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Highly efficient blue and white phosphorescent OLEDs based on an iridium complex

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1. Introduction

White organic light-emitting diodes (WOLEDs) have drawn much attention for their applications in flat-panel displays and solid-state lightings [1]. WOLEDs can be classified into three types according to the emitters used: fluorescent devices (F-WOLEDs), phosphorescent devices (P-WOLEDs) and hybrid devices employing both fluorescent and phosphorescent emitters (F/P-WOLED) [2]. WOLEDs based on phosphorescent emitters have been shown to achieve an internal quantum efficiency of 100% as compare to the theoretical value of 25% in fluorescent OLEDs [3]. Recent works have reported impressive power efficiency up to 124 lm/W by utilizing three primary colors (RGB) phosphorescent materials and light out-coupling enhancement [4]. In fact, much recent progress on new materials, novel device structures, and light extraction methods have been developed to meet the requirements of WOLEDs [5]. Meanwhile, one bottleneck for further enhancement

ABSTRACT

A new blue-emitting iridium complex, Iridium(III) bis [(2,3,4-trifluorophenyl) -pyridinato-N, C^{2'}] picolinate (Ir(tfpd)₂pic) has been designed, synthesized and used to fabricate blue phosphorescent organic light-emitting devices with a efficiency up to 41.4 lm/W (52.6 cd/A). Furthermore, by combining the blue phosphorescence of Ir(tfpd)₂pic and the yellow emission of iridium(III) bis [2-(2-naphthyl)-pyridine] (acetylacetonate) (Ir(npy)₂acac), highly efficient white emission with a maximum efficiency of 49.0 lm/W (54.5 cd/A) has been achieved. The results indicate that Ir(tfpd)₂pic is a promising blue phosphor for the applications in highly efficient blue and white OLEDs.

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of the efficiency of WOLEDs is the low performance of the blue phosphorescent emitters. For instance, maximum power efficiencies for red and green monochromatic device have been reached 42.7 lm/W (Iridium(III) bis-(2-phenylquinolyl-N, $C^{2'}$) acetylacetonate, PQIr) [6], and 133 lm/W (tris-(phenylpyridine)Iridium, Ir(ppy)₃) [7], respectively. While, FIrpic (Iridium (III) bis [(4, 6-difluorophenyl)-pyridinato-N, $C^{2'}$] picolinate) and FIr6 (Iridium(III) bis-(4', 6'-difluorophenylpyridinato) tetrakis (1-pyrazolyl) borate) are still the best and most frequently used blue phosphorescent emitters. Performance of FIrpic and FIr6 based devices are still below the requirements for applications in solid-state lightings [8]. Thus, development of high performance blue phosphorescent emitters is essential for further improvement of WOLEDs.

Fluorine substituted Ir(III) complexes have been used for designing blue phosphors due to their high quantum yields and color tunability. Conversion of C—H bonds in these complexes to C—F bonds may have several potential benefits, such as reduction of irradiative exciton decay, enhancement of photoluminescence (PL) efficiency and electron mobility etc. [9]. Tokito et al. reported two fluorine substituted Ir(III) complexes, in which penta-fluorophenyl group is bound to either *meta*- or *para*- position of the phenyl ring. Interestingly, the small difference in the two complexes lead to significant difference in PL efficiency [9b]. Chen et al. synthesized a iridium complex, which is similar to FIrpic





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except for a *tert*-butyl substituted at the 4-position in pyridine. The material also shows much different EL performance compared with Flrpic [9c]. In our previous report, an Ir complex with a phenyl-pyridine ligand substituted by three fluorines at the *meta-*, *para*-positions gives a light blue emission peaked at 479 nm. An optimized device using the complex as dopant exhibits a maximum current efficiency of 37.6 cd/A [9d]. It can be seen that differences in the number of fluorine and their substitution positions can have considerable effects on the properties and performance in these Ir(III) complexes [10].

In this work, we designed and synthesized a new blue phosphor, Iridium(III) bis[(2,3,4-trifluorophenyl)-pyridinato-N,C^{2'}] picolinate (Ir(tfpd)₂pic). Highly efficient blue phosphorescent OLEDs were fabricated by using this new phosphor. By doping Ir(tfpd)₂pic into 9, 9-spirobifluoren-2-yl-diphenyl-phosphine oxide (SPPO1), blue emission with a maximum efficiency of 41.4 lm/W (52.6 cd/A) has been realized. Performances of the blue phosphorescent OLEDs are among the best-reported values. Furthermore, WOLEDs have been fabricated by using a simple dual-emissive-layer device structure, combining the blue emitting dopant Ir(tfpd)₂pic with a yellow phosphor, Iridium(III) bis [2-(2-naphthyl)-pyridine] (acetylacetonate) (Ir(npy)₂acac). Highly efficient white emission with a maximum efficiency of 49.0 lm/W (54.5 cd/A) has been achieved. These results suggest that Ir(tfpd)₂pic should be a promising blue phosphor for the applications in highly efficient blue and white OLEDs.

