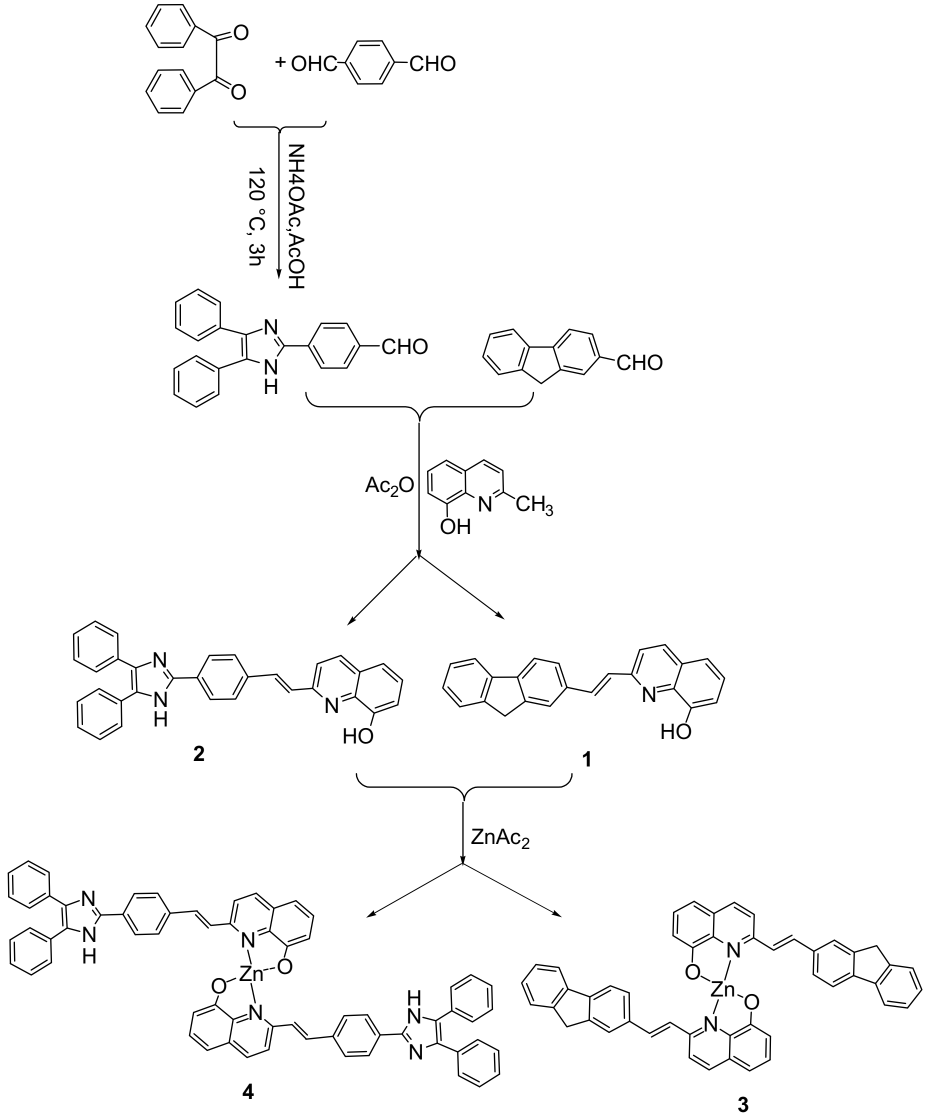

Scheme 1.

