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Novel carbazole/pyridine-based host material for solution-processed blue phosphorescent organic light-emitting devices

Wei Jiang, Lian Duan, Juan Qiao, Guifang Dong, Deqiang Zhang, Liduo Wang, Yong Qiu*

Key Lab of Organic Optoelectronics and Molecular Engineering of Ministry of Education, Department of Chemistry, Tsinghua University, Beijing 100084, PR China

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ABSTRACT

3,6-Bis(3,5-di(pyridin-3-yl)phenyl)-9-phenyl-9H-carbazole, a novel host material for solution-processed blue phosphorescent organic light-emitting devices was synthesized by a Suzuki coupling reaction. The optical, electrochemical and thermal properties of this novel crabazole have been characterized. The compound exhibits a high glass-transition temperature (Tg = 161 °C) and high triplet energy (E_T = 2.76 eV). Additionally, atomic force microscopy measurements indicate that high-quality amorphous films of this novel compound can be prepared by spin-coating. Solution-processed blue phosphorescent organic light-emitting devices were obtained using the carbazole as the host material for the phosphorescence emitter iridium(III) bis(4,6-difluorophenylpyridinato)- picolinate and their electroluminescence properties were evaluated.

1. Introduction

Organic lighting-emitting diodes (OLEDs) have attracted a great deal of attention due to their potential applications in full-color flat-panel displays and lighting sources [1–3]. Recently, phosphorescent OLEDs have attract great interest because they can harvest both singlet and triplet excitons for emission, theoretically yielding 100% internal quantum efficiency [4,5]. In phosphorescent OLEDs, to reduce the quenching associated with the relatively long excited state lifetimes of triplet emitters and triplet–triplet annihilation, triplet emitters are normally used as emitting guests in a host material. The fabrication of blue phosphorescent devices is still challenging because the extent of conjugation and molecular size of the host materials must be constrained to achieve a high triplet energy. This requirement makes it difficult for the molecules to acquire the morphological stability. Several carbazole-based host materials are frequently used in phosphorescence devices [6–12]. For example, 4,4′-bis(9-carbazolyl)-2,2′-biphenyl (CBP) is commonly used as a host material, but not for blue triplet emitters because of the lower triplet energy gap (E_T) of 2.53 eV relative to those of blue triplet emitters such as iridium(III) bis(4,6-difluorophenylpyridinato)-picolinate (FIrpic) (E_T = 2.62 eV) [13]. One of the earliest hosts used for blue electrophosphorescence devices is 1,3-bis(9-carbazolyl)benzene (mCP) (E_T = 2.90 eV) [9]. However, the low glass-transition temperatures (Tg) of CBP and mCP leads to ready crystallization, especially when the dopant concentration is low [14].

Concerning the fabrication of devices and displays, vapor deposition requires vacuum processing, which is a relatively costly process. In order to simplify the fabrication process of OLEDs and reduce the cost of larger-area displays, much research effort have been devoted to develop OLEDs with the emitting layer prepared by solution-processing techniques [15–20]. A good host material for a solution-processed blue phosphorescent OLED should have the following features: high triplet energy, good charge carrier transport properties, high solubility and film-forming ability. In our previous paper, we have demonstrated solution-processed blue phosphorescence OLEDs using a carbazole unit as the host with a high-efficiency [11,18,21]. In this paper, we synthesized a novel carbazole derivative 3,6-bis(3,5-di(pyridin-3-yl)phenyl)-9-phenyl-9H-carbazole (BDPPC), using of a carbazole monomer as the rigid core with two pyridine containing groups linked to the core through the 3, 6 positions. Additionally, pyridine units are introduced into the molecular structure to ensure good solubility to form high-quality films. As a result, the newly synthesized

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* Corresponding author. Key Lab of Organic Optoelectronics and Molecular Engineering of Ministry of Education, Department of Chemistry, Sipailou 2#, Tsinghua University, Beijing 100084, PR China. Tel.: +86 10 62779988; fax: +86 10 62795137.

E-mail address: qiuy@mail.tsinghua.edu.cn (Y. Qiu).

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compounds possess the following important characteristics: high triplet energy levels (2.76 eV) because of the nonconjugated linkage; the capability of forming stable amorphous thin films as a result of the highly twisted configuration of the molecules and good solubility in most common solvents. The application of this new compound as a host material for solution-processed blue phosphorescence devices has been demonstrated.