1.1. Experimental details

The cyclometalating ligand 1-(2,3,4-trifluorophenyl)-pyridine was prepared via a Suzuki coupling reaction between 1bromopyridine and 2,3,4-trifluorophenyl boronic acid. Synthesis of the final Ir complex involved two key steps. In the first step, IrCl3 was reacted with excess of the synthesized ligands to produce a chloro-bridged Ir dimer. This dimer can be readily converted to the monomeric complex Ir(tfpd)2pic by replacing the bridging chlorides with picolinic acid in the presence of Na2CO3. The synthetic pathway toward the preparation of Ir(tfpd)2pic is outlined in Scheme 1. The molecular structure of Ir(tfpd)2pic has been confirmed with 1H nuclear magnetic resonance: (300 MHz, CDCl3) d: 5.53–5.58 (t, 1H), 5.80–5.85 (t, 1H),7.00–7.04 (t, 1H), 7.21–7.26 (t, 1H), 7.41–7.47 (m, 2H),7.77–7.85 (m, 3H), 7.95–8.00 (t, 1H), 8.23–8.35 (m, 3H). 8.73–8.75 (d, 1H); mass spectrometry (ESI⁺):m/z 732.1(MH⁺).Calcd for C₂₈H₁₄O₂N₃F₆Ir : 730.64. and elemental analysis: Anal. Calcd. For C₂₈H₁₄O₂N₃F₆Ir C: 46.03%, H: 1.93%, N: 5.75%. Found: C: 45.89%, H: 1.95%, N: 5.59%.

Absorption and fluorescence spectra were recorded respectively with a Perkin Elmer Lambda 2S UV–Vis spectrophotometer and a Perkin Elmer LS50B Luminescence spectrophotometer. The highest occupied molecular orbital (HOMO) value of the Ir complex was measured via ultraviolet photoelectron spectroscopy (UPS) with a VG ESCALAB 220i-XL ultrahigh vacuum (UHV) surface analysis system). The lowest unoccupied molecular orbital (LUMO) value was determined from the difference between the HOMO energy and the energy gap determined from the optical absorption edge.

Indium tin oxide (ITO) coated glass substrates with a sheet resistance of 30 Ω /square were used as anode for OLEDs, all layers were deposited by vacuum thermal evaporation. Before successive deposition, the substrates were patterned by traditional lithography, cleaned and dried in an oven at 120 °C. *The ITO substrates were then treated with ultraviolet-ozone before loading into a deposition* chamber. All organic layers and cathode were sequentially deposited onto the ITO substrates under a base pressure of 10⁻⁶ *Torr. Current density-voltage-luminance (J-V-L) characteristics, CIE coordinates, and electroluminescent (EL)* spectra were measured with a programmable Keithley model 237 source and a Photoresearch PR650 photometer.

2. Result and discussions

The molecular structure of $Ir(tfpd)_2pic$ is similar to that of FIrpic except for one more fluorine atom at the 5-position of the phenylpyridine ligand. Fig. 1 shows an absorption and a photoluminescence (PL) spectra of $Ir(tfpd)_2pic$ in a dilute CH_2Cl_2 solution at room temperature. The 254 nm absorption peak is attributed to the π - π^* transition of the trifluorophenylpyridine ligand and the weak absorption peaks located at 365 and 430 nm are likely to be originated from the ¹MLCT (metal to ligand charge transfer) and ³MLCT transitions, respectively [11]. The dilute $Ir(tfpd)_2pic$ solution



Scheme 1. Synthetic routes of Ir(tfpd)₂pic.

