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Non-interlayer and color stable WOLEDs with mixed host and incorporating a new orange phosphorescent iridium complex

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ABSTRACT

In this work, we have synthesized a new phosphorescent iridium complex (Bppya)₂Ir(acac), as an orange dopant for organic light-emitting diodes (OLEDs). The device achieved an external guantum efficiency (EOE) of 22.4% a current efficiency of 49.5 cd A^{-1} and a power efficiency of 38.9 lm W⁻¹, which are among the highest values for orange OLEDs. Furthermore, color stable and high efficiency phosphorescent white OLED (WOLED) was demonstrated by utilizing a mixed-host in the emissive layers (EMLs) composed of a blue phosphor and the new orange phosphorescent emitter, as well as by avoiding the use of interlayers that commonly exist between different EMLs in WOLEDs. Our WOLED presented decent white emission with maximum efficiencies of 25.3 cd A⁻¹ and 12.6%. Furthermore, the 1931 Commission Internationale de l'Eclairage color coordinates exhibited extremely small variation of $(0.3940 \pm 0.0102, 0.4323 \pm 0.0046)$ in a wide luminance range from 49 to 38,035 cd m⁻² when driving voltages increased from 4 V to 12 V. The root cause for this excellent color stability is the utilization of the mixed-host to obtain bipolar transport properties in EMLs as well as the eliminating of interlayer between different EMLs, which, on one hand, effectively broaden the exciton recombination zone; on the other hand, reduce the accumulation of triplet excitons at the emissive interface.

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1. Introduction

White organic light-emitting devices (WOLEDs) have drawn intensive attention due to their potential application as a solid-state flat light source and a back light unit for liquid crystal displays (LCDs). Except utilizing single

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molecular white-emitter, WOLEDs are generally obtained by incorporating either three primary color emitters (red, green, and blue) or two complementary emitters (e.g., sky blue and yellow) in a single- or multi-layer(s) structure. Although it is theoretically simple for three-color based WOLEDs to achieve the standard white light with the Commission Internationale de l'Eclairage (CIE) coordinates of (0.33, 0.33) because of the simultaneous presence of three primary colors, this method usually results in processing complexity and high operational voltages [1–4]. Accordingly, the strategy of two emitters with complementary colors may solve the problem and has ascended





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to be one important method for the fabrication of WOLEDs [5–7].

To achieve high efficiency WOLED devices, the incorporation of phosphorescent emitter(s) is almost a prerequisite, due to its (their) ability of harvesting the electrically generated 25% singlet and 75% triplet excitons. However, efficiency roll-off needs to be sincerely considered mainly because triplet exciton has long life time, causing severe exciton quenching at high exciton density with the increase of driving voltages. In this aspect, cohost for phosphor was proved to be an effective way not only for reducing roll-off, but also for high device-efficiency, mainly because of the expansion of recombination zone [8–10].

Besides efficiency, color stability is another big challenge in WOLEDs. The main causes leading to color shift in WOLED devices with multiple emitting layers (EMLs) include alteration of recombination zone [11,12], variation of energy transfer situation [13], changing of exciton quenching rate [14,15], as well as electric field or temperature dependent charge mobility [16]. For example, it has been reported that the recombination zone will shift with increasing driving voltage in multi-EML structure when it has a different field-dependent mobility between the hole transporting layer (HTL) and the electron transporting layer (ETL), and/or different carrier trapping situation at the EML [12]. To date, many efforts have been taken to improve the color stability in multi-EML WOLED devices. Among them, the carrier/exciton balance/confinement is a popular approach. It was proved that the sandwiched EMLs with the narrow band-gap emitting layer inserted between wide band-gap ones were beneficial to color stability [17,18]. In addition, EMLs containing mixed hosts with both hole-rich and electron-rich features were demonstrated to broaden recombination zone and facilitate color stabilization [19]. However, depending on the employed functional materials, device structure needs to be carefully designed. Another general strategy to achieve color stable WOLEDs is to insert an interlayer between different emitting layers [20,21]. Unfortunately, the power efficiency generally decreases because of the carrier-hurdle by the additional interlayer(s). Meanwhile, such interlayers complicate fabrication procedure.