2. Experimental

2.1. General information

All reactants and solvents, unless otherwise stated, were purchased from commercial sources and used as received. ^{1}H NMR and ^{13}C HMR spectra were measured on a Bruker ARX600 NMR spectrometer with tetramethylsilane as the internal standard. Elemental analysis was performed on an Elementar Vario EL CHN elemental analyzer. Mass spectrometry was performed with a Thermo Electron Corporation Finnigan LTQ mass spectrometer. Absorption spectra were recorded with a UV–vis spectrophotometer (Agilent 8453) and PL spectra were recorded with a fluorospectrophotometer (Jobin Yvon, FluoroMax-3). TGA was recorded with a Netzsch simultaneous thermal analyzer (STA) system (STA 409PC) under a dry nitrogen gas flow at a heating rate of 10 °C min^{-1}. Glass-transition temperatures were recorded by DSC at a heating rate of 10 °C min^{-1} with a thermal analysis instrument (DSC 2910 modulated calorimeter). Cyclic voltammetry was performed on a Princeton Applied Research potentiostat/galvanostat model 283 voltammetric analyzer in CH_2Cl_2 solutions (10^{-3} M) at a scan rate of 100 mV s^{-1} with a platinum plate as the working electrode, a silver wire as the pseudo-reference electrode, and a platinum wire as the counter electrode. The supporting electrolyte was tetrabutylammonium hexafluorophosphate (0.1 M) and ferrocene was selected as the internal standard. The solutions were bubbled with argon for 10 min before measurements. The film surface morphology was measured with AFM (Seiko Instruments, SPA-400).

2.2. Device fabrication and performance measurements

In a general procedure, indium–tin oxide (ITO)-coated glass substrates were pre-cleaned carefully and treated by UV ozone for 4 min. A 40 nm PEDOT:PSS aqueous solution was spin-coated onto the ITO substrate and baked at 200 °C for 10 min. The substrates were then taken into a nitrogen glove box, where FIrpic-doped host layer was spin-coated onto the PEDOT:PSS layer from 1,2-dichloroethane solution and annealed at 80 °C for 30 min. The substrate was then transferred into an evaporation chamber, where the TPBI was evaporated at an evaporation rate of 1–2 Å/s under a pressure of 4×10^{-4} Pa and the Cs_2CO_3/Al bilayer cathode was evaporated at evaporation rates of 0.2 and 10 Å/s for Cs_2CO_3 and Al, respectively, under a pressure of 1×10^{-3} Pa. The current–voltage–brightness characteristics of the devices were characterized with Keithley 4200 semiconductor characterization system. The electroluminescent spectra were collected with a Photo Research PR705 Spectrophotometer. All measurements of the devices were carried out in ambient atmosphere without further encapsulations.

2.3. Materials

2.3.1. Synthesis of 3,6-bis(3,5-dichlorophenyl)-9-phenyl-9H-carbazole

A mixture of 3,6-dibromo-9-phenylcarbazole (1.5 mmol), 3,5-dichlorophenylboronic acid (3.2 mmol), and $Pd(PPh_3)_4$ (0.06 mmol) were added to 1,4-dioxane solution (50 mL) then, 2 M K_2CO_3 solution (5 mL), which was dissolved in H_2O, was added to the reaction mixture. The mixture was heated under reflux for 24 h. After the reaction finished, the reaction mixture was extracted with dichloromethane and water. The organic layer was dried by anhydrous $MgSO_4$ and filtered. The product was isolated by silica gel column chromatography using petroleum ether/ethyl acetate (10:1) eluent to afford a white solid (5.20 g, 65.0%). ^{1}H NMR (600 MHz, $CDCl_3$, TMS) δ: 8.36 (*d*, $J = 1.4$ Hz, 2H), 7.67 (*t*, $J = 8.2$, 7.6 Hz, 2H), 7.62–7.58 (*m*, 8H), 7.53 (*t*, $J = 6.9$, 7.6 Hz, 1H), 7.48 (*d*, $J = 8.2$ Hz, 2H), 7.34 (*s*, 2H); ^{13}C-NMR($CDCl_3$, δ): 144.79, 141.51, 135.37, 130.22, 128.10, 127.07, 126.59, 125.75, 125.68, 123.95, 119.14, 110.68; MS (MALDI–TOF) [*m*/*z*]: calcd for $C_{30}H_{17}Cl_4N$, 533.3; found, 533.5. Anal. Calcd. for $C_{30}H_{17}Cl_4N$: C, 67.57; H, 3.21; N 2.62. Found: C, 67.66; H, 3.32; N 2.55.

