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Novel furo[3,2-c]pyridine based iridium complex for highperformance organic light-emitting diodes with over 30% external quantum efficiency

Accepted 00th January 20xx DOI: 10.1039/x0xx00000x

Received 00th January 20xx,

Zhimin Yan,^{a,b} Yanping Wang,^c Jiaxiu Wang,^b Yue Wang^{*,a}, Junqiao Ding^{*,b}, Lixiang Wang^{*,b}

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A novel furo[3,2-c]pyridine based Ir complex, namely (pfupy)₂Ir(acac), has been developed by replacing sulfur with oxygen in the C^N ligand. Compared with the thiophenecontaining (pthpy)2Ir(acac), the LUMO level is elevated while the HOMO level keeps almost unchanged for the resultant furancontaining (pfupy)₂Ir(acac). As a consequence, the emissive maximum is blue-shifted from 556 nm of (pthpy)₂Ir(acac) to 538 nm of (pfupy)₂Ir(acac) together with an improved photoluminescence quantum yield of 0.80. The corresponding device based on (pfupy)₂Ir(acac) realizes a record-high external quantum efficiency (EQE) of 30.5% (110.5 cd/A) without any outcoupling technology. Even at a luminance of 1000 and 5000 cd/m², the EQE still remains at 26.6% (96.4 cd/A) and 25.6% (92.7 cd/A), respectively, indicative of the gentle efficiency roll-off. The results clearly demonstrate the great potential of furan-based functional materials applied in OLEDs.

The development of organic electronics has promoted the ongoing search for new organic semiconducting materials,¹⁻¹¹ such as furan-based compounds.¹²⁻¹⁶ Compared with the thiophene analogues, these furan derivatives have a tendency to be more tightly packed¹³ because of the smaller atomic radius of oxygen (1.52 Å) than sulfur (1.80 Å), which facilitates carrier transport in the solid state.¹⁷ Meanwhile, the larger electronegativity of oxygen relative to sulfur may deepen the highest occupied molecular orbital (HOMO) and lowest

^a State Key Laboratory of Supramolecular Structure and Materials College of Chemistry, Jilin University Chanachun, 130012, P. R. China

- *E-mail: junqiaod@ciac.ac.cn, lixiang@ciac.ac.cn
- ^c School of Materials Science and Engineering

unoccupied molecular orbital (LUMO) levels, implying the possibility of higher open circuit voltages.¹⁸ In addition, they are highly emissive, whose photoluminescence quantum yields (PLQYs) are found to be impressively enhanced after the exchange of sulfur for oxygen.¹⁹ Inspired by such characteristics, nowadays a series of furan-based semiconductors has been successfully exploited for organic solar cells (OSCs) and organic field-effect transistors (OFETs). For example, a 2D benzo[1,2-b:4,5-b']difuran conjugated polymer PBDF-T1 was presented as a promising candidate for highly efficient OSCs, revealing a distinguished power conversion efficiency of 9.4%.¹⁸ And a furan-thiophene quinoidal compound TFT-CN was designed to show high electron mobility up to 7.7 cm² V¹ s⁻¹, two orders of magnitude



Figure 1. The molecular structures of $(pthpy)_2 lr(acac)$ and $(pfupy)_2 lr(acac)$ (a) and their corresponding HOMO/LUMO distributions obtained from DFT calculations (b).

^{*}E-mail: yuewang@jlu.edu.cn

^{b.} State Key Laboratory of Polymer Physics and Chemistry

Changchun Institute of Applied Chemistry, Chinese Academy of Sciences Changchun, 130022, P. R. China

Changchun University of Science and Technology

Changchun 130022, P. R. China

⁺Electronic Supplementary Information (ESI) available: Experimental details, synthesis and characterization data, crystal structures and packing diagrams, TGA, CV, electroluminescent characteristics of OLEDs. CCDC 1541442-1541443 See DOI: 10.1039/x0xx00000x

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higher than that of the only thiophene-based quinoidal TTT-CN.²⁰ However, in the aspect of organic light-emitting diodes (OLEDs), the study of the respective functional materials containing furan remains less attention.²¹

In this communication, we report a novel iridium (Ir) complex (pfupy)₂Ir(acac) by using furo[3,2-c]pyridine as the ligand. As depicted in Figure 1, the replacement of sulfur by oxygen in the C^N ligand leads to an up-shift of the Ir complex's LUMO level without varying its HOMO level. Compared to the thiophene-containing reference $(pthpy)_2 Ir(acac)$ ($\lambda_{em} = 556$ nm, $\Phi_{PL} = 0.68$), a hypsochromic emission of 538 nm is observed for the resultant furancontaining complex (pfupy)2Ir(acac) associated with a PLQY of 0.80. Without any out-coupling technology, the corresponding device based on (pfupy)₂Ir(acac) gives a maximum external quantum efficiency (EQE) as high as 30.5% (110.5 cd/A), which slightly decays to 25.6% (92.7 cd/A) even at a high luminance of 5000 cd/m^2 . To the best of our knowledge, this is the first report on furan-based functional materials used for highperformance OLEDs.