In this work, we demonstrated color stable and noninterlayer full phosphorescent WOLEDs by introducing a mixed-host in the EML consisting of a blue phosphor FIrpic{iridium(III)bis[4,6-(difluorophenyl)pyridinato-N,C2'] picolinate} and a new orange phosphorescent emitter. Specifically, the new phosphor is a heteroleptic iridium complex with main ligand of C^N = N type [22,23]. By device physics study and optimization, WOLED with maximum efficiencies of 25.3 cd A⁻¹ and 12.6% was achieved. Furthermore, the emission color was very good and stable in a wide luminance range. When voltages increased from 4 V to 12 V, the CIE coordinates only varied from (0.4042, 0.4369) to (0.3838, 0.4277), with the brightness changing from 49 to 38,035 cd m⁻². Considering the good CIE color, the device efficiencies are decently high. We demonstrated that the employment of the mixed-host with bipolar transport properties as well as the eliminating of interlayer between different EMLs was accounted for this good device performance, which, on one hand, effectively broaden the exciton recombination zone; on the other hand, reduce the accumulation of triplet excitons at the emissive interface.

2. Experimental

2.1. Material characterization

The ¹H NMR spectra were recorded on a Bruker Ultra Shield Plus 400 MHz spectrometer. UV-vis absorption spectra were recorded using a Shimadzu UV-3600 spectrophotometer. Photoluminescence (PL) spectra at room temperature were measured on a Shimadzu RF-5301PC fluorophotometer. The PL quantum efficiency and lifetime were performed with an Edinburgh Instruments FLS920 spectrometer. Electrochemical analysis was performed on a Bioanalytical Systems CHI660E operating in cyclic voltammetry (CV) mode. Glassy carbon, platinum wire, and $Ag/AgNO_3$ (0.01 mol L⁻¹) were employed as working, auxiliary, reference electrode, respectively. Tetrabutylammonium perchlorate (0.1 M) dissolved in dichloromethane (DCM) for oxidation solutions was used as electrolyte. The scan rate was 100 mV s⁻¹. High resolution mass spectrometry (HRMS) data were measured on an Ion Spec 4.7 Tesla FTMS instrument, operating at MALDI/DHB mode.

2.2. OLED device fabrication and characterization

The OLED devices were fabricated on indium tin oxide (ITO) coated glass substrates with a sheet resistance of 25Ω square⁻¹. Before device fabrication, ITO glass substrates were precleaned carefully with de-ionized water, acetone, and ethanol, and then treated with O₂ plasma for 30 s. All layers were grown in succession by thermal evaporation without breaking vacuum ($<3.0 \times 10^{-3}$ Pa). The current efficiency-luminance-power efficiency characteristics and electroluminescence (EL) characteristics of the devices were measured with a computer-controlled Keithley 2400 source meter and fiber spectroscopy calibrated by a PR655 spectrophotometer at room temperature. The device emission area (0.1 cm^2) was defined by the overlapping of ITO anode and Al cathode. All measurements were carried out under ambient conditions without device encapsulation.

2.3. Material synthesis

The new iridium complex, bis[3,6-bis(phenyl)-pyridazinato]iridium(acetylacetonate) [(Bppya)₂Ir(acac)], was synthesized following the procedures depicted in Scheme 1.

2.3.1. Synthesis of Ligand HBppya

Trans-1,2-Dibenzoylethylene (0.75 g, 3.18 mmol) was dissolved in acetic acid (20 mL) by heating. After cooling to room temperature, an excess amount of hydrazine monohydrate (13 mL) was added dropwise. The reaction mixture was kept stirring at room temperature for another hour, and then refluxed overnight. After cooling down, the solution was poured into ice to give a white precipitate. Subsequent recrystallization from chloroform yielded the pure product (0.28 g, 38%).



Scheme 1. Synthesis of iridium(III) complex (Bppya)₂Ir(acac).