2.3.2. Synthesis of 3,6-bis(3,5-di(pyridin-3-yl)phenyl)-9-phenyl-9H-carbazole (BDPPC)

A mixture of 3,6-bis(3,5-dichlorophenyl)-9-phenyl-9H-carbazole (0.5 mmol), 3-pyridineboronic acid (2.2 mmol), $Pd_2(dba)_3$ (0.05 mmol), PCy_3 (0.05 mmol) and K_3PO_4 (2.2 mmol) were added to 1,4-dioxane solution (50 mL). The mixture was refluxed under nitrogen for 24 h. After the reaction finished, the reaction mixture was extracted with dichloromethane and water. The organic layer was dried by anhydrous $MgSO_4$ and filtered. The product was isolated by silica gel column chromatography using CH_2Cl_2/CH_3OH (10:1) eluent to afford a white solid (0.12 g, 31.0%). ^{1}H NMR (600 MHz, $CDCl_3$, TMS) δ: 9.01 (*d*, $J = 2.0$ Hz, 6H), 8.67 (*d*, $J = 4.1$ Hz, 6H), 8.53 (*s*, 3H), 8.05 (*d*, $J = 7.6$ Hz, 6H), 7.97 (*s*, 6H), 7.80 (*d*, $J = 9.6$ Hz, 3H), 7.74 (*s*, 3H), 7.70–7.64 (*m*, 6H), 7.57–7.53 (*m*, 5H), 7.46–7.44 (*m*, 6H); ^{13}C-NMR($CDCl_3$, δ): 148.97, 148.53, 143.80, 141.36, 139.53, 136.59, 134.79, 132.88, 130.21, 128.02, 127.13, 126.12, 125.98, 124.51, 124.13, 123.81, 119.25, 110.68; MS (MALDI–TOF) [*m*/*z*]: calcd for $C_{50}H_{33}N_5$, 703.8; found, 703.6. Anal. Calcd. for $C_{50}H_{33}N_5$: C, 85.32; H, 4.73; N 9.95. Found: C, 85.30; H, 4.68; N 9.90.