As described in Scheme S1, a modified procedure was adopted for the synthesis of (pfupy)₂Ir(acac). Starting from furfural that is cheap and widely used in industry, a Knoevenagel reaction was performed to give 2-furanacrylic acid (1). Then 1 was converted to its corresponding azide intermediate (2), followed by a cyclization to afford furo[3,2c]pyridin-4-ol (3). During the cyclization, diphenyl ether was initially utilized as the high boiling point solvent,²² inevitably leading to the block of the constant pressure funnel caused by its a little higher melting point of 28 °C than room temperature. To solve this problem, two methods were tried: i) A hot wind was provided to melt diphenyl ether, but failed because the azide 2 was partly decomposed before dropped into the reactive flask; ii) Diphenyl ether had better be replaced by another solvent simultaneously with low melting and high boiling points. So Dowtherm A (m.p. = 11 °C, b.p. = 258 °C) came into our sights, which is developed by Dow chemical company as the heat transfer fluid. In Dowtherm A, the azide 2 could be well dissolved and the cyclized product 3 was found to be recrystallized when cooling down. With the ease achievement of 3, the furo[3,2-c]pyridine based C^N ligand 5 could be facilely obtained after the chloro-hydroxyl exchange and subsequent Suzuki cross-coupling. Finally, the desired



Figure 2. ORTEP diagrams of $(pfupy)_2 Ir(acac)$ (a) and $(pthpy)_2 Ir(acac)$ (b) with thermal ellipsoids drawn at 50% probability level. For clarity, H atoms and solvent are removed.



Figure 3. UV-Vis absorption spectra in dichloromethane at 1×10^{5} M and PL spectra in toluene at 1×10^{5} M for $(pfupy)_2 Ir(acac)$ compared with $(pthpy)_2 Ir(acac)$.

furan-containing Ir complex (pfupy)₂Ir(acac) was prepared according to a well-known two-step route.²³ For comparison, the thiophene-based anologue (pthpy)₂Ir(acac) was also synthesized as the reference. Their corresponding molecular structures were fully characterized by ¹H NMR spectroscopy, MALDI-TOF mass spectrometry and elemental analysis (Figure S1). In addition, they are both thermally stable, possessing a decomposition temperature over 350 °C without any glass transition in the range of 25-300 °C (Figure S2).

Single crystals of (pthpy)₂Ir(acac) and (pfupy)₂Ir(acac) suitable for X-ray diffraction analysis were grown from the mixed solvents of dichloromethane and methanol. As one can

Complex	λ_{abs}^{a} [nm]	λ _{em} ^b [nm]	$\Phi_{_{\textrm{PL}}}^{}\textrm{c}}$	τ ^d [μs]	HOMO/LUMO ^e [eV]	T _d ^f [^o C]	k _r ^g [s ⁻¹]	k _{nr} ^g [s ⁻¹]
(pthpy)2lr(acac)	299, 362, 439, 495	556	0.68	1.08	-5.14/-2.52	369	6.3×10 ⁵	3.0×10 ⁵
(pfupy) ₂ Ir(acac)	279, 355, 426, 479	538	0.80	1.04	-5.15/-2.40	354	7.7×10 ⁵	1.9×10 ⁵

Table 1. Photophysical, electrochemical and thermal properties of (pthpy)₂Ir(acac) and (pfupy)₂Ir(acac).

^a Measured in 10⁻⁵ M dichloromethane solution; ^b Measured in 10⁻⁵ M toluene solution; ^c Measured in N₂-saturated toluene solution with Ir(ppy)₃ ($\Phi_{PL} = 0.97$) as the reference; ^d Estimated from the transient PL spectrum measured in N₂-saturated toluene solution excited by a 355 nm pulse; ^e HOMO = - e(E_{ox, onset} + 4.8 V), LUMO = - e(E_{red, onset} + 4.8 V), where E_{ox, onset} and E_{red, onset} are the onset value of the first oxidation and reduction waves, respectively; ^f Decomposition temperature corresponding to a 5% weight loss; ^g Calculated according to the equations: $\tau = 1/(k_r + k_{nr})$ and $\Phi = k_r/(k_r + k_{nr})$.