2.3.2. Synthesis of (Bppya)₂Iridium(acac)

IrCl₃·3H₂O (1 mmol) and the ligand HBppya (2.5 mmol) were added to the mixture of 2-ethoxyethanol and water (v/v = 3:1, 16 mL). After refluxing under nitrogen for 24 h. the reaction mixture was cooled to room temperature. The orange precipitate was collected by filtration and washed with water, ethanol and hexane successively. The solid was then pumped dry completely to give the crude chloro-bridged dimer complex. Without further purification, the dimer was added to a mixture of Na_2CO_3 (5 mmol), acetyl acetone (1.5 mmol), and 2-ethoxyethanol (10 mL). After refluxing under nitrogen overnight, the solution was cooled to room temperature. The orange precipitate was filtered off and washed with water, ethanol and hexane. The crude product was purified by chromatography on silica gel using petroleum ether/dichloromethane as mobile phase. (Bppya)₂Iridium(acac): Yield 52%. ¹H NMR $(400 \text{ MHz}, \text{CDCl}_3) \delta$ 7.98 (d, J = 7.8 Hz, 4H), 7.50 (d, J = 14.0, 6.5 Hz, 3H), 7.38 (d, *J* = 8.0, 5.4 Hz, 7H), 7.05 (d, *J* = 9.1 Hz, 2H), 6.76 (t, J = 7.5 Hz, 2H), 6.56 (t, J = 7.6 Hz, 2H), 5.99 (d, J = 7.8 Hz, 2H), 2.05 (s, 6H). HRMS (FAB, m/z): calcd for C₃₇H₂₉IrN₄O₂ 752.1898, found M⁺ 752.1891.

3. Results and discussion

As shown in Fig. 1a, (Bppya)₂Ir(acac) reveals intense UV absorption bands between 225 and 320 nm (extinction coefficient ε of 40,000–55,000 M⁻¹ cm⁻¹), which are contributed from the spin-allowed $(\pi - \pi^*)$ transitions of the coordinated 3,6-bis(phenyl)-pyridazinato group. The less intense bands between 350 and 440 nm (ε = 5000-12,000 M^{-1} cm⁻¹) can be assigned to an inter ligand charge transfer (¹LLCT) and a metal-to-ligand charge transfer $(S_0 \rightarrow {}^1MLCT)$ [5,13,14]. The iridium complex also shows weak absorption bands above 450 nm ($\varepsilon < 5000 \text{ M}^{-1}$ cm⁻¹). These absorptions are likely attributed to $S_0 \rightarrow {}^3MLCT$ and ${}^3(\pi - \pi^*)$ transitions due to the effect of strong spin-orbit coupling in iridium complex. The (Bppya)₂Ir(acac) shows intense orange photoluminescence with the emission peak of 582 nm. Its photoluminescence quantum yield (PLQY) is 0.31, measured in a degassed CH₂Cl₂ solution at room temperature. In addition, the



Fig. 1. (a) Absorption and emission spectra of $(Bppya)_2 lr(acac)$ in $CH_2 Cl_2$ and (b) cyclic voltammogram of $(Bppya)_2 lr(acac)$ recorded in $CH_2 Cl_2$, scan rate: 100 mV s⁻¹.

photoluminescence of $(Bppya)_2 Ir(acac)$ exhibits almost single-exponential decay with emission lifetime of 0.57 µs, indicating the triplet nature of the emission.

Cyclic voltammetry was used to investigate the electrochemical behavior of the iridium(III) complex and determine its energy levels, as shown in Fig. 1b. The HOMO energy level was calculated from the oxidation potential (E_{ox}), while the LUMO energy level was derived by combining the HOMO level with the optical bandgap obtained from the absorption edge [24,25]. To determine the oxidation potentials, the reference electrode was calibrated using a ferrocene/ferrocenium salt couple, which is assumed to have a redox potential with an absolute energy level of -4.80 eV in vacuum. The HOMO energy value was calculated using the following equation: $E_{HOMO} =$ -(4.8 + $E_{ox} - E_{Fc}$) eV, where E_{ox} and E_{Fc} are the oxidation potentials (vs. Ag/Ag⁺) of the complex and ferrocene, respectively [26]. The HOMO level of (Bppya)₂Ir(acac) is -5.41 eV and its LUMO level is -2.80 eV.