2. Results and discussion

2.1. Synthesis and characterization

Scheme 1 shows the synthetic route to chemically modify 8-hydoxyquinoline, allowing to fine-tune its optical and thermal properties. The two complexes were derived from substituted styryl-8-hydroxyquinol bearing fluorene and imidazole group which were from the reaction of 2-methyl-8-hydroxyquinoline and corresponding aldehydes in acetic anhydride. The substitution of an electron-donating substituent at the 4- or 2-position of 8-hydroxy quinoline will provoke a blue shift of the absorption spectrum of the corresponding complexes. Imidazole derivatives show unique chemical and physical properties because they contain imidazole heterocycle which has better thermal stability, and benzene rings can increase the degree of conjugation of the organic molecule. From simple MOPAC molecular modeling, two benzene rings twist out of the imidazole plane, which may help prevent intermolecular electrostatic interactions between the molecules.[23] Although thus structure may decrease the degree of the conjugation of the molecule, the imidazole moieties are electron rich and electron donating due to the lone-pair electrons in the sp^3 nitrogen atoms.

The thermal properties of the zinc(II) complexes **3** and **4** were determined by differential scanning calorimetry (DSC) and thermal gravimetric analysis(TGA) measurement from 20 to 1000 °C under nitrogen atmosphere with a heating rate of 10 °C/min. The thermal decomposition temperature of compound **3** is 395 °C, while compound **4** is 435 °C due to the good stability of imidazole ring. And the glass transition temperatures of compound **3** and **4** are 187 and 144 °C, respectively. The results indicate that both of them possess good thermal stability.

2.2. Photophysical properties

The absorption maximum ($\lambda^{abs}{}_{max}$) of the two complexes in DMF were 351 nm and 377 nm, respectively, and showed a blue-shift compared to ligands (360 nm and 384 nm). The emission spectra of the ligands **1** and **2** in different solvents showed large solvatochromism, which indicates that these molecules permit strong intramolecular charge transfer from the donor to the acceptor. The emission maximum λ_{em} of compound **2** increases as the solvent polarity increases, and the emission color ranges from blue to green. As shown in Figure 1, the solid-state PL maximum of **3** and **4** located at 598 and 607, respectively, which is red-shifted

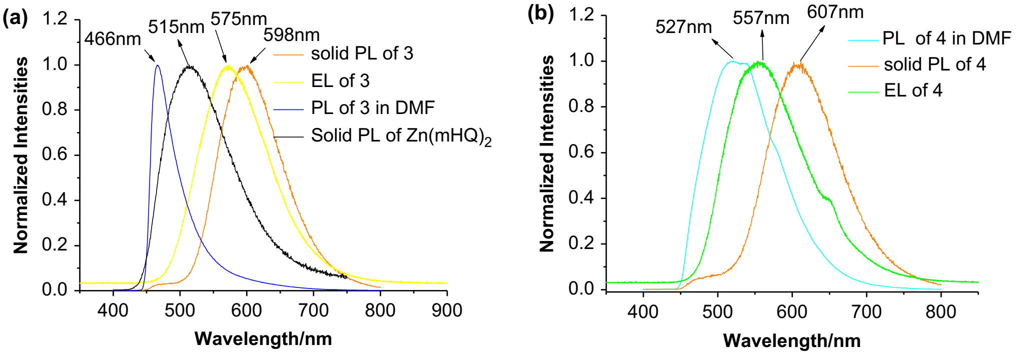

Figure 1. PL emission spectra and EL spectra of complex **3**(a) and **4**(b).

compared to the solution emission (466, 527 nm) and the solid-state emission of Zn(mHQ)$_2$ (515 nm).

The fluorescence quantum yields (Φ_{PL}) of the complexes **3** and **4** were measured in DMF solutions at concentrations of 1×10^{-6} mol/L and then calculated by Eq. 1, where φ_s and φ_{std} stand for the quantum yields of unknown and standard samples (φ_{std}=0.71, for RhB in ethanol),[24] A_s (<0.05) and A_{std} are the solution absorbance at the excitation wavelength (λ_{ex}), I_s and I_{std} are the integrated emission intensities, and η_s and η_{std} are the refractive indices of the solvents for unknown and standard samples respectively. The emission quantum yields of the complexes **3** and **4** measured in DMF are 0.168 and 0.273, respectively, which are higher than that of Alq$_3$ in DMF (0.116).[25]

$$\varphi_s = \varphi_{std}(A_{std}/A_s)(I_s/I_{std})(\eta_s/\eta_{std})^2 \quad (1)$$