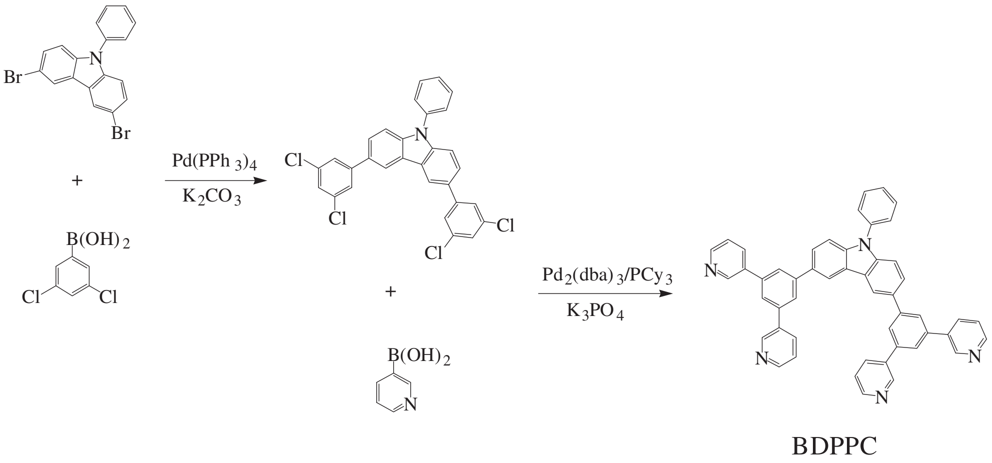
3. Results and discussion

3.1. Synthesis and characterization

Scheme 1 illustrates the synthetic procedures for BDPPC. Initially, the starting 3,6-dibromo-9-phenylcarbazole was synthesized according to methods previously described [18]. 3,6-bis(3,5-dichlorophenyl)-9-phenyl-9H-carbazole was prepared from 3,6-dibromo-9-phenylcarbazole and 3,5-dichlorophenylboronic acid catalyzed by $Pd(PPh_3)_4$ in 1,4-dioxane. Then the Suzuki cross-coupling reactions of 3,6-bis(3,5-dichlorophenyl)-9-phenyl-9H-carbazole with the 3-pyridineboronic acid led to BDPPC in the presence of $Pd_2(dba)_3/PCy_3$ (yield: 31.0%). The target compound was purified by silica column chromatography and recrystallization. ^{1}H NMR, ^{13}C-NMR, mass spectrometry, and elemental analysis were employed to confirm the chemical structures of above-mentioned compounds as described in the experimental section.

3.2. Thermal analysis

Thermal properties of BDPPC were investigated by thermal gravimetric analyses and differential scanning calorimetry. The introduction of pyridine containing groups to 3- and 6- positions of carbazole renders the molecule non-planar. Such a molecular configuration is strongly beneficial to the thermal stability, as indicated by the high decomposition temperature (Td, corresponding to 5% weight loss) of 510 °C and very high *T*g of 161 °C



Scheme 1. Synthetic routes toward BDPPC.

(Fig. 1), which are much higher than those of analogous carbazole-based host materials such as CBP (62 °C) and mCP (60 °C). The morphologies of BDPPC were characterized by atomic force microscopy (AFM) using standard tapping mode. As can be seen from Fig. 2, the surface of neat BDPPC film is pinhole-free and quite smooth, and the root-mean-square (RMS) value is 0.32 nm. The surface morphology of the spin-coated films was found to be stable after thermal treatment at 100 °C for 6 h. These results demonstrate that BDPPC is capable of forming amorphous films through solution-processing.

3.3. Photophysical properties

Fig. 3(a) depicts the UV–vis absorption and photoluminescent (PL) spectra of BDPPC in CH_2Cl_2. The absorption peak at around 295 nm could be assigned to the n–π^* transitions of the carbazole moiety, while the longer-wavelength absorption at around 325 nm can be attributed to π–π^* transitions from the carbazole moiety to the pyridine moiety [22]. The PL emission peak at 392 nm for BDPPC was significantly overlapped with the metal-to-ligand charge-transfer absorption peak of FIrpic, indicating that the Förster energy transfer from the host materials to the FIrpic would be efficient. Fig. 3(a) also depicts the phosphorescence spectrum of BDPPC measured from CH_2Cl_2 at 77 K. The triplet energy of BDPPC, measured from the low-temperature photoluminescence spectra, was 2.76 eV, higher than reported for the common triplet blue-emitter FIrpic. Using host materials that possess high triplet energy is a provision for effective confinement of the triplet excitons on the guest and, consequently, for prevention of back energy transfer between the host and dopant molecules. To further confirm their exciton confinement property, the transient photoluminescence decay of 5wt% FIrpic doped into BDPPC and CBP was measured (Fig. 3(b)). The CBP: FIrpic film exhibits an extremely long decay component, which can attributed to the exothermic back energy transfer from FIrpic to CBP [23], while the BDPPC: FIrpic film clearly exhibits a mono-exponential decay curve and a relative lifetime of 1.3 μs, indicating that the triplet energy transfer from FIrpic to BDPPC was completely suppressed [24].

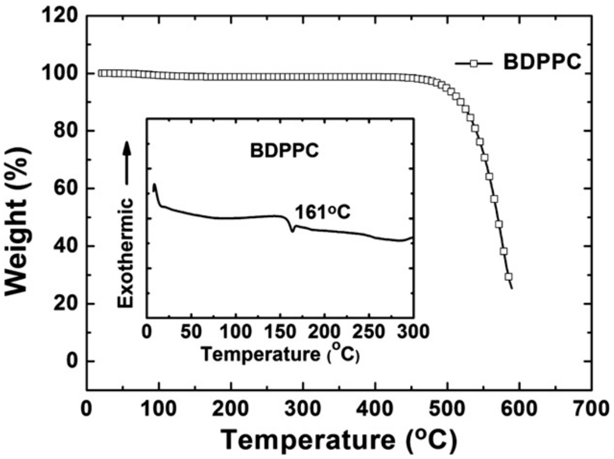


Fig. 1. TGA traces of BDPPC recorded at a heating rate of 10 °C min^{-1}. Inset: DSC measurement recorded at a heating rate of 10 °C min^{-1}.