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DOI: 10.1039/C7TC03937A

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see in Figure 2, both the Ir complexes assume the same pseudo-octahedral coordination geometry around the Ir atom, retaining the mutual trans-N,N- and cis-C,C-chelate disposition of the chloride-bridged precursors. The average Ir-C bond length of (pfupy)₂Ir(acac) (1.980(6) Å) is close to that of (pthpy)₂Ir(acac) (1.979(4) Å), whereas the average Ir-N and Ir-O bond lengths of (pfupy)₂Ir(acac) (2.042(5) Å and 2.158(4) Å, respectively) are longer to those of (pthpy)₂Ir(acac) (2.034(3) Å and 2.150(2) Å, respectively) (Table S1-S3). Furthermore, a π - π interaction between furo[3,2-c]pyridine and phenyl ring with a contact distance of 3.460-3.704 Å is observed in the furancontaining complex (pfupy)₂Ir(acac) (Figure S3). This is quite the different from thiophene-containing complex (pthpy)₂Ir(acac), showing an obvious packing between two neighboring thieno[3,2-c]pyridine units (Figure S4).

Cyclic voltammetry (CV) was used to investigate the electrochemical property of (pfupy)₂Ir(acac) compared with (pthpy)₂Ir(acac). Similar to (pthpy)₂Ir(acac), (pfupy)₂Ir(acac) exhibits a guasi-reversible oxidation wave (Figure S5). The HOMO energy level of (pfupy)₂Ir(acac) (-5.15 eV) is nearly the same as that of (pthpy)₂Ir(acac) (-5.14 eV). During the cathodic sweeping, nevertheless, an irreversible reduction appears at a more negative potential for (pfupy)₂Ir(acac). Its corresponding LUMO energy level is determined to be -2.40 eV, which is about 0.12 eV higher than that of (pthpy)₂Ir(acac) (-2.52 eV). Density functional theory (DFT) calculations were further carried out on the basis the geometries from the X-ray diffraction data. As for both the complexes (pthpy)₂Ir(acac) and $(pfupy)_2 Ir(acac)$, the HOMO mainly comes from the π orbital of phenyl and $t_{\rm 2g}$ orbital of Ir ion, while the LUMO is distributed on phenyl and thieno[3,2]pyridyl or furo[3,2c]pyridyl segments (Figure 1b). Reasonably, on going from (pthpy)₂Ir(acac) to (pfupy)₂Ir(acac), the calculated HOMO levels keep almost unchanged, and the LUMO ones are found to be up-shifted owing to the more electron-rich character of furan than thiophene.¹³ The trend is in well agreement with the CV experiments.

Figure 3 illustrates the UV-Vis and photoluminescence (PL) spectra in solutions for (pthpy)₂Ir(acac) and (pfupy)₂Ir(acac). As can be clearly seen, they both show multiple absorption bands: the strong bands below 400 nm are attributed to the spinallowed ${}^{1}\pi$ - π * transition of the cyclometalated C^N ligands; the 400-450 nm shoulders can be assigned to the spin-allowed metal to ligand charge transfer transition (¹MLCT); and the weak absorption above 450 nm originates from the mixing ${}^{3}\pi$ - π^{*} and ${}^{3}\text{MLCT}$ transitions. 24 Despite of this, the absorption spectrum of (pfupy)2Ir(acac) moves towards a shorter wavelength with respect to (pthpy)₂Ir(acac). Correspondingly, the maximum emission is blue-shifted from 556 nm of (pthpy)₂Ir(acac) to 538 nm of (pfupy)₂Ir(acac), which is consistent with the above-mentioned electrochemical behaviour. Furthermore, attributable to the faster radiative decay rate and lower non-radiative decay rate, (pfupy)2Ir(acac) has an improved PL quantum efficiency of 0.80 as well as a close lifetime of 1.04 µs (Table 1 and Figure S6). These observations clearly demonstrate the superiority of furancontaining materials to thiophene-containing ones.



DOI: 10.1039/C7TC03937A

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Figure 4. The luminance dependence on EQE. Inset: EL spectra at a driving voltage of 6 V.