exhibits strong blue emission at 483 nm. The shape of the PL spectrum is similar to that of FIrpic except for a mild red-shift of 12 nm. [12]. The fluorescence quantum yield of Ir(tfpd)₂pic in CH₂Cl₂ solution was measured to be 0.69 using FIrpic (Φ_f : 0.50) as a calibration standard. The higher PL quantum yield of Ir(tfpd)₂pic could be attributed to the additional fluorine atom attached to the ligands than the case for FIrpic. replacing the C-H bond with a C-F bond with lower vibrational frequency can reduce the rate of nonradiative decay and thus enhance the PL efficiency [9a]. The HOMO level of Ir(tfpd)2pic determined with UPS is 6.2 eV. The LUMO level was determined from the HOMO level and the optical absorption edge is 3.5 eV. It should be pointed out that the HOMO level of the iridium complex corresponds to the 5d-orbital if the Ir atom has substantial mixing with the π -orbitals of the trifluorophenylpyridine ligands, while the LUMO level is determined by the π^* -orbitals of the trifluorophenylpyridine ligand and independent of the picolinate ligand [13]. Compared with FIrPic, both HOMO and LUMO energy levels are reduced by introducing more electron-withdrawing fluorine atoms to the chelating ligand.

Blue OLEDs were fabricated by doping Ir(tfpd)₂pic in SPPO1 with a device structure of ITO/NPB (35 nm)/mCP (15 nm)/x% Ir(tfpd)2pic:SPPO1 (30 nm)/SPPO1 (25 nm)/LiF (1 nm)/Al (120 nm). In the devices, ITO (indium tin oxide) and Al are the anode and the cathode respectively; 4, 4'-bis[N- (1-naphthyl)-N-phenyl amino] biphenyl (NPB) is the hole-transporting layer (HTL). The 1,3-bis (carbazol-9-yl)benzene (mCP) is used as a hole-transporting layer (HTL) and an exciton blocking laver (EBL), since a stepped progression of the HOMO energy level can be formed between NPB and SPPO1, which can facilitate hole injection and transporting. In addition, mCP possesses a high-lying triplet energy of 2.9 eV, which can block the back energy transfer from the emitter to the hole transporter. SPPO1 also has a high triplet energy of 2.9 eV [8c], was used as both the host for the blue dopant and the electrontransporting layer (ETL). The Ir(tfpd)₂pic doped SPPO1 layer is the emitting layer (EML). Other than Ir(tfpd)₂pic, all other organic materials of OLED grade were purchased from Lumtec Ltd and used without further purification.

The devices with different Ir(tfpd)₂pic doping concentrations (4, 6 and 8%) show almost the same EL spectra centered at 488 nm (Fig. 2). Similarity of the PL and the EL spectra suggests that the EL is indeed due to the emission from Ir(tfpd)₂pic. With increasing applied voltage, all these devices showed almost unchanged EL spectra, which indicate that effective charge capture and complete energy transfer occurred between SPPO1 and Ir(tfpd)₂pic.



Fig. 1. Absorption and photoluminescence (PL) spectra of lr(tfpd)₂pic in dichloromethane solution, inset shows molecule structure of lr(tfpd)₂pic.



Fig. 2. EL spectra of the blue OLEDs with different dopant concentrations, the inset shows the molecule structure of SPPO1.

Key performance parameters of the blue OLEDs with different Ir(tfpd)₂pic concentrations are listed in Table 1. Fig. 3 shows the efficiencies and J-V-L characteristics of the blue device with 6% Ir(tfpd)₂pic doped in SPPO1. Operating voltages requires for giving a luminance of 1, 1000 and 10000 cd/m^2 are respectively 3.9, 7.1 and 9.7 V. The device shows a maximum efficiency of 41.4 lm/W (52.6 cd/A). Performances of the present blue phosphorescent OLEDs are among the best reported as summarized in Table 2. [5g,8b,8c,14-20] Notably, the efficiency of our device is much higher than that of the FIrPic-based device using the same host and electron transporting material [8c]. It is also worth noting that the efficiency roll-off at high current densities is relatively mild in the present device (Fig. 3). Even at a luminance of 1000 cd/m^2 , the current efficiency and power efficiency of the device are still as high as 43.8 cd/A and 25.2 lm/W, respectively. This is obviously better than typical blue phosphorescent OLEDs. The mild efficiency rolloff should be attributed to the efficient excition blocking of mCP and SPPO1. In addition, more fluorination on the ligand may hinder self-quenching due to molecular interactions and improve holeelectron balance in charge injection [21].

A convenient approach to obtain white light emission is to use blue and yellow emitters. Owing to the successful development of the high efficiency blue OLEDs, we accordingly tried to fabricate

Table 1

Characteristics of blue and white OLEDs with different doping concentrations (volume) of $Ir(tfpd)_2pic$ and $Ir(npy)_2acac$ (Type a for $Ir(tfpd)_2pic$, Type b for $Ir(npy)_2acac$).