2.3. Electroluminescence

In order to investigate the EL properties of bis-[2′-2″-(9*H*-fluoren-2-yl)-vinyl-8-hydroxyquinoline] zinc(II) (**3**) and bis-[2′-4″-(4,5-diphenyl-1*H*-imidazol-2-yl) styryl-8-hydroxyquinoline] zinc(II) (**4**), two single-layer devices (as shown in Fig. 2) of tin oxide(ITO)/**3** or **4**/Al were fabricated with a contrastive device fabricated by bis(2-methyl-8-hydroxyquinoline)zinc(II) [i.e., Zn(mHQ)], marked with device I, II and III.

Zinc complex compounds **3** and **4** having high emission quantum yields, were subjected to EL studies on an indium tin oxide (ITO) anode. Figure 3 shows the electroluminescence properties of the single-layer OLEDs with the structures of ITO/**3**, **4** or Zn(mHQ)$_2$ (50 nm)/Al(100 nm), which were fabricated by the conventional vacuum deposition and subsequently investigated for their electroluminescent (EL) properties. The single-layer device I fabricated by **3** exhibited lemon-yellow emission with the peak centered at 575 nm, and device II fabricated by **4** exhibited yellow-green emission with the peak centered at 557 nm, which showed a red shift compared with device III fabricated by Zn(mHQ)$_2$, as shown in Figure 2a. Note, however, that the electroluminescence (EL) spectrum of devices fabricated using **4** showed a shift toward shorter wavelengths with a maximum at 557 nm compared to a maximum of 607 nm in solid-state photoluminescence spectra, and a shift toward longer wavelengths compared to a maximum of 527 nm in solution photoluminescence spectra. It may be excimer formation between adjacent lumophors in solid and intermolecular processes that are greatly reduced in dilute solution, leading to a blue-shift of the emission spectra.

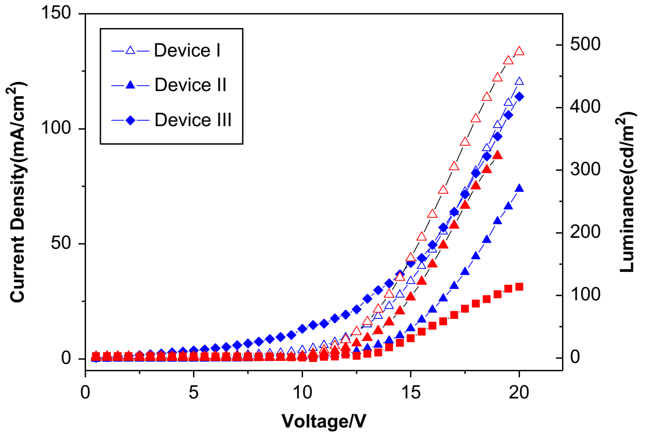

Figure 3. Current density–voltage–luminance curves (*J*–*V*–*L*) characteristics for the three single-layer OLEDs. Red lines stand for the current density-voltage curves and blue lines stand for luminance-voltage curves.

Figure 3 shows the current density and luminance versus applied voltage characteristics for the three single-layer devices. The single-layer device I exhibited good performance with a maximum

