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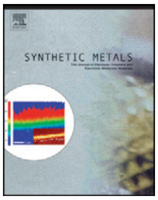

New rhenium complexes containing 4,5-diazafluorene ligand for high-efficiency green electrophosphorescence

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ABSTRACT

Two novel tricarbonyl rhenium complexes featuring 4,5-diazafluorene (DF)-based ligand, i.e., Re-DF and Re-EPDF (EPDF, 9,9-di-(4-ethoxyphenyl)-9-*H*-4,5-diazafluorene), were designed, synthesized and characterized by ^{1}H NMR and mass spectroscopy. The green organic light-emitting diodes (OLEDs) based on these complexes with the configuration of ITO/*m*-MTDATA (10 nm)/NPB (20 nm)/CBP: Re-complex (30 nm)/Bphen (10 nm)/Alq_3 (30 nm)/LiF (1 nm)/Al (100 nm) were fabricated. The devices based on Re-DF showed a maximum current efficiency of 20.7 cd/A and a peak luminance of 2506 cd/m^2, respectively. And the Re-EPDF doped devices exhibited a maximum current efficiency of 13.5 cd/A and a luminance of 3208 cd/m^2. Moreover, the 20 wt.% Re-EPDF doped device still provided a maximum current efficiency of 13.2 cd/A.

1. Introduction

Transition-metal-based phosphorescent materials have received considerable attention on account of their potential applications as highly efficient electroluminescence (EL) emitters in organic light-emitting diodes (OLEDs) [1–5]. Under electrical excitation, both singlet and triplet excitons are utilized for light emission in these complexes so that nearly 100% internal quantum efficiency can be obtained. The highly efficient phosphorescence emission is attributed to the singlet-to-triplet intersystem crossing induced by heavy metal atom and the large enhancement of radiative decay rate from the resulting triplet state. Therefore, immense efforts have been devoted to exploiting the transition-metal-based complexes which could be used as dopant emitters in OLED devices. Among those complexes, quite a few Os(II) [6,7], Ir(III) [4,8–10] and Pt(II) [11,12] complexes have been reported to be very effective materials in OLEDs.

In order to further extend the scope, many works have been focused on the rhenium (I) (Re^I) analogues [13,14], of which the d^6 electronic configuration is coincident with that of the corresponding Os(II) and Ir(III) systems. In addition, Re^I complexes present relatively short excited-state lifetime (<1 μs), excellent thermal and photochemical stability and so on [15–17]. Thus they are also anticipated to exhibit excellent EL performances. Although many rigid 1,10-phenanthroline derivatives were utilized to prepare tricarbonyl Re-complexes which had been used as highly efficient emitters in OLEDs [18–21], they emitted almost yellow-orange colors. For full-color displays, the emitters with tunable emission color are crucial. Extending or reducing the π-conjugation of the ligand has been proved to be an effectual way to tune the emission color of Ir^{III} complexes [22,23]. So this inspires us to initiate the ligand, 4,5-diazafluorene (DF), to tune the emission color of Re-complexes. The methylene bridge of DF distorts the bipyridine portion of the molecular so as to reduce the nitrogen–metal overlap [24]. Furthermore, modification of DF by introducing two strategically positioned ethoxyphenyl groups as steric spacer can minimize the intermolecular action at high doping concentration, leading to highly efficient EL performances [25].

In this paper, two related Re-complexes containing DF-based ligand, i.e., Re-DF and Re-EPDF (EPDF = 9, 9-di-(4-ethoxyphenyl)-9-*H*-4,5-diazafluorene), were designed, synthesized, characterized, and used as highly efficient green phosphorescence emitters in OLEDs. As a result, the green OLEDs based on Re-DF and Re-EPDF exhibited the maximum current efficiency of 20.7 and 13.5 cd/A, the peak luminance of 2506 and 3208 cd/m^2, respectively.