Туре	Conc.	$\eta_{\rm c}({\rm cd}/{\rm A})$	$\eta_{\rm p}({\rm lm/w})$	V@1 cd/m ² (V)	V@1000 cd/m ² (V)	V@20 mA/cm ² (V)	$CIE[x, y]^c$
Blue ^a	4.0%	45.91	32.77	3.93	6.99	8.65	0.18, 0.47
Blue ^a	6.0%	52.64	41.35	3.94	7.11	8.72	0.18, 0.48
Blue ^a	8.0%	49.65	39.00	4.00	7.13	8.75	0.18, 0.48
White ^b	0.5%	57.42	45.10	3.65	5.63	6.70	0.24, 0.50
White ^b	1.0%	54.54	48.96	3.62	5.65	6.74	0.32, 0.51
White ^b	2.0%	48.26	43.33	3.60	5.72	6.73	0.37, 0.53
White ^b	2.5%	46.41	41.66	3.53	5.81	6.79	0.39, 0.53
White ^b	3.0%	44.37	39.83	3.43	5.73	6.72	0.40, 0.53

 η_c , η_p are the maximum current efficiency and power efficiency respectively. ^a Blue OLED: ITO/NPB(35 nm)/mCP(15)/x%Ir(tfpd)₂pic:SPPO1(30 nm)/SPPO1 (25 nm)/LiF(1 nm)/Al (120 nm).

 b White OLED: ITO/NPB(40 nm)/x%Ir(npy)_2acac:mCP(10 nm)/6%Ir(tfpd)_2 pic:SPPO1(20 nm)/TPBi(40 nm)/LiF(1 nm)/Al(120 nm).

^c The value taken at $L = 100 \text{ cd/m}^2$.



Fig. 3. Current efficiency and power efficiency versus current density of the blue OLED with 6% of Ir(tfpd)₂pic doped in SPPO1, inset depicts I-V-L characteristics of the device.

 Table 2

 Performance of Recently reported blue phosphorescent emitting OLEDs.

Emitter	Host	$V_{\mathrm{on}}\left(V\right)$	η_c^a (cd/A)	η_p^b (lm/w)	Emisdion peak (nm)	Ref.
Ir(tfpd)2pic	SPPO1	3.9	52.6	41.4	488	This work
FIrPic	SPPO1	_	37.2	27.1	475	[8c]
FIr6	UGH2	_	_	18.0	460	[14]
FIrN4	SimCP	_	_	7.2	462	[8b]
Ir(dfppy) ₂ (tpip)	mCP	3.2	25.5	23.5	489	[15]
Ir(dbfmi)	PO9	2.6	28.6	35.9	445	[16]
$Ir(P_{-}C_{2})(PPh_{3})(OAc)$	CzSi&UGH2	4.1	22.3	16.9	473	[17]
Ir(fbppz) ₂ (dfbdp)	CzSi&UGH2	4.4	11.3	8.6	458	[18]
Ir(mpmi) ₂ (pypz)	BSB-CzSi	4.2	28.1	19.6	480	[19]
(dfbmb) ₂ Ir(fptz)	CzSi&UGH2	4.0	6.3	4.0	458	[20]
FIrPic	SiCa	3.5	41.5	31.0	475	[5g]

^a Maximum current efficiency.

^b Maximum power efficiency.

two-color WOLEDs by using another yellow emitter. In our previous report, Iridium(III) bis [2 - (2-naphthyl)-pyridine] (acetylacetonate) (Ir(npy)₂acac) based device exhibits highly efficient yellow emission with a maximum power efficiency of 22 lm/W, a emission peak



Fig. 4. EL spectra of various white OLEDs with different concentrations of yellow dopant, insets shows a photograph of WOLEDs at 5 V and the molecule structure of $Ir(npy)_2acac$.