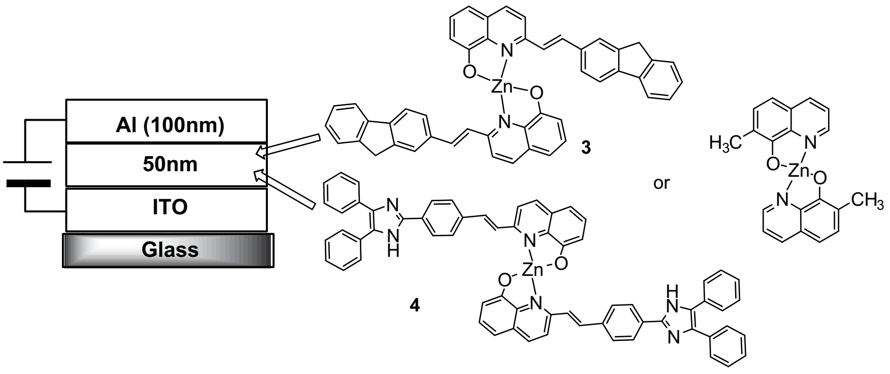

Figure 2. Structure of the single-layer OLED.

luminance of 489 cd m^{-2} and a luminance efficiency of up to 0.41 cd A^{-1}, whereas device II exhibited a maximum luminance of 323 cd m^{-2} and a luminance efficiency of up to 0.54 cd A^{-1}, which are higher than the device III (114 cd m^{-2}, 0.1 cd A^{-1}). From the results, we can see that fluorene-based complex **3** and imidazole-based complex **4** are good light-emitting materials to fabricate single-layer OLED. The curves indicate that the luminance under the same potential increased in the order **III**<**II**<**I**, suggesting that using these light-emitting materials as the active organic layer can enhance the performance in single-layer device. The external quantum efficiency, η_{ext}, which is defined as the ratio of number of emitted electrons in the forward viewing direction to the number of injected electrons, is useful in understanding the fundamental physical mechanisms responsible for light emission within an OLED. In this paper, the external quantum efficiencies of the three devices are 0.56, 0.62 and 0.2 at maximum luminance, respectively. However, in the present study, the conditions have not been optimized, such as electrodes and the thickness of the active organic layer, so the current densities in the devices are low, which may be due to the insufficiency of the carriers.

3. Conclusion

Two newly synthesized light-emmting materials bis-[2′-2″-(9*H*-fluoren-2-yl)-vinyl-8-hydroxyquinoline] zinc(II) (**3**) and bis-[2′-4″-(4,5-diphenyl-1*H*-imidazol-2-yl)styryl-8-hydroxyquinoline] zinc(II) (**4**) containing 8-hydroxyquinoline and fluorene or imidazole have been used in the single-layer OLEDs for the first time. The two materials have good stabilities and their thermal decomposition temperatures are 395 °C and 435 °C. They also exhibit better fluorescence quantum yields (0.168 and 0.273) than Alq_3 (0.116). The optical properties of these complexes are influenced by the styryl substituents, and emit orange in solid state, which is red-shifted compared with the solution emission. The two complexes exhibit intriguing ambipolar carrier transport properties and can be used to fabricate single-layered EL devices possessing good performance, compared with that of $Zn(mHQ)_2$. Because of good luminescence properties and high thermal stability, the two compounds **3** and **4** show great potential for use as an active organic layer in single-layer OLEDs.

4. Experimental section

4.1. Chemicals and instruments

4-(4,5-Diphenyl-1*H*-imidazol-2-yl)benzaldehyde was synthesized by Weidenhagen reaction according to the literature.[26] 2-Methyl-8-hydroxyquinoline and 2-Fluorenecarboxaldehyde were purchased from Tokyo Kasei Kogyo Co. All chemicals were used without further purification. Solvents were distilled prior to use. All reactions were carried out under nitrogen atmosphere. The experimental course was monitored by thin layer chromatography (TLC). Melting points were determined using an XT-4 microscope melting point inspection instrument and the thermometer was uncorrected. Infrared (IR) spectra were recorded on a BRUKE Tensor27 Fourier transform infrared (FTIR) spectrometer and measured as KBr pellets. ^{1}H nuclear magnetic resonance (^{1}H NMR) spectra and ^{13}C nuclear magnetic resonance (^{13}C NMR) were recorded on a Bruker DRX-400 MHz spectrometer with TMS as internal standard and DMSO-d_6/$CDCl_3$ as solvent. ESI-MS spectra were performed with DECA XP MAX LCQ. UV visible spectra were measured with a Shimadzu UV-2550 spectrophotometer. Fluorescence spectra and efficiency were acquired at 28 °C on an Edinburgh Co. FL920 spectrofluorimeter. Thermal analyses were performed with a TGA/DSC Q600 SDT TA Instuments apparatus at an elevation temperature of 10 °C/min.