Based on this new material, we studied both the monochromic and white OLED devices. The related material structures and energy levels are depicted in Fig. 2 [27-31]. Firstly, monochromic device with a traditional device configuration of ITO/NPB (4,4'-bis[N-(1-naphthyl)-Nphenylamino]-biphenyl) (40 nm)/(Bppya)₂Ir(acac): CBP (6 wt%, 30 nm)/BCP (2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline) (8 nm)/Alg₃ (tris(8-hyroxyguinolinato)aluminum(III)) (30 nm)/LiF (1 nm)/Al (100 nm) was fabricated. As shown in Fig. 3, this device exhibited strong orange (peaked at 580 nm) electroluminescence (EL) with an external quantum efficiency (EQE) of 22.4%, a current efficiency of 49.5 cd A⁻¹ and a power efficiency of 38.9 lm W⁻¹ under driving voltage of 4 V, which are among the highest values for orange OLEDs. Additionally, the broad EL band from 520 nm to 748 nm is highly desired for WOLEDs. Furthermore, device efficiencies were very stable, at a high luminance of 1000 cd/m^2 , these values still remained as high as 45.7 cd A^{-1} , 22.3 lm W^{-1} , and 17.5%, further showing its potential for constructing WOLED.



Fig. 3. Current efficiency–luminance–power efficiency characteristics for the monochromic device. The inset is normalized EL spectra at different voltages.

With regards to WOLED devices employing $(Bppya)_2$ Ir(acac), a traditional blue phosphor FIrpic was selected and a series of devices both with $(W_1 \text{ and } W_2)$ and without (W_3) interlayers between the orange and blue EMLs were fabricated. For W_1 series, the device structure was ITO/ NPB(40 nm)/TCTA(5 nm)/(Bppya)_2Ir(acac):CBP (6 wt%, 10 nm)/mCP (2 nm in device W_{1-1} or 5 nm in device W_{1-2})/ FIrpic:mCP (6 wt%, 10 nm)/TPBi (30 nm)/LiF (1 nm)/AI. Here, mCP (3,5'-N,N'-dicarbazole-benzene) serves as host for FIrpic and the interlayer material, TPBi acts as the electron-transporting layer. TCTA [4,4',4"-tri(N-carbazoly1)triphenylamine] was inserted between NPB and CBP for the



Fig. 2. The energy levels and structures of related material used in the monochromic and white OLEDs.

purpose of flattening the HOMO injection gap between NPB (HOMO = 5.4 eV) and CBP (HOMO = 6.0 eV). The HOMO of TCTA (5.7 eV) is located in between NPB and CBP, which can provide a buffer gradient for hole injection and reduce the energy barrier effectively. On the other hand, the LUMO of TCTA (2.3 eV) is the same as NPB and higher than CBP (2.9 eV), which can help to block electron drifting out of CBP to NPB layer, making the recombination of excitons in EML maximally. The two emitters are arranged with sequence of orange to blue from anode to cathode.

It can be seen from Fig. 4a that with the interlayer of mCP = 2 nm, device W_{1-1} exhibited white color with maximum efficiency of 5.0 cd A^{-1} and good CIExy of (0.3550, 0.3813). This low efficiency is inconsistent with the monochromic device employing (BPPya)₂Iracac, which shows high efficiency of 49.5 cd A^{-1} (see above). Therefore, we suspect that in W_{1-1} , the emission of orange color should be mainly produced by energy transfer from FIrpic EML due to the thinner interlayer of 2 nm, but not direct recombination in that layer. To verify this, we increased the interlayer to 5 nm (device W_{1-2} , Fig. 4b). As a result, due to the block of energy transfer by thicker interlayer, the FIrpic emission predominated in device W_{1-2} , confirming that

in device W_1 series the recombination zone locates in the blue-emitter region near the cathode. Therefore, due to the intrinsic low electroluminescent efficiency of FIrpic based device [19], as well as the quenching of this blue EL by the orange EML, the efficiencies of W_1 series are low.