3.4. Electrochemical analysis

The electrochemical properties of BDPPC were studied in solution using cyclic voltammetry (CV) using tetrabutylammonium hexafluorophosphate($TBAPF_6$) as the supporting electrolyte and ferrocene as the internal standard. As shown in Fig. 4, during the anodic scan in dichloromethane, an oxidation peak is observed at 1.54 V, which was ascribed to the oxidation of the carbazole unit of BDPPC. No reduction waves were detected. On the basis of the onset potentials for oxidation, we estimated the HOMO energy of BDPPC to be −5.55 eV, with regard to ferrocene (−4.8 eV below vacuum) [25]. The highest occupied molecular orbital (HOMO) energy level of BDPPC is approaching the work function of 4-ethylenedioxythiophene):poly(styrene-4-sulfonate) (PEDOT:PSS, −5.2 eV), thereby facilitating the injection of holes to the emitting layer [20]. Through subtraction of the optical energy gap from the HOMO energy level, we calculated the energy level of the lowest unoccupied molecular orbital (LUMO) to be −2.02 eV. To gain more insight into the electronic structure of BDPPC, density functional theory (DFT) calculations were performed at the B3LYP/6-31G(d) level for the geometry optimization. As shown in Fig. 4, the HOMO level of BDPPC is mainly located at the electron-donating carbazole fragments, whereas the LUMO level has a contribution from the electron-accepting pyridine units. The calculated values of HOMO and LUMO energy levels are in agreement with the electrochemical experimental results.

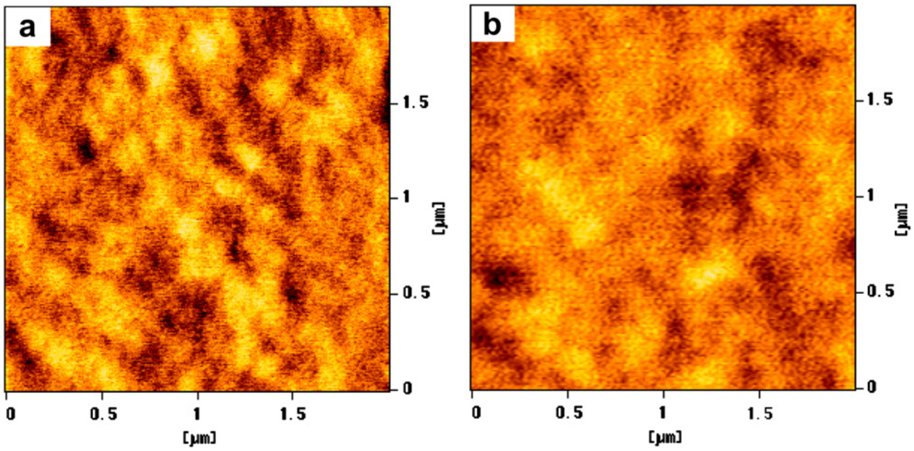


Fig. 2. AFM topographic images of (a) neat BDPPC and (b) neat BDPPC film thermal treated at 100 °C for 6 h.