4.2. Synthesis

*4.2.1. Synthesis of 2′-2″-(9H-fluoren-2-yl)-vinyl-8-hydroxyquinoline (**1**) and 2′-4″-(4,5-diphenyl-1H-imidazol-2-yl)styryl-8-hydroxyquinoline (**2**)*

A mixture of 2-methyl-8-hydroxyquinoline (0.954 g, 6 mmol), 2-fluorene carboxaldehyde (1.164 g, 6 mmol) or 4-(4,5-diphenyl-1*H*-imidazol-2-yl)benzaldehyde (1.95 g, 6 mmol)and acetic anhydride (25 mL) was stirred and heated at 125 °C–130 °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. The solid was washed with water twice, and then purified by silica gel (100–200 mesh) column chromatography using ethyl acetate/petroleum ether as an eluent.

2′-2″-(9*H*-Fluoren-2-yl)-vinyl-8-hydroxyquinoline (**1**): yellow solid. Yield 0.715 g, 75%; mp 160–162 °C; R_f=0.45 (ethyl acetate/petroleum ether, V:V=1:5); IR (KBr) ν: 3407.69, 3050.32, 2927.29, 1633.02, 1555.06, 966.25; MS (ESI) *m*/*z* (%): 337.40 [$(M+H)^+$]; ^{1}H NMR ($CDCl_3$): δ 8.13 (d, *J*=8.80 Hz, 1H), 7.67 (d, *J*=8.80 Hz, 2H), 7.58 (d, *J*=7.2 Hz, 1H), 7.178 (d, *J*=6.4 Hz, 1H), 7.28–7.36 (m, 2H), 7.38–7.48 (m, 3H), 7.79–7.85 (m, 4H), 3.97 (s, 2H); ^{13}C NMR ($CDCl_3$): δ 153.72, 151.98, 143.82, 143.67, 142.54, 141.20, 137.96, 136.34, 134.95, 134.71, 129.71, 128.37, 127.37, 127.29, 127.05, 126.58, 125.29, 125.08, 123.55, 120.32, 120.09, 117.65, 110.13, 36.83. Calculated for $C_{24}H_{17}NO$: C, 85.94%; N, 4.18%; H, 5.11%. Found: C, 85.90%; N, 4.20%; H, 5.15%.

2′-4″-(4,5-Diphenyl-1*H*-imidazol-2-yl)styryl-8-hydroxyquinoline (**2**): yellow solid. Yield 0.95 g, 34%, mp 228–230 °C, R_f=0.22 (ethyl acetate/petroleum ether, V:V=3:8). MS (ESI) *m*/*z*: 467[$(M+H)^+$]; UV (in DMF) $\lambda_{\max}$: 294, 384 nm; IR (KBr, cm^{-1}) ν: 3342.28, 3049.45, 1623.94, 1506.72, 1243.32, 968.51, 836.42, 766.45, 697.62; ^{1}H NMR (DMSO-d_6) δ (ppm): 12.85 (s, 1H), 8.32 (d, *J*=8.4 Hz, 1H), 8.19 (m, 3H), 7.84 (d, *J*=13.5 Hz, 1H), 7.82 (d, *J*=13.5 Hz, 1H), 7.59–7.33 (m, 14H), 7.11 (d, *J*=7.2 Hz, 1H); ^{13}C NMR (101 MHz, DMSO) δ 153.81, 153.38, 145.65, 138.63, 136.95, 136.71, 134.26, 130.66, 128.85, 128.61, 128.13, 128.00, 127.51, 126.02, 121.49, 118.01, 111.64. Calculated for $C_{32}H_{23}N_3O$: C, 82.56%; N, 9.03%; H, 4.98%. Found: C, 82.95%; N, 8.98%; H, 5.07%.

