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Carbazole-pyrene derivatives for undoped organic light-emitting devices

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ABSTRACT

Two carbazole–pyrene derivatives, namely 3,6-dipyrenyl-9-(4'-tert-butylphenyl) carbazole (BPyC) and 3,6-dipyrenyl-9-(4'-pyrenylphenyl) carbazole (TPyC), have been designed and synthesized for application in organic light–emitting devices (OLEDs). While the two compounds have similar chemical structures and photoluminescent properties, OLEDs based on them show distinct electroluminescence (EL) spectra. The BPyC-based devices show a single peak saturated blue emission with CIE coordinates of (0.15, 0.18); whereas the TPyC-based devices exhibit two emission peaks at blue and yellow hues with CIE coordinates of (0.22, 0.29). The difference in their EL spectra is attributed to the substitution of the *t*-butyl unit of BPyC with a pyrenyl group to form TPyC, which effectively increases the electron-accepting TPBI. A high external quantum efficiency of 3.11% is achieved in the TPyC-based devices. Influences of chemical structure and fluorescent quantum yield on the efficiency of exciplex emission are discussed.

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1. Introduction

Organic light-emitting device (OLED) is emerging as a leading next generation technology for full-color flat-panel displays and ambient illumination sources because of its huge market potential and excellent performance in terms of brightness, operating voltage and color gamut. Incorporation of extrinsic dyes into conductive hosts is an effective way to customize emitting color of OLEDs that is particularly useful for fabricating white OLEDs with two complementary or three primary colors [1,2]. Electroluminescence (EL) in terms of Commission Internationale de L'Eclairage (CIE) coordinates can be fine-tuned and optimized through precise control of dopant concentration and thickness for each light-emitting layer (EML). However, this approach would complicate device configuration as well as fabrication process, and inevitably increases the total manufacturing cost. In addition, the shortened device lifetime due to morphological phase separation caused by operation induced heating is another challenge for doped OLEDs.

Recent studies [3–9] have demonstrated a simple way to tailor the emitting color via interfacial excited-state charge-transfer complex (termed exciplex) formation to control emission peak location and/or intensity by using different binary combinations of electron-donor/holetransporting materials (HTMs) and electron-acceptor/electron-transporting materials (ETMs), i.e., $hv_{\text{exciplex}} = E_{\text{LUMO}}$ (ETM) – E_{HOMO} (HTM), ignoring the band bending at the organic/organic interface [10]. In the equation, E_{HOMO} and E_{LUMO} are the energies of the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO), respectively. However, only certain combinations of materials were reported to be capable of giving

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exciplex emission and their external quantum efficiencies $(\eta_{\rm E})$ are usually low (e.g., m-MTDATA/Alq₃ (0.22%) [4], 2-TNATA/Alq₃ (0.30%) [5] and TPD/PBD (1.10%) [6]). Thus, the appearance of exciplex is often considered as one of the main reasons responsible for poor device performance. On the other hand, Palilis et al. recently reported exciplexbased OLEDs using NPB/PPSPP, and achieved an exceptionally high $\eta_{\rm E}$ of 3.40% [7]. Later, our group demonstrated the TPyPA/BPhen based exciplex emitting devices with $\eta_{\rm E}$ as high as 3.93% [8]. It is proposed that matched carrier mobilities, high photoluminescence quantum yield (Φ_{PL}), and suitable electron energy levels of organic materials are the prerequisites for efficient exciplex-based OLEDs [8]. These findings provide guidelines for designing organic materials with desirable physical properties to facilitate high efficiency exciplex emission.

In addition to triphenylamine [4,5,8], both pyrene [8,11] and carbazole [9,12] and their derivatives have also been employed for exciplex emission, owing to their excellent luminescence characteristics, structural stabilities, and particularly their strong electron-donating tendencies when contacting with different electron-acceptors. However, the influences on how the chemical structures of organic materials affecting the nature of exciplex emission have seldom been studied. In this work, two carbazolebased compounds end-capped with one t-butyl and two pyrene groups, namely 3,6-dipyrenyl-9-(4'-tert-butylphenyl) carbazole (BPyC) and three pyrene groups, namely, 3,6-dipyrenyl-9-(4'-pyrenylphenyl) carbazole (TPyC) have been synthesized. While the two compounds have very similar structures and photoluminescent spectra, OLEDs using them as host emitters show distinct EL characteristics. Particularly, the BPyC-based devices ($\eta_{\rm E}$ = 2.21%) show only a single blue emission peak while the TPyC-based devices show two emission peaks with higher maximum $\eta_{\rm F}$ of 3.11%. Reason for these differences is attributed to the higher degree of electron-donating property with increasing number of pyrene groups. This results in the formation of the yellow exciplex emission which shifts the EL spectrum from saturated blue (BPyC) to near white (TPyC). It is worth noting that both carbazole derivatives have $\Phi_{\rm PI}$ of about 95%, which are much higher than that of our previously reported triphenylamine TPyPA (Φ_{PL} = 80%) [8]. This results in a higher maximum $\eta_{\rm F}$ of the TPyC-based devices, as compared to that of a similarly structured TPyPAbased OLED ($\eta_{\rm E}$ = 2.83%). Based on these results the influences of chemical structures and $\Phi_{\rm PL}$ on the efficiency of exciplex emission are discussed.

