

Communication

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Communication

Two Novel Neutral Cyclometalated Iridium(III) Complexes Based on 10,11,12,13Tetrahydrodibenzo[a,c]phenazine for Efficient Red Electroluminescences

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Abstract: Two novel neutral phosphorescent iridium(III) complexes (Ir1 and Ir2) were rationally designed and synthesized with high yields using 10,11,12,13-tetrahydrodibenzo[a,c]phenazine as the main ligand. The two complexes showed bright red phosphorescence (625 nm for Ir1, and 620 nm for Ir2, in CH₂Cl₂), high luminescence quantum efficiency (0.32 for Ir1, and 0.35 for Ir2), obvious solvatochromism and good thermostability. Then, they were used to fabricate high-efficiency red OLEDs via vacuum evaporation, the maximum CE, PE, and EQE of the red devices based on Ir1 and Ir2 are 13.47/15.22 cd/A, 10.35/12.26 lm/W, and 10.08/7.48%, respectively.

Keywords: Iridium(III) complexes; red emission; phosphorescence; OLEDs

1. Introduction

In the last two decades, OLEDs (Organic Light-Emitting Diodes), as a potential technology for solid-state lighting and displays, have attracted more and more attentions in academia and industry due to their unique characteristics, such as wide viewing angle, ultra-thin thickness, full-color displays, flexibility, and so on [1–8]. Same as device structures, materials especially the emissive layer (EML) materials in OLEDs play a vital role on the device performances. As we know, there exists 25% singlet excitons and 75% triplet excitons resulted from the electrical excitation. However, transitional fluorescent materials can only utilize 25% singlet excitons, which limits the internal quantum efficiency [9,10]. In order to achieve high-efficiency devices, phosphorescent transition-metal complexes, like iridium(III) complex, platinum(II) complex, are usually introduced into OLEDs as EML owing to their significant properties, such as tunable phosphorescent wavelength, abundant triplet-excited states, high luminescence quantum efficiency, large spin-orbital coupling (SOC) constant and excellent photo- and chemical stability [11,12]. Moreover, heavy atom effect can effectively enhance the progress of intersystem crossing from excited-singlet state to excited-triplet state. For instance, in the iridium(III) complex-based OLEDs, both excited-singlet and excited-triplet state excitons can be captured simultaneously, thus obtaining 100% internal quantum efficiency theoretically [13,14]. Red emission is a crucial factor in three primary colors, and has aroused tremendous interests, thus designing and fabricating novel efficient red iridium(III) complexes are of great significance and practical values [15,16].

Recently, several new red iridium(III) complexes were reported to achieve high-performance OLEDs [17–20]. For example, in 2019, Zheng et al. reported two rapid room temperature synthesis of red iridium(III) complexes with Ir–S–P–S structures for efficient OLEDs with a maximum power

efficiency of 44.40 lm/W and a maximum external quantum efficiency of 24.90%. Zuo et al. reported a series of iridium(III) complexes based on thienylpyridine to fabricate yellow-to-deep red OLEDs with low efficiency roll-off. Despite great efforts have been devoted to designing and synthesizing new red iridium(III) complexes, the most common approach is to enlarge the degree of conjugation or introduce donor and acceptor groups, thereby reducing the energy gap between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO), the obtained complexes are often with poor solubility and low yield. Thus, it is necessary to explore novel efficient red iridium(III) complexes with appreciable yields and simple structure, and it is still an arduous assignment owing to the lacking versatility of the reported luminogens.

Herein, two high-efficient red neutral iridium(III) complexes (Ir1 and Ir2) using 10,11,12,13-tetrahydrodibenzo[a,c]phenazine as the main ligand, and acetylacetone and picolinic acid as auxiliary ligand, respectively, have rationally designed and synthesized with high yield and good solubility. Both complexes showed excellent photophysical properties and intense red phosphorescence with high luminescent quantum efficiency. Then Ir1 and Ir2 were used to fabricate red OLEDs via vacuum evaporation. It is worth noting that, different auxiliary ligand showed great influence on the device performances. The maximum emission peaks of the devices based on Ir1 and Ir2 were at 612 and 591 nm, respectively. Among them, the maximum brightness and maximum EQE of the Ir1-based device were 25970 cd/m² and 10.08%, respectively.