2. Experimental

All chemical reagents were used as received without further purification unless otherwise mentioned and the solvents were

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dried by standard methods prior to use. All reactions were monitored by HPLC. ^{1}H NMR spectra were obtained on Bruker AC 500 spectrometer using tetramethylsilane (TMS) as the internal standard. Mass spectroscopy (MS) was performed on Agilent 1100 LC/MSD Trap VL. UV–vis. absorption spectra were recorded using a PerkinElmer Lambda 900 spectrophotometer. Photoluminescence (PL) spectra were measured on a PerkinElmer LS 55 fluorescence spectrophotometer. The excited-state lifetime was detected by a system equipped with a TDS 3052 digital phosphor oscilloscope pulsed Nd:YAG laser with a THG 355 nm output. Cyclic voltammetry experiments were conducted using a CHI832B electrochemical analyzer with a scan rate of 200 mV/s at room temperature (RT). All measurements were performed with a conventional three-electrode configuration, a polished Pt plate as the working electrode, a Pt mesh as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode with 0.1 M tetra-*n*-butylammonium hexafluorophosphate as the supporting electrolyte. Prior to each electrochemical measurement, the solutions were purged with nitrogen to remove the dissolved oxygen.

2.1. The synthesis of Re-complexes

2.1.1. Synthesis of 4,5-diazafluoren-9-one (DFO) [24]

1,10-Phenanthroline (5.9 g, 30 mmol) and KOH (3.06 g, 55 mmol) were added to 200 mL of water and brought to reflux. Potassium permanganate (15.2 g, 95 mmol) in 125 mL of water was added dropwise to the refluxing mixture. After addition, the solution was refluxed for 0.5 h and filtered to remove MnO_2. When the solution was cooled, crude products were precipitated as yellow needles. The desired product was obtained by recrystallization with water. Yield: 27%. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ (ppm): 8.8 (d, J = 2.48 Hz, 2H), 8.0 (d, J = 1.02 Hz, 2H), 7.4 (t, J = 3.73 Hz, 2H); MS (APCI): *m/z* 183 [M + H$^+$].

2.1.2. Synthesis of DF

DFO (1.82 g, 10 mmol) and hydrazine hydrate (85%, 10 mL) were placed in a steel bomb and heated to 170 °C for 20 h. When the solution was cooled, a white solid was filtered from the dark red liquid and then washed with water. The white prisms were obtained by recrystallization in 58% yield. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ (ppm): 8.765 (d, J = 4.875 Hz, 2H), 7.911 (d, J = 7.72 Hz, 2H), 7.322 (t, J = 3.809 Hz, 2H), 3.900 (s, 2H); MS (APCI): *m/z* 169.0 [M + H$^+$].

2.1.3. Synthesis of 9, 9-di-(4-hydroxyphenyl)-9-H-4,5-diazafluorene (HPDF)

DFO (1.82 g, 10 mmol) was dissolved in molten phenol (9.4 g, 100 mmol). The mixture was heated at 50 °C and then dry hydrogen chloride was bubbled for about 3 h. The mixture was reacted for 8 h and then diluted with NaOH solutions to pH 8 to give a white solid, which was collected and washed with hot water. The product was further purified by crystallization from ethanol to give white crystal. Yield: 40%. ^{1}H NMR (500 MHz, DMSO-d^6, TMS): δ (ppm): 6.66 (d, 4H), 6.92 (d, 4H), 7.42 (dd, 2H), 7.93 (d, 2H), 8.67 (d, 2H), 9.44 (s, 2H); MS (APCI): *m/z* 353.2 [M + H$^+$].

2.1.4. Synthesis of EPDF

To a mixture of HPDF (0.352 g, 1.0 mmol) dissolved in 5.0 mL of *N*,*N*-dimethylformamide (DMF) was added potassium carbonate (0.552 g, 4.0 mmol). The mixture was heated at 120 °C for 1 h, and then bromoethane (0.224 mL, 3.0 mmol) was added. The mixture was further reacted for 10 h and then cooled. The reaction mixture was quenched with 0.2 M HCl and extracted with CH_2Cl_2. The extract was washed with water three times, and then was dried over anhydrous Na_2SO_4. After removal of the solvent, the residue was subjected to purification by chromatography on silica gel to afford a white powder. Yield: 50.8%. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ (ppm): 8.723 (d, J = 4.735 Hz, 2H), 7.748 (d, J = 7.73 Hz, 2H), 7.271 (t, J = 6.19 Hz, 2H), 7.074 (d, J = 8.58 Hz, 4H), 6.759 (d, J = 8.62 Hz, 4H), 3.977 (m, 4H), 1.384 (t, 6H); MS (APCI): *m/z* 409.3 [M + H$^+$].