2. Experimental

2.1. Materials and instruments

All reagents and solvents for the synthesis of BPyC and TPyC were purchased from either Sigma–Aldrich or Acros Organics and used as received without further purification. ¹H NMR spectra were recorded with the use of a Bruker DPX-400 spectrometer (400 MHz). Mass spectrometry was performed on a PE SCIEX LC/MS spectrometer. Elemental analyses were carried out on a Vario EL Elementar by the Flash EA 1112 method. Modulated differential scanning calorimetry (MDSC) and thermogravimetric analysis (TGA) were performed under a nitrogen atmosphere at a heating rate of 10 °C min⁻¹ on a TA Instruments MDSC 2910 and TGA Q50, respectively. Solution Φ_{PL} were determined by using the method of Demas and Crosby [13] with a degassed aqueous solution of quinine sulfate as a reference. Optical absorption and photoluminescence (PL) spectra were measured with a Perkin-Elmer Lambda 2S UV/vis spectrometer and a Perkin-Elmer LS 50B luminescence spectrometer, respectively. Ultraviolet photoelectron spectroscopic (UPS) studies were performed in a VG ESCALAB 220i-XL surface analysis system with an unfiltered He I (21.22 eV) gas discharge lamp under a base vacuum of 10^{-10} Torr.

2.2. Fabrication and characterization of OLEDs

Transparent indium tin oxide (ITO) pre-coated and patterned glass slides with a sheet resistance of 30 Ω square⁻¹ were used as anodic substrates. Prior to successive film deposition, they were thoroughly cleaned with Decon 90, rinsed in de-ionized water, dried in an oven, and finally treated in an ultraviolet-ozone cleaner. OLEDs consisting of a hole-transporting layer (HTL) of N,N'-diphenyl-1,1'biphenyl-4,4'-diamine (NPB), a hole-transporting lightemitter of BPyC or TPyC, an electron-transporting layer (ETL) of 1,3,5-tris(N-phenylbenzimidazol-2-yl) benzene (TPBI) or 4,7-diphenyl-1,10-phenanthroline (BPhen), and a LiF/Al cathode. All films were thermally deposited sequentially without vacuum break in an evaporation chamber under a base pressure of $\sim 10^{-6}$ Torr. Deposition rates were monitored with quartz oscillation crystals and controlled at a rate of $0.1-0.2 \text{ nm s}^{-1}$ for both organic and metal layers. A shadow mask was used to define the cathode and to make four 0.1 cm² devices on each substrate. Current density-voltage-luminance (I-V-L) characteristics, EL spectra, and CIE coordinates were measured simultaneously with a programmable Keithley model 237 power source and a Photoresearch PR650 spectrometer. All measurements were carried out at room temperature under ambient atmosphere without any encapsulation.

3. Results and discussion

3.1. Material syntheses

Molecular structures of BPyC and TPyC and their synthetic routes are shown in Scheme 1. Incorporation of the 9-phenylcarbazole group is expected to enhance holetransporting properties of the two compounds. BPyC and TPyC can be easily prepared with high yields using typical Suzuki coupling reactions from 3,6-dibromo-9-(4-*tert*butylphenyl)-9*H*-carbazole [14], 3,6-dibromo-9-(4-*b*romophenyl)-9*H*-carbazole [15], and commercial starting material pyrene-1-boronic acid, respectively, according to reported procedures [16]. Molecular structures of the final compounds were confirmed by ¹H nuclear magnetic resonance, mass spectrometry, and elemental analysis.



Scheme 1. Synthetic routes and molecular structures of 3,6-dipyrenyl-9-(4'-tert-butylphenyl) carbazole (BPyC) and 3,6-dipyrenyl-9-(4'-pyrenylphenyl) carbazole (TPyC).