*4.2.2. Synthesis of the metal complexes **3** and **4***

$Zn(CH_3COO)_2$ (0.092 g, 0.5 mmol) was dissolved in tetrahydrofuran (15 mL), and then added drop-wise to the solution of compound **1** (0.336 g, 1 mmol) and **2** (0.466 g, 1 mol) in tetrahydrofuran (50 mL). After stirred for 24 h under nitrogen atmosphere, yellow precipitates were obtained, and then filtered off, washed with tetrahydrofuran and methanol 3–5 times respectively, and dried in vacuum. Due to the solubility of the zinc(II) complexes **3** and **4** is not good, ^{1}H NMR spectra didn't obtain.

Bis-[(*E*)-2′-2″-(9*H*-fluoren-2-yl)-vinyl-8-hydroxyquinoline] zinc (II) (**3**). Yield 0.33 g, 91%, mp >300 °C, IR (KBr) ν: 3050.40, 2920.55, 2854.32, 1622.48, 1555.06, 1102.20, 520.50, 468.06; FABMS: *m*/*z*: 732.18[$(M-H)^+$]. Calculated for $C_{48}H_{32}N_2O_2Zn$: C, 78.53%; N, 3.82%; H, 4.39%. Found: C, 78.23%; N, 3.80%; H, 4.33%.

Bis-[2′-4″-(4,5-diphenyl-1*H*-imidazol-2-yl) styryl-8-hydroxy quinoline] zinc(II) (**4**). Yield 0.32 g, 64%, mp >300 °C, IR (KBr, cm^{-1}): 3048.08, 2920.45, 2854.33, 1628.11, 1598.22, 1503.66, 1433.51, 1102.08, 960.99, 834.25, 696.30; FABMS: *m*/*z*: 993. Calculated for $C_{64}H_{44}N_6O_2Zn$: C, 77.30%; N, 8.45%; H, 4.46%. Found: C, 77.41%; N, 8.39%; H, 4.38%.

4.3. Devices fabrication and charaterization

In order to investigate the EL properties of bis-[(*E*)-2′-2″-(9*H*-fluoren-2-yl)-vinyl-8-hydroxyquinoline] zinc(II) (**3**) and bis-[2′-4″-(4,5-diphenyl-1*H*-imidazol-2-yl) styryl-8-hydroxyquinoline] zinc(II) (**4**), two single-layer devices (as shown in Fig. 1) of tin oxide(ITO)/**3** or

4/Al were fabricated with a contrastive device fabricated by bis(2-methyl-8-hydroxyquinoline)zinc(II) [i.e., Zn(mHQ)], marked with device I, II and III. 150-nm thick indium-tin oxide(ITO)/glass substrate with a resistance of 15 Ω per square was purchased form China Southern Glass Holding Co. Ltd., and were pretreated with ultrasonic cleaning using acetone, methanol, distilled water, and anhydrous ethanol, respectively, and then were dried by nitrogen gas. All devices were fabricated by thermal deposition of the organic materials and aluminium onto ITO/glass substrate below a pressure of 5×10^{-4} Pa without breaking vacuum. The organic materials and the cathode metals were evaporated at rates of 0.1–0.2 nm/s. None of the devices were encapsulated, and measurements were carried out at room temperature under ambient conditions. The brightness was measured with a ST-86LA spot photometer and a close-up lens providing a focal spot of 5 mm. The current density-voltage-luminance characteristics were recorded on a computer-controlled Keithley 2400 Sourcemeter unit with a calibrated silicon diode.

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Supplementary data

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.tet.2009.06.025.

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