For the purpose of enlarging orange emission by direct recombination in the orange-emitting zone, electron transporting toward orange-emitting layer must be enhanced. Therefore, device W₂ series were made with structure of ITO/NPB (40 nm)/TCTA (5 nm)/(Bppya)₂ Ir(acac):CBP (6 wt%, 10 nm)/CBP:TPBi (5 nm)/FIrpic:TPBi (6 wt%, 10 nm)/TPBi(30 nm)/LiF (1 nm)/Al. Here beside using electron transport TPBi instead of hole transport dominated mCP to host FIrpic in order to improve electron transport toward anode, the electron transport property of the interlayer was also enhanced by using mixture of CBP:TPBi. The ratios of CBP:TPBi are 1:1 (W₂₋₁), 2:1 (W_{2-2}) and 4:1 (W_{2-3}) . As expected (Fig. 5a), due to the direct recombination in both orange- and blue-EMLs, blue and orange emissions were realized with greatly improved efficiencies. For example, the maximum efficiency of





Fig. 4. (a and b) are current efficiency–luminance–power efficiency characteristics for devices W_{1-1} and W_{1-2} , respectively. The insets on the left are EMLs of devices W_{1-1} and W_{1-2} , Orange stands for (Bppy)₂lr(acac) based EML, Blue stands for Flrpic based EML. The insets on the right are normalized EL spectra at 4 V. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Fig. 5. (a) Current efficiency-luminance characteristics for device W_2 series, the insets are EL spectra at 4 V and the EML structure and (b) normalized EL intensity-wavelength characteristics at different voltages for device W_{2-3} .

 W_{2-3} (20.5 cd A^{-1}) increased almost four folds compared to device W_1 series (5.0 cd A^{-1}). Nevertheless, due to the tradeoff between exciton formation in blue and the orange EMLs, the EL spectra were not always white, varying with the ratio of the interlayer components (inset of Fig. 5a), e.g., the blue emission increased with the increase of CBP in the interlayer of CBP:TPBi. Among these devices, W_{2-3} (with a ratio of 4:1 in CBP:TPBi interlyer) achieved a warm white emission with a maximum current efficiency of 20.5 cd A^{-1} and CIE coordinate of (0.3814, 0.4207) at 4 V. However, as shown in Fig. 5b, the white color only presented at low driving voltage. With the increase of voltage, orange emission gradually dominated.

Seeking device mechanisms in device W₂ series, energy levels and the transport properties of materials in the EMLs and the interlayer were examined. Since CBP and TPBi are mainly hole and electron transporting materials, respectively, at the double interfaces (Orange-CBP/CBP:TPBi/ Blue-TPBi) in the EML of W₂ series, the holes will transport across Orange-CBP and accumulate near the interface of CBP:TPBi/Blue-TPBi; accordingly, the electrons will mainly fill the LUMO of TPBi, reaching the interface of Orange-CBP/CBP:TPBi. This will lead to the interface dominated orange and blue exciton generation hence narrow recombination zone (see inset of Fig. 5a). Additionally, considering the relative position of LUMOs and HOMOs in CBP and TPBi, there was no barrier for electron to inject from blue doped TPBi into orange doped CBP, while there was about 0.3 eV energy barrier for hole injection from orange doped CBP to blue doped TPBi. Therefore with the increase of voltage, the unbalanced electron/hole injection into the blue and orange EMLs should be account for the unstable white spectra in W_{2-3} .