3.2. Chemical and photophysical properties

The rigid molecular structures in the two carbazole derivatives are beneficial to thermal stability, as manifested by their high decomposition temperatures (T_d) of 496 and 516 °C, and melting temperatures (T_m) of 293 and 245 °C for BPyC and TPyC, respectively (T_d corresponds to 5% weight loss in the TGA measurements whereas T_m was determined by MDSC) although no obvious glass transitions were observed.

Optical absorption and PL spectra of BPyC and TPyC vacuum-deposited solid thin-films on quartz plates are depicted in Fig. 1. The longest wavelength absorption maxima ($\lambda_{abs, max}$) appear at 358 and 369 nm for BPyC and TPyC, respectively, while the PL spectra show intense blue emissions peaked at 443 nm (BPyC) and 465 nm (TPyC) under excitation at 385 nm. It is well recognized that an increase in conjugation length would generally result in red-shifts in both λ_{abs} and PL. Indeed, the addition of a pyrene arm (from BPyC to TPyC) does red-shift the absorption and PL peaks by respectively 11 and 22 nm. From the PL quantum yield measurement, Φ_{PL} of BPyC and TPyC in solution are 94.8% and 95.0%, respectively. These high Φ_{PL} values may be attributed to large Stoke shifts (difference between positions of band maxima of the absorption and emission spectra) in both organic materials that can effectively avoid re-absorption of the emitted photons during the PL process.



Fig. 1. Optical absorption (Abs) and photoluminescence (PL) spectra of BPyC and TPyC solid thin-films on quartz substrates. Inset: PL spectra of 10% TPBI:TPyC, TPBI, and TPyC thin-films, respectively.

UPS was employed to measure the E_{HOMO} values of the carbazole derivatives, in which they are found to be 5.57 and 5.67 eV for BPyC and TPyC, respectively. By subtracting from the optical band gaps determined from long wavelength onsets of the absorption bands, E_{LUMO} are estimated to be 2.58 eV (for BPyC) and 2.71 eV (for TPyC). Table 1 summarizes the thermal and photophysical data of BPyC and TPyC.

3.3. Device characteristics

Devices with the structures of ITO/EML (100 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm) (w/o NPB) and ITO/NPB (70 nm)/EML (30 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm) (w/NPB) were fabricated to evaluate the properties of BPyC or TPyC as hole-transporting EML. Fig. 2 depicts EL spectra of the BPyC-based OLEDs with (upper) and without (lower) the NPB layer viewed in the normal direction at the luminance of 10, 100, and 1000 cd m^{-2} . Both devices exhibit similar pure blue emissions with mild change in CIE coordinates of (0.17, 0.25) (Device w/o NPB) and (0.15, 0.18) (Device w/NPB) over a wide range of luminance from 10 to 1000 cd m^{-2} . This suggests that the hole and electron recombination is well confined within the emissive BPyC layer. In addition, these undoped blue-emitting devices show high efficiencies (as shown in Fig. 3) and low operating voltages (as shown in Fig. 4) in which current and power efficiencies ($\eta_{\rm C}$ and $\eta_{\rm P}$), and $\eta_{\rm E}$ and operating voltage at 20 mA cm⁻² are 4.3 cd A⁻¹, 4.5 lm W⁻¹, 2.49%, and 3.7 V for device without NPB, and are 2.9 cd A^{-1} , 2.7 lm W^{-1} , 2.21%, and 3.5 V for device with NPB. After



Fig. 2. Electroluminescence spectra of the BPyC-based OLEDs without (lower) and with (upper) NPB viewed in the normal direction at the luminance of 10, 100, and 1000 cd m^{-2} , respectively.



Fig. 3. (a) Current efficiency (η_c), external quantum efficiency (η_E) and (b) power efficiency (η_P) as a function of current density. Device configurations: ITO/BPyC (100 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm); ITO/ NPB (70 nm)/BPyC (30 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm).

Table 1

Thermal and photophysical properties of BPyC and TPyC. T_d and T_m were determined by thermogravimetric analysis (TGA) and modulated differential scanning calorimetry (MDSC) measurements at a same heating rate of 10 °C min⁻¹ under a nitrogen atmosphere whereas T_d corresponds to 5% weight loss. λ_{abs} and λ_{PL} were measured with solid thin-film. E_{opt} was estimated from the long wavelength onset of the absorption band. E_{HOMO} was defined by ultraviolet photoelectron spectroscopy (UPS) while E_{LUMO} was estimated by $E_{LUMO} = E_{HOMO} - E_{opt}$. Φ_{PL} was measured in solution.