To evaluate the electroluminescence (EL) property, the device of (pfupy)₂Ir(acac) was fabricated with a configuration of ITO/MoO₃ (10 nm)/TAPC (60 nm)/TCTA (5 nm)/ CBP : x wt.% (pfupy)₂Ir(acac) (20 nm)/BmPyPB (35 nm)/LiF (1 nm)/AI (Figure S7). Here MoO₃ and LiF serve as the hole- and electroninjecting materials, respectively; TAPC (4,4'-(cyclohexane-1,1diyl)bis(N,N-di-p-tolylaniline)) and BmPyPB (3,3",5,5"tetra(pyridin-3-yl)-1,1':3',1"-terphenyl) act as the hole- and electron-transporting materials, respectively; TCTA (tris(4-(9Hcarbazol-9-yl)phenyl)amine) is selected as the buffer layer²⁵ as well as exciton-blocking material; and (pfupy)₂Ir(acac) is doped into the host CBP (4,4'-di(9H-carbazol-9-yl)-1,1'-biphenyl) as the emitting layer. The triplet energy of CBP (2.6 eV) is higher than that of (pfupy)₂Ir(acac) (2.3 eV), thus avoiding the triplet energy back transfer from (pfupy)₂Ir(acac) to CBP. And the doping concentration is tuned in the range of 4-12 wt.% to optimize the device performance.

The EL spectrum matches well with the PL counterpart (Inset in Figure 4), suggesting that the EL is mainly from the Ir complex (pfupy)₂Ir(acac). Meanwhile, it is almost independent on the doping concentration, giving similar Commission International De L'Eclairge (CIE) coordinates of (0.44, 0.55). Despite of this, with the increasing doping concentration, the turn-on voltage (at 1 cd/m²) is reduced from 3.2 V to 2.8 V, and the current density at the same driving voltage is found to be enhanced gradually (Figure S8a). Given the much elevated HOMO of (pfupy)₂Ir(acac) than CBP, the high doping concentration could promote the direct exciton generation on (pfupy)₂Ir(acac), leading to the lower driving voltage and improved efficiency. Consequently, the best device performance is achieved at a 12 wt.% content, showing a peak current efficiency of 110.5 cd/A, a peak power efficiency of 124.0 lm/W and a peak EQE of 30.5% (Figure 4, Figure S8b and Table S4). Even at a high luminance of 1000 and 5000 cd/m^2 , the EQE still maintains at 26.6% and 25.6%, respectively, revealing an attenuation of about 12.8% and 16.1% relative to the maximum value. As discussed above, the fast radiative decay rate may responsible for such a slow efficiency roll-off at high luminance, which is beneficial to prevent the triplettriplet annihilation (TTA)²⁶ and triplet-polaron annihilation (TPA).²⁷ To our knowledge, the performance including the

DOI: 10.1039/C7TC03937A Journal Name

efficiency and its gentle efficiency roll-off is among the best ever reported for green devices without any out-coupling technology.²⁸⁻³⁰

Under the same conditions, the reference device based on 12 wt.% $Ir(ppy)_3$ doped into CBP gives an inferior EQE of 24.9% despite the different CIE coordinates (Figure S9 and Table S4). Therefore, the obtained over 30% EQE for (pfupy)₂Ir(acac) is without our expectation. The EQE can be well described by the following equation:³¹

 $EQE = \gamma \times \eta_{r} \times \eta_{PL} \times \eta_{out} = \gamma \times \eta_{PL} \times \eta_{out}$ (1)

where γ is the ratio of the recombined carriers to the injected carriers; η_r is the efficiency of radiative exciton production; η_{PL} is the PL quantum efficiency and η_{out} is the light out-coupling constant. Assuming that both γ and η_r are close to 1 as well as the determined η_{PL} of 0.80, η_{out} is estimated to be at least 0.38 according to the equation (1), much higher than the theoretical value.³² Consistent with the literatures,³³⁻³⁵ the favorable horizontal orientation of (pfupy)₂Ir(acac) in CBP may tentatively contribute to the high η_{out} and thus over 30% EQE when free of any out-coupling technology. Further experiments should be performed to verify this hypothesis, but they are now beyond the aim of this work.

In summary, we have newly designed and synthesized a furo[3,2-c]pyridine based Ir complex $(pfupy)_2$ Ir(acac) by changing sulfur with oxygen in the C^N ligand. Such a small variation of the molecular structure brings about the elevated LUMO level and unaffected HOMO level, thus resulting in a blue-shifted emission. The corresponding device of $(pfupy)_2$ Ir(acac) realizes a state-of-art EQE of 30.5% at maximum and 25.6% even at 5000 cd/m². In view of the achieved promising device efficiency associated with the gentle roll-off at high luminance, we believe that this work will shed light on the development of furan-based functional materials used for OLEDs in the future.

Acknowledgements

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This work was supported by the National Key Research and Development Program of China (No. 2016YFB0400701) and the National Natural Science Foundation of China (No. 51573183, 91333205, 21174144 and 51322308).

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A novel furo[3,2-c]pyridine Ir complex has been developed by replacing S with O in the C^N ligand, which shows a blue-shifted emission and over 30% EQE.