In order to overcome this problem, we fabricated devices (W₃ series) employing CBP and TPBi as mixed-host for both (Bppya)₂Ir(acac) and FIrpic to allow for bipolar charge transporting in the whole EML, thus broadening the recombination zone. In the meantime, the interlayer between different EMLs was eliminated and the balanced white emission is expected achievable by varying the thickness of the blue and orange EMLs. The device structure was ITO/NPB(40 nm)/TCTA(5 nm)/FIrpic:CBP:TPBi/(Bppya)₂Ir(acac): CBP:TPBi/FIrpic:CBP:TPBi/TPBi(30 nm)/LiF/Al. Here, the ratio of CBP:TPBi is 4:1, and the concentration of the blue and the orange phosphors is always 6%. We adopted the EML pattern of blue/orange/blue, considering that, on one hand, the efficiency of FIrpic based device is generally lower than that of (Bppya)₂Ir(acac) one; on the other hand, as demonstrated in W₁ series that the excited state energy of the blue emitter can partially transfer to that of orange emitter without or with thin interlayer. Two blue EMLs can enhance blue emission, which will be good for balanced white emission. The thickness of the EML (blue/orange/ blue) was chosen as 10 nm/10 nm/10 nm for W_{3-1} and 12 nm/6 nm/12 nm for W₃₋₂ (see Fig. 6a). As a result, devices W₃₋₁ and W₃₋₂ exhibited good white emission with maximum efficiencies of $25.3 \text{ cd } A^{-1}$ (12.6%) and 20.4 cd A^{-1} (10.4%), and color rendering indexes (CRI) of 63 and 61, respectively (Fig. 6b and c). At brightness of 1000 cd m⁻², their efficiencies still remained as 19.4 cd A⁻¹ and 15.9 cd A^{-1} , indicative of small efficiency roll-off.



Fig. 6. (a) EML structures of W_{3-1} and W_{3-2} , (b) the current efficiency– luminance–power efficiency curves for device W_{3-1} and W_{3-2} and (c) the normalized EL intensity–wavelength characteristics for device W_{3-1} and W_{3-2} , the insets are CIE coordinates under different voltages.

Particularly, the white colors were very stable as shown in Fig. 6c. For example, when voltages increased from 4 V to 12 V, the CIE coordinates showed small alteration of $(0.3940 \pm 0.0102, 0.4323 \pm 0.0046)$ for W₃₋₁, and $(0.3353 \pm 0.0032, 0.4252 \pm 0.0048)$ for W₃₋₂ in a wide luminance range of 49–38,035 cd m⁻² and 47–30,253 cd m⁻², respectively. Evidently the utilization of CBP and TPBi as mixed-hosts greatly improved device performance in comparison with only CBP- or TPBi-hosted devices. It can be attributed to bipolar transport of the mixed-hosts, which could effectively broaden the exciton recombination zone as well as reduce the accumulation of triplet excitons at the emissive interface. Additionally, this is also account for the low efficiency roll-off.

4. Conclusions

In conclusion, we have developed a new iridium complex (Bppya)₂Ir(acac), which is capable of producing highly efficient orange and white OLEDs. For the orange OLEDs, a high external quantum efficiency of 22.4% and a current efficiency of 49.5 cd A^{-1} can be achieved. More importantly, a series of color stable and non-interlayer full phosphorescent WOLEDs were fabricated by introducing a mixed-host in the emissive layer (EML) composed of a blue phosphor FIrpic and a new orange phosphorescent emitter. Result shows that the maximum device efficiencies are 25.3 cd A⁻¹/20.4 cd A⁻¹ and 12.6%/10.4%, respectively. When voltages increase from 4 V to 12 V, the CIE coordinates exhibit small alteration of (0.3940 ± 0.0102) , 0.4323 ± 0.0046)/(0.3353 ± 0.0032, 0.4252 ± 0.0048) in a wide luminance range of 49–38,035 cd $m^{-2}/47$ –30,253 cd m^{-2} . The root cause for excellent color stability is the employment of the mixed-host with bipolar transport properties as well as the eliminating of interlayer between different emission zone, which, on one hand, effectively broaden the exciton recombination zone; on the other hand, reduce the accumulation of triplet excitons at the emissive interface. The mixed-host strategy is a promising way for development of high color stable OLED light sources for use in LCD backlights and indoor planar lighting applications.

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