Compound	$T_{\rm m}/T_{\rm d}~(^{\circ}{\rm C})$	$\lambda_{abs, max} (nm)$	λ_{PL} (nm)	Stokes shift (nm)	$\lambda_{abs, onset} (nm)/E_{opt} (eV)$	$E_{\rm HOMO}/E_{\rm LUMO}~({\rm eV})$	$arPsi_{ ext{PL}}$ (%)
ВРуС	293/496	358	443	85	415/2.99	5.57/2.58	94.8
ТРуС	245/516	369	465	96	419/2.96	5.67/2.71	95.0



Fig. 4. Current density and luminance characteristics as a function of voltage in various devices. Device configurations: ITO/TPyC (100 nm)/ TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm); ITO/NPB (70 nm)/TPyC (30 nm)/ TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm); ITO/BPyC (100 nm)/TPBI (20 nm)/ LiF (0.5 nm)/Al (100 nm); ITO/NPB (70 nm)/BPyC (30 nm)/TPBI (20 nm)/ LiF (0.5 nm)/Al (100 nm).

insertion of NPB at the ITO/BPyC interface, the $E_{\rm HOMO}$ of NPB can function as a stepping-stone for hole-injection, similar to the case of ITO/m-MTDATA/NPB [17]. This improvement for hole-injection definitely reduces the driving voltage. It is believed that the amounts of hole and electron are balanced inside the device w/o NPB. However, the excess of holes in the device w/NPB eventually reduces the device efficiency. Nevertheless, these relatively high device efficiencies and low driving voltage are comparable or even better than current state-of-the-art undoped emitters with saturated blue fluorescence [18–20].

Unexpectedly, the extra pyrene arm in TPyC leads to large difference in EL properties. Fig. 5 depicts EL spectra of the TPyC-based OLEDs with (middle) and without (lower) NPB viewed in the normal direction at the luminance of 10, 100, and 1000 cd m⁻². In contrast with the PL spectrum of neat thin-film, both devices demonstrate two broad emission peaks at ca. 475 and 558 nm. The blue emission peak at 475 nm is contributed from the TPyC emission,



Fig. 5. Electroluminescence spectra of the TPyC-based OLEDs without (lower) and with (middle) NPB viewed in the normal direction at the luminance of 10, 100, and 1000 cd m⁻², respectively. Electroluminescence spectra for devices with the structures of ITO/TPyC/BPhen/LiF/Al and ITO/ NPB/TPyC/BPhen/LiF/Al (upper) are also inserted.

whereas the red-shifted band centered at 558 nm appears to be arising from the exciplex emission formed at the TPyC/TPBI interface. This can be confirmed with the PL spectrum of the TPBI:TPyC blended film as shown in the inset of Fig. 1. It is considered that coupling between the strong electron-donating TPyC and the strong electronaccepting TPBI results in exciplex emission at their interface. Combining with the intrinsic blue emission at 475 nm, near white light emission with CIE coordinates of (0.21, 0.26) and (0.22, 0.29) were obtained for the devices without and with the NPB layer, respectively. In fact, the CIE coordinates change even closer to white emission of (0.24, 0.30) and (0.25, 0.33) when the ETL (i.e., TPBI) is replaced by BPhen (as shown in the two top-most spectra of Fig. 5).

Fig. 6 demonstrates efficiencies of the TPyC-based devices as a function of current density. With the insertion of NPB layer, $\eta_{\rm C}$, $\eta_{\rm P}$, and $\eta_{\rm E}$ dramatically increase to 6.4 cd A⁻¹, 5.3 lm W⁻¹, and 3.11%, respectively, much higher than those of device without the NPB layer (3.8 cd A⁻¹, 3.3 lm W⁻¹, and 2.01%). More importantly, the $\eta_{\rm E}$ of TPyC-based device is considerably higher than that of our previously reported triphenylamine TPyPA-based device ($\eta_{\rm E} = 2.83\%$) with the same device configuration. One possible explanation for this difference is that the $\Phi_{\rm PL}$ of TPyC (95%) is much higher than that of TPyPA (80%). It should be noted that as $\Phi_{\rm PL}$ of both TPyC and TPyPA materials are very high in solution, various solid-state quenching effects could be much more important than the yields in solution. The exact solid-state quenching effects in both



Fig. 6. (a) Current efficiency (η_C), external quantum efficiency (η_E) and (b) power efficiency (η_P) as a function of current density. Device configurations: ITO/TPyC (100 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm); ITO/ NPB (70 nm)/TPyC (30 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm).