2. Results

2.1. Structural Characterization

Complexes Ir1 and Ir2 were rationally designed and synthesized as illustrated in Figure 1. At first, the main ligand 10,11,12,13-tetrahydrodibenzo[a,c]phenazine was synthesized through cyclization reaction between 9,10-phenanthraquinone and 1,2-diaminocyclohexane at 80°C in ethanol, and obtained by recrystallization with high yield. Then, the cyclometalated iridium(III) dimers were prepared in the light of the reported literatures. At last, complexes Ir1 and Ir2 were obtained by the reaction of cyclometalated iridium(III) dimers with acetylacetone and picolinic acid using anhydrous K₂CO₃ in a mixture of dichloromethane and methanol at room temperature, respectively. The targeted complexes Ir1 and Ir2 were separated as red powders, their chemical structures were verified completely by nuclear magnetic resonance (NMR) and mass spectra. Ir1 and Ir2 exhibited good solubility in common organic solvents owing to the naphthenic hydrocarbon unit in the main ligand. Because of the symmetric characteristic of the auxiliary ligand acetylacetone, the two main ligands in Ir1 showed the identical resonance signals, on the contrary, another auxiliary ligand picolinic acid is asymmetric, as a result, the two main ligands in Ir2 exhibited two distinct resonance signals as illustrated in ¹H NMR spectra (Supplementary Materials) [21]. The other resonance signals are consistent with the structures of both complexes. Moreover, the photophysical properties of both complexes have been studied via UV/vis absorption spectrometry, and steadystate and transient phosphorescence spectrometry.

Figure 1. Synthetic route and chemical structures of complexes Ir1 and Ir2.

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2.2. Photophysical Properties

Next, the absorption and emission spectra of Ir1 and Ir2 in CH2Cl2 were studied at room temperature as shown in Figure 2, and the photophysical data have been summarized in Table 1. The absorption spectra of both complexes showed similar profile, and the intense absorption peak below 300 nm can be ascribed to π - π * transition of the main ligand, the relatively weak peak between 322 to 400 nm may be ascribed to a mixture of the ¹MLCT (singlet metal-to-ligand charge transfer) and ³MLCT (triplet metal-to-ligand charge transfer). Notably, there existed a weak absorption peak at the range of 472 to 565 nm, which may be assigned to 3MLCT [22–25]. Both Ir1 and Ir2 exhibited bright red emission in CH₂Cl₂ in naked eye when excited by 365 nm as depicted in the insert in Figure 2b. The maximum phosphorescent peaks of Ir1 and Ir2 were at 625 and 620 nm with the emission full width at half maximum of 66 and 59 nm, respectively. The lifetimes of Ir1 and Ir2 in the degassed CH₂Cl₂ are 140 and 130 ns, respectively, which showed typical exponential decay, and the relatively short lifetime is beneficial to the device performances. Impressively, the two complexes exhibited high phosphorescent quantum efficiency of 0.32 and 0.35, respectively. Then, the phosphorescent behaviors of both complexes in degassed CH₂Cl₂ at various concentrations in the range of 1.0×10⁻³ to 1.0×10-6 M were studied as shown in Figure 3a,b. The emission spectra showed a maximum phosphorescent peak at 625 nm for Ir1 and 620 nm for Ir2, respectively. The similar profile indicated that there existed almost no or very weak intermolecular interactions between the adjacent molecules in the solution. The low-temperature (77 K) phosphorescent spectra were then investigated as shown in Figure 2d, Ir1 and Ir2 showed obvious blue-shift compared with the spectra at room temperature, peaking at 607 and 596 nm with a shoulder peak at 653 and 649 nm, and the rigidochromic shift for Ir1 and Ir2 are 18 and 24 nm, respectively. Furthermore, according to the highest-energy vibronic subband of emission spectra at 77 K, the triplet energy (T1) can be calculated to be 2.05 eV for Ir1 and 2.08 eV for Ir2 [21]. The phosphorescent properties of Ir1 and Ir2 at solid-states were then investigated. Both of them showed bright red emission in naked eye when excited by 365 nm UV light (Figure S2), and the maximum phosphorescent peaks were at 700 and 674 nm, respectively.

Besides, the phosphorescent behaviors of Ir1 and Ir2 in various degassed solvents (1,4-dioxane, 2-ethoxyethanol, DMF, DMSO, ethanol, methanol, n-hexane, and THF) at the same concentration were carried out and depicted in (Figure 3c,d). The maximum phosphorescent peak of both Ir1 and Ir2 changed significantly together with the solvents possessing different polarity. Taking Ir1 as an example, the maximum emission peak of Ir1 in 1,4-dioxane and methanol is 602 and 656 nm, respectively, which exhibited a large shift about 54 nm. The distinct shift indicated that the phosphorescent emission maybe resulted from a mixture of LC excited-state and 3MLCT [22–25].