2.1.5. Synthesis of Re-DF

DF (35 mg, 0.21 mmol) and pentacarbonylbromorhenium (81.2 mg, 0.2 mmol) were refluxed in 20 mL of toluene for 6 h under nitrogen atmosphere. After the mixture was cooled to RT, the solvent was removed under reduced pressure. The resulting yellow solid was purified by chromatography on silica gel. Yield: 91%. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ (ppm): 8.796 (d, J = 5.19 Hz, 2H), 8.105 (d, J = 7.645 Hz, 2H), 7.551 (t, J = 6.46 Hz, 2H), 4.333 (s, 2H).

2.1.6. Synthesis of Re-EPDF

Re-EPDF was prepared by similar procedures described for the synthesis of Re-DF. Yield: 89%. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ (ppm): 8.7287 (d, J = 5.09 Hz, 2H), 7.912 (d, J = 7.73 Hz, 2H), 7.48 (t, J = 6.43 Hz, 2H), 7.178 (d, J = 8.46 Hz, 2H), 6.963 (d, J = 8.505 Hz, 2H), 6.859 (d, J = 8.43 Hz, 2H), 6.763 (d, J = 8.495 Hz, 2H), 3.99 (m, 4H), 1.395 (t, 6H).

2.2. Fabrication and EL measurements of OLEDs

The devices were fabricated on pattered indium tin oxide (ITO) coated glass substrates with a sheet resistance of 25 Ω/sq by thermal evaporation in vacuum chamber below 2×10^{-4} Pa. The devices were prepared as the following structure: ITO/*m*-MTDATA (10 nm)/NPB (20 nm)/CBP: Re-complex (30 nm)/Bphen (10 nm)/Alq_3 (30 nm)/LiF (1 nm)/Al (100 nm), in which ITO was used as the anode, 4,4′,4″-tris[3-methylphenyl(phenyl)amino]triphenylamine (*m*-MTDATA), 4′-bis[*N*- (1-naphthyl)-*N*-phenyl-amino] biphenyl (NPB), 4,7-diphenyl-1,10-phenanthroline (Bphen), and tris(8-hydroxyquinoline) aluminum (Alq_3) were used as hole injection layer, hole transporting layer, exciton blocking layer and electron transporting layer, respectively, and LiF/Al as the cathode. Deposition rates and thicknesses of the layers were monitored in situ using oscillating quartz monitors. The evaporating rates were kept at 2–3 Å/s for organic layers and LiF layer, and 10 Å/s for Al cathode, respectively. EL spectra were measured with a Hitachi MPF-4 fluorescence spectrophotometer. The luminance–current–voltage (*L*–*I*–*V*) characteristics of the devices were measured with a 3645 dc power supply combined with a 1980a spot photometer and recorded simultaneously with measurements. All measurements were carried out at RT under ambient conditions.