Table 2

Electroluminescence performance of BPyC and TPyC-based devices. Device configurations: ITO/BPyC (100 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm); ITO/NPB (70 nm)/BPyC (30 nm)/LiF (0.5 nm)/Al (100 nm); ITO/TPyC (100 nm)/TPBI (20 nm)/LiF (0.5 nm)/Al (100 nm); ITO/NPB (70 nm)/TPBI (70 nm)/LiF (70 nm)

Device	$V_{\text{turn-on}}(V)$	V at 20 mA cm ⁻² (V)	$\eta_{\rm C} ({\rm cd} {\rm A}^{-1})$	$\eta_{ m P}({ m Im}{ m W}^{-1})$	$\eta_{\rm E}$ (%)	CIE(x, y)
BPyC NPB/BPyC TPyC NDB/TD:/C	2.7 2.7 2.9	3.65 3.48 4.58	4.25 2.94 3.76	4.45 2.72 3.34	2.49 2.21 2.01	0.17, 0.25 0.15, 0.18 0.21, 0.26
NPB/ IPyC	2.8	4.34	6.42	5.34	3.11	0.22, 0.29

thin-films are now under investigation. Nevertheless, the discrepancy of $\Phi_{\rm PL}$ on the device performance is in consistent with our previous study [8] and underscores the importance of high $\Phi_{\rm PL}$ in organic materials for achieving highly efficient exciplex-based OLEDs. Characteristics of all BPyC- and TPyC-based devices are summarized in Table 2.

It is interesting to note that, in our previous study, the use of a better ETL (i.e., BPhen) would effectively increase the intensity of exciplex emission and, in turns, results in a better balanced white emission. In particular, the CIE coordinates change from (0.18, 0.25) for devices with TPBI as ETL to (0.31, 0.35) for the BPhen-based TPyPA device. In addition, the replacement of BPhen results in a high $\eta_{\rm E}$ of 3.93%, much higher than that of the TPBI-based device (2.83%). These enhancements have been attributed to the different carrier mobility of BPhen and TPBI. Indeed, the intensity of exciplex emission is determined by the charge accumulation densities, which in turns depends on the relative hole mobility and electron mobility in HTL and ETL. In particular, the use of ETL with higher electron mobility would lead to higher exciplex emission intensity. As the electron mobility in TPBI is two orders of magnitude lower than that in BPhen, the relatively small electron mobility in TPBI would inevitably reduce the number of electron-hole pairs formed for exciplex emission, and in turns leads to a weak exciplex emission. However, it is not the case in our present study, in which there is only a mild shift on the CIE coordinates from (0.22, 0.29) to (0.25, 0.33) and a small increase on $\eta_{\rm E}$ from 3.11% to 3.16% when the ETL is replaced by BPhen. Such minor device performance improvement for the TPyC-based devices may be ascribed by the lower mobility in TPvC than that in TPvPA. Particularly, it is expected that the hole mobility of TPyC is much lower than that of TPyPA, and thus the use of better electron-transporting materials, e.g., BPhen, would not result in a dramatic increase on the interfacial charge accumulation density at the HTL/ETL interface. It is also the reason for the larger driving voltage for the TPyC-based devices. For instance, to generate a current density of 20 mA cm⁻², the driving voltages of the TPyC-based device with TPBI or BPhen as ETL are 4.34 and 3.76 V, respectively, while those of the TPyPA-based device with TPBI or BPhen as ETL are 3.60 and 3.31 V, respectively [8].

4. Conclusions

We have designed and synthesized two carbazole-pyrene based fluorophores, namely BPyC and TPyC, to investigate how chemical structures of organic materials affecting exciplex emission. By employing the carbazole derivatives as emitters in contact with the electron-transporting TPBI, both devices exhibit distinct EL properties. Particularly, the TPyC-based devices display two emission peaks with CIE coordinates of (0.22, 0.29); while the BPyC-based devices show a single blue emission peak with CIE coordinates of (0.15, 0.18). More importantly, a relatively high η_E of 3.11% can be obtained for the TPyC-based device. This discrepancy on the device performance is attributed to the substitution of *t*-butyl unit of BPyC with a pyrenyl group, resulting in an increase on the degree of electron-donating property and hence the presence of exciplex emission at the HTL/ETL heterojunction.

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