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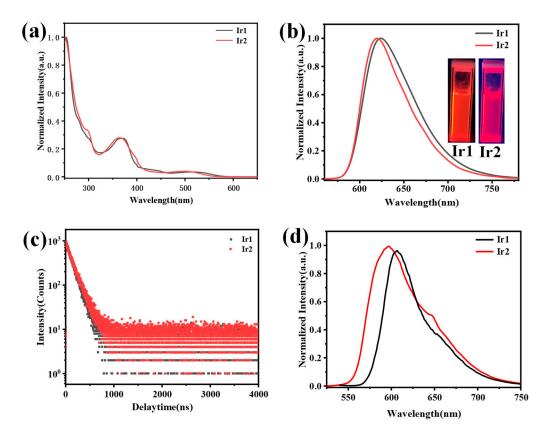


Figure 2. (a, b) The absorption spectra (left) and emission spectra (right) of Ir1 (black line) and Ir2 (red line) in dichloromethane, insert: the photographs of Ir1 and Ir2 in CH₂Cl₂ solution. (c) The decay curves of phosphorescent lifetime for Ir1 and Ir2 in solid states, (d) The low temperature emission spectra of Ir1 and Ir2 at 77 K.

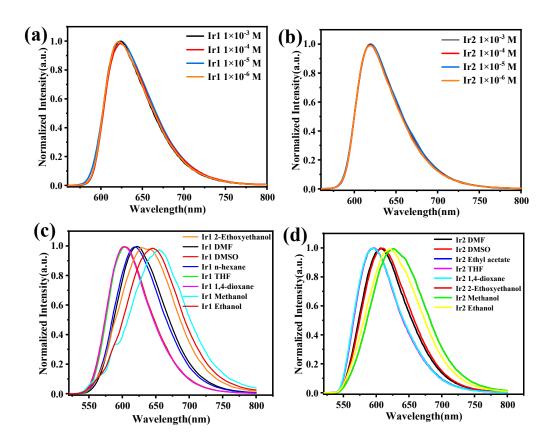


Figure 3. (a, b) Emission spectra of Ir1 and Ir2 at different concentrations in dichloromethane at room temperature, (c, d) Emission spectra of Ir1 and Ir2 in different solvents at the concentration of 1.0×10^{-5} M

2.3. Cyclic Voltammograms and TGA Analysis

Furthermore, the cyclic voltammetry was then carried out in acetonitrile to assess the electrochemical properties of Ir1 and Ir2. As showed in Figure 4a, both of them exhibited reversible oxidation wave, and the oxidation potential is determined to be 0.92 and 1.05 V. The Ir centered oxidation may be responsible for the positive oxidation potential. On the basis of cyclic voltammetry, the energy level of the HOMO and LUMO of Ir1 and Ir2 can be calculated to be -5.73/-3.74 and -5.86/-3.86 eV, respectively [25].

As we know, thermostability of EML materials in OLEDs based on vacuum evaporation is a very important factor. The thermogravimetric analysis was carried out under argon. The results showed that the temperatures of weight loss about 5% for Ir1 and Ir2 are at 401 and 301°C, respectively, suggesting that both compounds possessed excellent thermostability under argon.

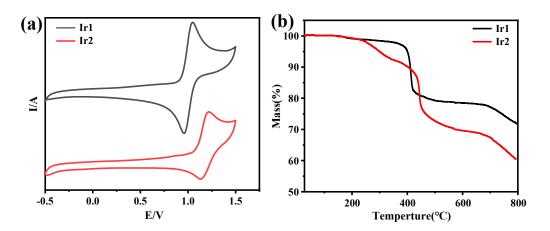


Figure 4. (a) The cyclic voltammograms of Ir1 and Ir2 under a scan rate of 100 mV/s in CH₃CN, (b) TGA curves of Ir1 and Ir2.

2.4. Electroluminescent Devices

To investigate the electroluminescence (EL) performances, OLEDs based on Ir1 and Ir2 were carefully fabricated adopting the structures of ITO / MoO₃ (3 nm) / TCTA (4,4′,4″-tris(carbazol-9-yl)triphenylamine, 40 nm) / Bepp₂ (bis[2-(2-pyridinyl)phenolato]berylliuM):4 wt% Ir (20 nm) / TPBi (1,3,5-tris(1-phenyl-1H-benzimidazol-2-yl)benzene, 50 nm) / LiF (1 nm) / Al (100 nm) as shown in Figure S2. Among them, ITO, MoO₃, TCTA, and TPBi acted as anode, hole injection layer, hole transport layer, and electron transfer layer, respectively. Bepp₂, a high-efficiency fluorescent material, acted as the host of phosphors Ir1 and Ir2, and was doped with the EML. The energy level of triplet-excited states for Bepp₂ is 2.6 eV, higher than Ir1 and Ir2, which can realize effective Forster energy transfer between the host material and the iridium (III) complexes. To overall assess the device performances, the doping concentration of 4% of weight was adopted. The energy levels of the red OLEDs were depicted in Figure S2. On the basis of the energy levels of devices, the energy level HOMO/LUMO of Ir1 and Ir2 were both within those of the Bepp₂ host material. Therefore, the excellent carrier capturing in the host-guest mixture system may be responsible for the dominant EL mechanism [26]. The performances of the red devices based on the two complexes were shown in Table 1 and summarized in Table 2.