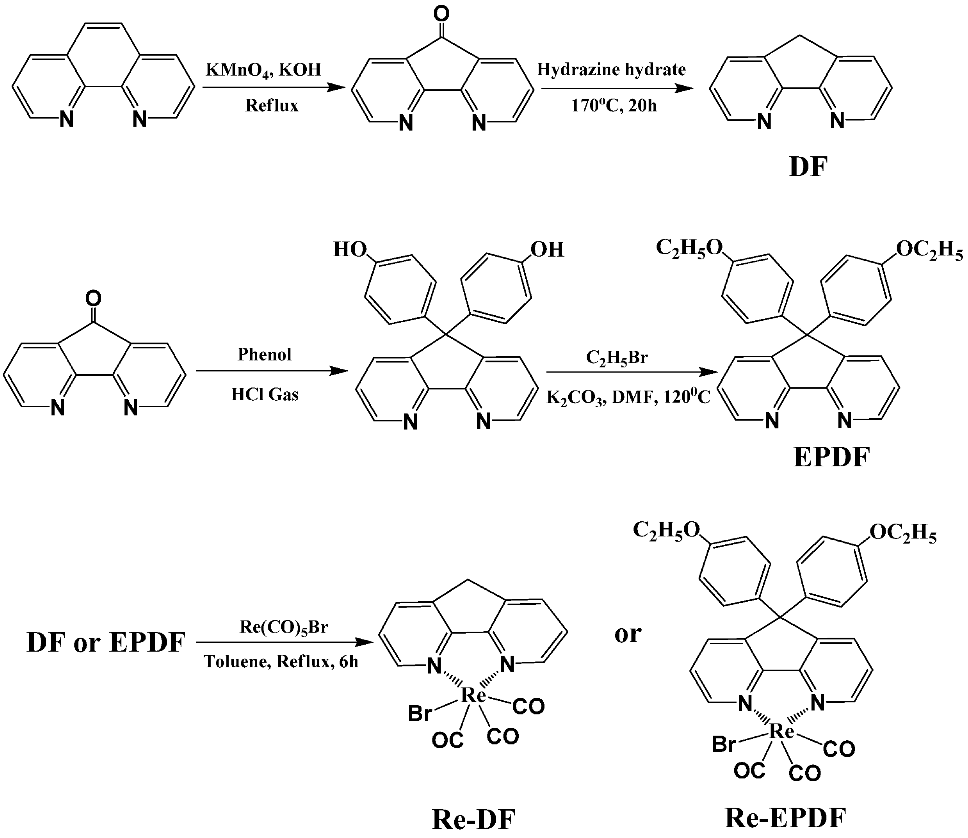
3. Results and discussion

3.1. Synthesis and characterization

The synthetic pathways of the Re-complexes are outlined as shown in Scheme 1. The ligand DF can be obtained from Wolff–Kishner reaction between 4,5-diazafluoren-9-one and hydrazine hydrate in moderate yield. In fact a concomitant dimerized by-product was observed, but it was easily removed from DF. And then the EPDF was conveniently prepared using the similar procedures [26]. Finally, the corresponding Re(I) complexes were obtained by direct complexation of DF and EPDF with $Re(CO)_5Br$ in 85–91% yield. The structures of those compounds were confirmed by ^{1}H NMR and MS.

3.2. Photophysical properties

The UV–vis absorption and PL spectra of Re-DF and Re-EPDF in dichloromethane solutions are shown in Fig. 1. And the PL spectrum of CBP film is also given in Fig. 1. By comparison to the absorption



Scheme 1. Synthetic route of two complexes.

of the free DF and EPDF ligands, the intense UV absorption bands below 350 nm are assigned to spin-allowed $^1\pi$–π^* transitions from the cyclometalated ligands. The relatively weak absorption bands in the range 350–480 nm are tentatively attributed to an admixture of metal-to-ligand charge transfer states, $d\pi(\mathrm{Re}) \rightarrow \pi^*(\mathrm{ligand})$ (^{1}MLCT and ^{3}MLCT). Unpon irradiation with 360 nm light, both Re-DF and Re-EPDF emit strong green PL at around 546 nm in dichloromethane solutions, which can be predominantly assigned to radiative transitions from ^{3}MLCT level [27]. The PL quantum yields of Re-DF and Re-EPDF measured in deaerated dichloromethane are 0.012 and 0.009 by using quinine sulfate as a reference which has a quantum yield of 0.546 [28].