Fig. 5. Current efficiency and power efficiency versus current density of the white OLED with 1% of $lr(npy)_2acac$, inset depicts J-V-L characteristics of the device.

at 550 nm along with a CIE coordinates (0.45, 0.54) [22]. Thus, Ir(npy)₂acac is used here as the yellow emitter to combine the blue emission of Ir(tfpd)₂pic to obtain white emission. WOLEDs with a configuration of ITO/NPB (40 nm)/x% Ir(npy)₂acac:mCP (10 nm)/ 6%Ir(tfpd)2pic:SPPO1 (20 nm)/TPBi (40 nm)/LiF (1 nm)/Al (120 nm) have been fabricated, 2, 2', 2"-(1,3,5-benzinetrivl)-tris(1-phenvl-1-H-benzimidazole) (TPBi) is used as the ETL. Fig. 4 shows normalized EL spectra of the WOLEDs which are greatly influenced by the doping concentrations yellow emitter. With increasing Ir(npy)₂acac concentration from 0.5 to 3.0%, relative intensity of the yellow emission increases gradually. This shift is partly due to the increase of the charge trapping sites formed by Ir(npy)₂acac and the changes of the exciton recombination zone. Fig. 5 shows the efficiency versus the current density of the 1% Ir(npy)₂acac device. Corresponding I-V-L curves are shown in the inset. Operating voltages required for giving a luminance of 1, 1000 and 10000 cd/m^2 are respectively 3.62, 5.65 and 7.59 V. The low operating voltages suggest effective charge transport and exciton recombination in the devices. Key performance parameters of the WOLEDs with different doping concentrations are listed in Table 1. With a doping concentration of 1% Ir(npy)2acac, the best performance of the WOLEDs can be achieved with a maximum efficiency of 49.0 lm/W (54.5 cd/A) and a CIE coordinates of (0.32, 0.52). Performance of the WOLEDs is also among the highest value reported for WOLEDs without using an optical out-coupling device or a p-i-n structure (Table 3).

Table	e 3

Performance of recently reported WOLEDs.

Emitter					CIE x, y	Ref.
Red	Green	Blue	Yellow	(lm/w)		
		Ir(tfpd) ₂ pic	lr(npy) ₂ acac	49.0	0.32, 0.52	This work
		FIrPic	Ir(npy) ₂ acac	29.2	0.32, 0.45	[23]
Ir(phq) ₃	Ir(ppy) ₃	FIrPic		26.0	0.42, 0.48	[24]
		FIrPic	(F-bt) ₂ Ir(acac)	34.0	0.35, 0.44	[25]
PQ2Ir		FIrPic		53.0	0.34, 0.40	[26]
		FIrPic	thiopyridinyl based Ir	32.0	0.40, 0.44	[27]
Ir(MDQ)2acac		FIrPic		49.3	0.49, 0.41	[28]
		FIrPic	(fbi) ₂ Iracac	45.2	0.34, 0.41	[29]
PQIr	Ir(ppy)3	FIr6		40.0	0.37, 0.40	[30]
			(bzq) ₂ lr(dipba)	48.8	0.35, 0.33	[31]
Ir(pq) ₂ acac	Ir(ppy) ₃	FCNIrPic		47.0	0.39, 0.46	[32]
Ir(2-phq) ₃	Ir(ppy) ₃	FIrPic		18.7	0.31, 0.51	[33]

^a Maximum power efficiency.

High efficiency of the WOLEDs is attributed to the following reasons. Firstly, the high PL quantum efficiency of $Ir(tfpd)_2pic$ is one of the key factors, which lead to more photons generation. Secondly, appropriate device structure and suitable energy levels can facilitate the injection and transporting for both electrons and holes. Morever, the HOMO and LUMO energy levels of $Ir(npy)_2acac$ and $Ir(tfpd)_2pic$ are respectively embraced by those of mCP and SPPO1. That is, $Ir(npy)_2acac$ and $Ir(tfpd)_2pic$ can act as traps for both electrons and holes in their respective hosts and thus enhances the chance of exciton formation.

3. Conclusions

In summary, highly efficient blue phosphorescent organic lightemitting devices have been achieved by using a new heteroleptic Iridium complex, Iridium(III) bis [(2,3,4-trifluorophenyl) -pyridinato-N, C²] picolinate (Ir(tfpd)₂pic). For a SPPO1: 6% Ir(tfpd)₂picdevice, blue emission with a maximum power efficiency of 41.4 lm/W (52.6 cd/A) has been realized with the emission peak centered at488 nm. To the best of our knowledge, performances of the bluephosphorescent OLEDs are among the best reported. Furthermore,the combination with Iridium(III) bis [2-(2-naphthyl)-pyridine](acetylacetonate) (Ir(npy)₂acac) as the yellow emitter yields highlyefficient white emission with a maximum efficiency of 49.0 lm/W(54.5 cd/A). The results indicate that Ir(tfpd)₂pic is a promising bluephosphor for applications in high performance blue and whiteOLEDs.

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