Table 1. Photophysical data of Ir1 and Ir2.

Complexes	Emission in CH2Cl2			Eg[eV]a	Eonsetox[eV]	T ₁ [eV] ^b
	λ _{em} [nm]	τ[μs]	$\Phi_{ ext{PL}}$	•		

Ir1	625	0.14	0.32	2.23	0.92	2.05
Ir2	620	0.13	0.35	2.23	1.05	2.08

a. E_g was estimated from absorption onset from UV-visible spectra. b. T_1 =1240/ λ_{77K} .

The turn-on voltages at 1 cd/m² of the device based on Ir1 and Ir2 were at 3.5 and 3.7 V, respectively, and these low values indicated poor injection barrier between electron transfer layers to the emissive layers. Notably, the normalized EL spectra of Ir1 and Ir2 exhibited obvious blue-shift compared with those of in CH2Cl2, peaking at 612 and 591 nm, respectively, which may be attributed to sensitivity to the surroundings of the triplet-excited states of these two complexes, and the host material Bepp2 may be an important influence on the triplet-excited states, which is consist with the obvious solvatochromism behaviors in different solvents. The 1931 Commission Internationale de L'Eclairage (CIE) coordinates of two devices are (0.61,0.37) and (0.51,0.43), respectively, which are corresponding to the red range. The L-V-J curves of the red devices were depicted in Figure 5b. The key parameters of devices were listed in Table 2. The maximum luminance of Ir1 and Ir2 are 25970 and 13450 cd/m², respectively. The maximum CE, PE, and EQE of the red devices based on Ir1 and Ir2 are 13.47/15.22 cd/A, 10.35/12.26 lm/W, and 10.08/7.48%, respectively.

Table 2. EL data for the red OLEDs based on Ir1 and Ir2.

Complex	X	$\lambda_{\mathrm{EL}}/$	CIE(x,y)	Von/	L _{max} /	CE _{max} /	PEmax/	EQE/
	%	nm		\mathbf{V}	cd∙m ⁻²	$cd \cdot A^{-1}$	$lm \cdot W^{-1}$	%
Ir1	4	612	(0.61,0.37)	3.5	25970	13.47	10.35	10.08
Ir2	4	591	(0.51,0.43)	3.7	13450	15.22	12.26	7.48

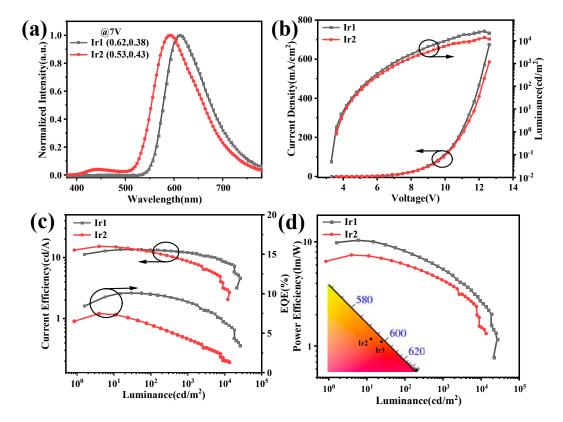


Figure 5. (a) The normalized EL spectra, (b) The J-V-L curves, (c) CE-L-EQE curves, and (d) PE-L curves of the red OLEDs based on Ir1 and Ir2 with the doping concentration of 4% in Bepp₂. 3. Conclusions.

3. Conclusions

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In summary, two novel neutral phosphorescent iridium(III) complexes (Ir1 and Ir2) have been successfully designed and synthesized with high yields and good solubility. Both complexes exhibited enhanced red emission in the solid and solution. The devices based on Ir1 and Ir2 were rationally fabricated by using Bepp2 as the host material with the doping concentration of 4%. The performances of these devices showed some differences, and the peak CE, PE, and EQE of the red devices based on Ir1 and Ir2 are 13.47/15.22 cd/A, 10.35/12.26 lm/W, and 10.08/7.48%, respectively. Owing to sensitivity to the surroundings of the triplet-excited states of these two complexes, EL spectra exhibited a degree of blue-shift compared with those of in CH₂Cl₂. Next, we will select more proper host materials and device structures to improve the red EL quality of the OLEDs. At last, we believe that our work will be of great significance for the development of novel red-emissive iridium complexes in future.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org, Figure S1: The phosphorescent spectra of Ir1 and Ir2 in solid states. Figure S2: Schematic diagram of device structure (a) of the red OLEDs and chemical structures (b) of the materials involved in the prepared devices.

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