3.3. Electrochemical properties

The electrochemical behaviors of Re-DF and Re-EPDF were investigated by cyclic voltammetry (CV) using a SCE as the reference electrode. The cyclic voltammograms of Re-DF and Re-EPDF are shown in Fig. 2 and exhibit irreversible metal-centered oxidation and ligand-based reduction in CH_2Cl_2 solutions. Re-DF shows irreversible anodic waves at $E_{1/2} = +1.27$ V with an onset oxidation potential of +1.16 V vs. SCE, and irreversible cathodic waves at $E_{1/2} = -1.44$ V with an onset reduction potential of −1.36 V vs. SCE. The anodic waves are associated with a Re^{I}-based oxidation process ($\mathrm{Re}^{\mathrm{I}}/\mathrm{Re}^{\mathrm{II}}$), and the cathodic waves are associated with a ligand

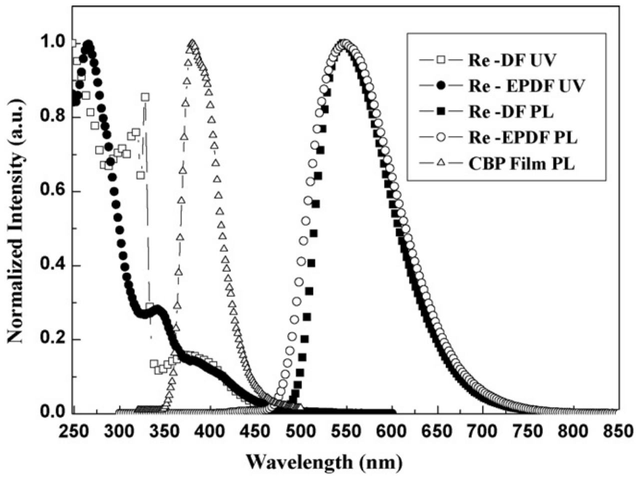


Fig. 1. Absorption and PL spectra of the two complexes and PL spectrum of CBP film.

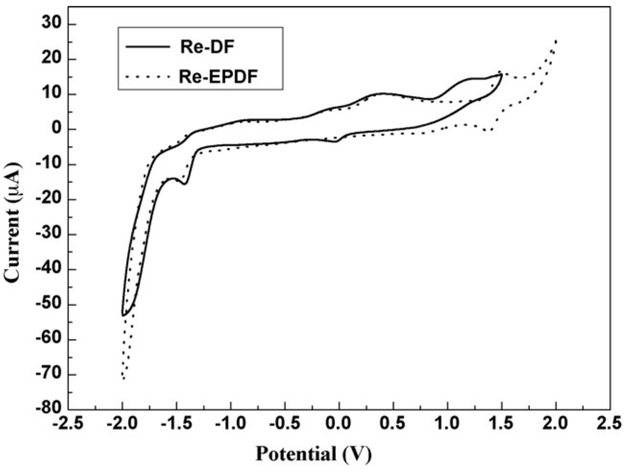


Fig. 2. Cyclic voltammograms of Re-DF and Re-EPDF measured in CH_2Cl_2 (vs. SCE) at a scan rate of 200 mV/s.

Table 1
The energy level data of Re-DF and Re-EPDF.

Complex	$E_{\mathrm{onset}}^{\mathrm{Ox}}$(V)	E_{onset}^{Red} (V)	E_{HOMO} (eV)	E_{LUMO} (eV)	$E_{\mathrm{g}}^{\mathrm{el}}$ [a] (eV)	$E_{\mathrm{g}}^{\mathrm{opt}}$ [b] (eV)
Re-DF	+1.16	−1.36	−5.91	−3.39	2.52	2.59
Re-EPDF	+1.26	−1.31	−6.01	−3.44	2.57	2.62

[a] $E_{\mathrm{g}}^{\mathrm{el}}$ (electrochemical band gap) = $E_{\mathrm{LUMO}} - E_{\mathrm{HOMO}}$.

[b] $E_{\mathrm{g}}^{\mathrm{opt}}$ is optical band gap from the optical absorption spectra.

(L)-based reduction process ($[\mathrm{Re^IBr(CO)_3(L)}]/[\mathrm{Re^IBr(CO)_3(L^{\cdot-})}]^-$) [29,30]. Re-EPDF shows a similar redox behavior. The energy levels of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) are calculated from the onset oxidation ($E_{\mathrm{onset}}^{\mathrm{Ox}}$) and reduction potentials (E_{onset}^{Red}) with the formula $E_{\mathrm{HOMO}} = -4.75 - E_{\mathrm{onset}}^{\mathrm{Ox}}$ (−4.75 V for SCE with respect to the zero vacuum level) and $E_{\mathrm{LUMO}} = -4.75 - E_{\mathrm{onset}}^{Red}$ to be −5.91 and −3.39 eV for Re-DF, −6.01 and −3.44 eV for Re-EPDF, respectively. The energy band gaps of Re-DF and Re-EPDF are thus determined to be 2.52 and 2.57 eV, which are close to those (2.59 and 2.62 eV) obtained from the optical absorption spectra. The detailed energy level data of Re-DF and Re-EPDF are summarized in Table 1.

3.4. EL properties

The Re-complexes doped devices were fabricated in order to investigate their EL properties. We observed that the doping concentrations of Re-complexes in the devices greatly affected EL performances. Fig. 3 presents the EL spectra of 10 wt.% Re-complexes doped devices at 12 V. The OLEDs based on Re-DF and Re-EPDF exhibit broad, green phosphorescence emissions at 545 and 544 nm which basically coincide with their PL spectra. The close resemblance between the PL and EL spectra suggests that two complexes are thermally stable in vacuum and the EL emissions appear to originate from the ^{3}MLCT excited-state which is the same as that of PL process. As shown in Fig. 1, there is a sufficient overlap between PL spectrum of CBP film and MLCT absorption bands of two complexes, indicating that the efficient Förster energy transfer based on the doped devices maybe occurs. The CIE coordinates of EL devices based on Re-DF and Re-EPDF are (0.392, 0.572) and (0.393, 0.562), respectively, which are almost independent of the applied bias and doping concentrations. In addition, it is found that the emission wavelengths cannot be shifted by introducing two steric ethoxyphenyl groups on the 9th position of DF.

Fig. 4 shows *L–I–V* characteristics of 10 wt.% Re-DF and 20 wt.% Re-EPDF doped devices. The 10 wt.% Re-DF doped devices exhibits the peak luminance of 2506 cd/cm^2 at 16 V, and for 20 wt.% Re-EPDF doped device the maximum luminance is 3208 cd/cm^2 at 16 V. The turn-on voltages of all devices are lower than 5 V.

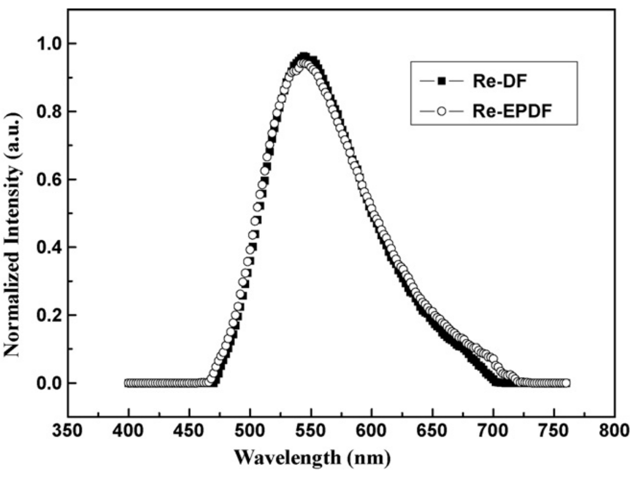


Fig. 3. EL spectra of 10 wt.% Re-complexes based devices at 12 V.

Current efficiency vs. current density characteristics of two Re-complexes based devices are shown in Fig. 5. Both 15 wt.% Re-DF and Re-EPDF doped devices exhibit the maximum current efficiencies of 20.7 cd/A at 13 cd/cm^2 and 13.5 cd/A at 38 cd/cm^2, respectively. The EL efficiencies of the devices decrease slowly at first with increasing current, implying that the effect of triplet-triplet annihilation is almost neglectable probably due to the short excited-state lifetime, i.e., 164 ns for Re-DF and 157 ns for Re-EPDF. It is well known that the saturation of the phosphorescent excited-sites is responsible for the fast decrease of the EL efficiencies at high current density exceeding 100 mA/cm^2 [31]. Just as indicated in inset of Fig. 5, the 20 wt.% Re-DF doped device exhibits a maximum current efficiency of 8.0 cd/A at 42 cd/cm^2, whereas the 20 wt.% Re-EPDF doped device behaves a maximum current efficiency of 13.2 cd/A at 78 cd/cm^2.

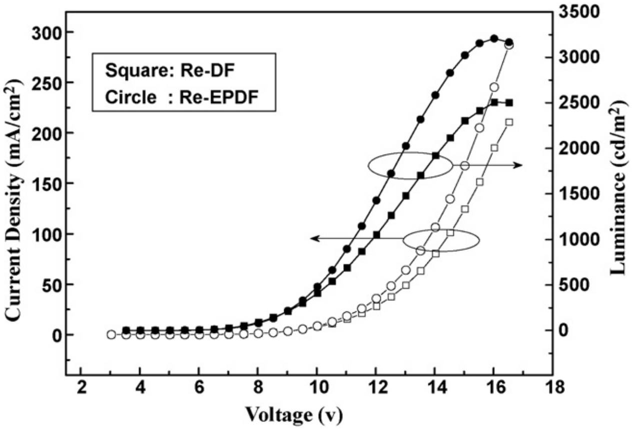


Fig. 4. *L–I–V* characteristics of the devices based on 10 wt.% Re-DF and 20 wt.% Re-EPDF.

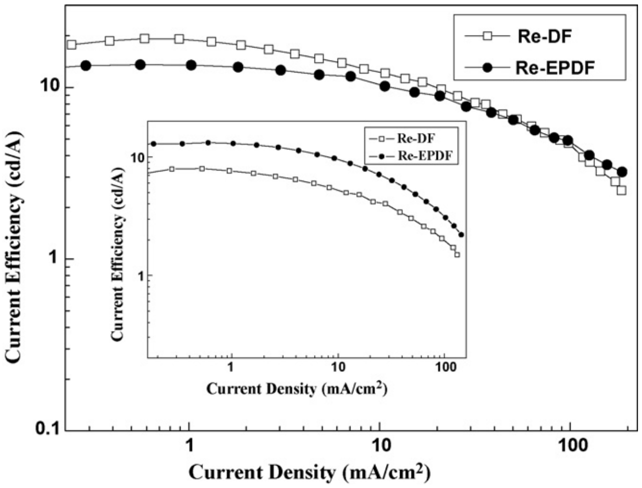


Fig. 5. Current efficiency vs. current density characteristics of 15 wt.% Re-complexes based devices. Inset: current efficiency vs. current density characteristics of 20 wt.% Re-complexes based devices.

Interestingly, the maximum current efficiency of the 20 wt.% Re-EPDF doped device is almost invariable compared with that of the 15 wt.% Re-EPDF doped device. Furthermore, the efficiencies of Re-EPDF doped device are greatly higher than those of the Re-DF doped device at the same 20 wt.% doping level. The excellent performances of the device with 20 wt.% Re-EPDF may be ascribed to the suppressed concentration quenching which results from the influence of attaching two repulsively steric ethoxyphenyl groups on the 9th position of DF.

4. Conclusions

In summary, we reported the synthesis and characterization of two novel rhenium complexes, i.e., Re-DF and Re-EPDF, employing 4,5-diazafluorene derivatives as ligands. The highly efficient green electrophosphorescent devices based on two complexes have been demonstrated. We find that the introduction of two ethoxyphenyl groups on the 9th position of DF cannot alter the emission wavelength of Re-EPDF in comparison to Re-DF, while the concentration quenching effect is greatly alleviated. The devices based on Re-DF exhibit a maximum current efficiency of 20.7 cd/A which is comparable to that of the best Re-complex based device reported in Ref. [32], suggesting that Re-DF is a promising candidate for green OLEDs applications although the device's luminance is not satisfactory. Our results also indicate that molecular modification is an effective method to reduce the concentration quenching and accordingly improve the EL efficiency of the phosphorescent devices.

Acknowledgements

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