3.5. Electroluminescent properties

To assess the utility of this solution-processable compound as a host material in OLEDs, we fabricated single-layer blue electrophosphorescent devices with BDPPC as the host and 10wt% FIrpic as the dopant. The single-layer device with the configuration ITO/PEDOT:PSS/Host:FIrpic(10wt%)/Cs_2CO_3/Al has been fabricated by spin-coating. Fig. 5(a) presents the current density–voltage–luminance (*I*–*V*–*L*) curve of BDPPC-based device, which was turned on at 4.5 V (corresponding to 1 cd m^{-2}) and had a maximum brightness of 1100 cd m^{-2}. As revealed in Fig. 5(b), the maximum luminance efficiencies of this single-layer blue-emitting device were 0.2 cd A^{-1}, and the corresponding external quantum efficiencies and power efficiencies were 0.11% and 0.14 lm W^{-1}. Generally, for single-layer devices, severe exciton quenching exists at the cathode interface, resulting in degraded device performance [26]. To improve the performance of the device, a thin TPBI electron-transporting and exciton-confining layer was inserted between the light-emitting layer and the cathode. A device with configuration of ITO/PEDOT:PSS/Host:FIrpic(10wt%)/TPBI(30 nm)/Cs_2CO_3/Al has been fabricated. The brightness of the devices with a TPBI layer was significantly increased to 14,400 cd m^{-2}, compared with that of single-layer device. After the insertion of the TPBI layer, the maximum efficiency of the BDPPC-based device was largely improved to 7.1 cd A^{-1}, and the corresponding external quantum efficiencies and power efficiencies were 3.6% and 5.2 lm W^{-1}. As shown in Fig. 5(c), the electroluminescence (EL) spectra of BDPPC-based devices are almost identical with the CIE coordinates of (0.16, 0.37) and (0.15, 0.32) for single-layer and double-layer devices, respectively, corresponding to the emission of FIrpic. Furthermore, no additional emission coming from BDPPC was observed, indicative of efficient energy transfer from the host to FIrpic.

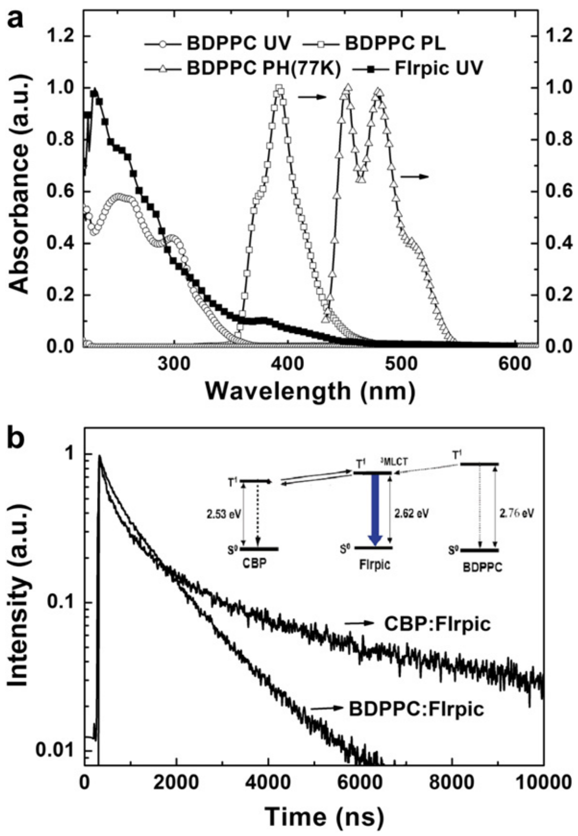


Fig. 3. (a) Normalized absorption, emission and phosphorescence (77 K) spectra of BDPPC and FIrpic in CH_2Cl_2; (b) Transient photoluminescence decay of BDPPC: 5wt% Firpic and CBP: 5wt% FIrpic.

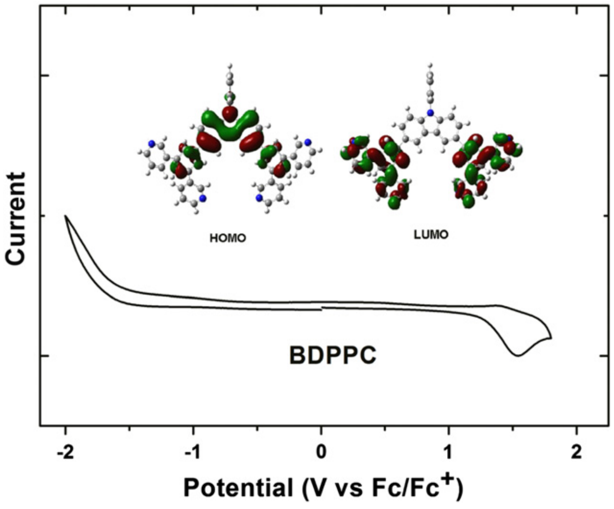


Fig. 4. The CV curves of BDPPC in CH_2Cl_2 solutions (10^{-3} M). Inset: optimized geometries and calculated HOMO and LUMO density maps for BDPPC.

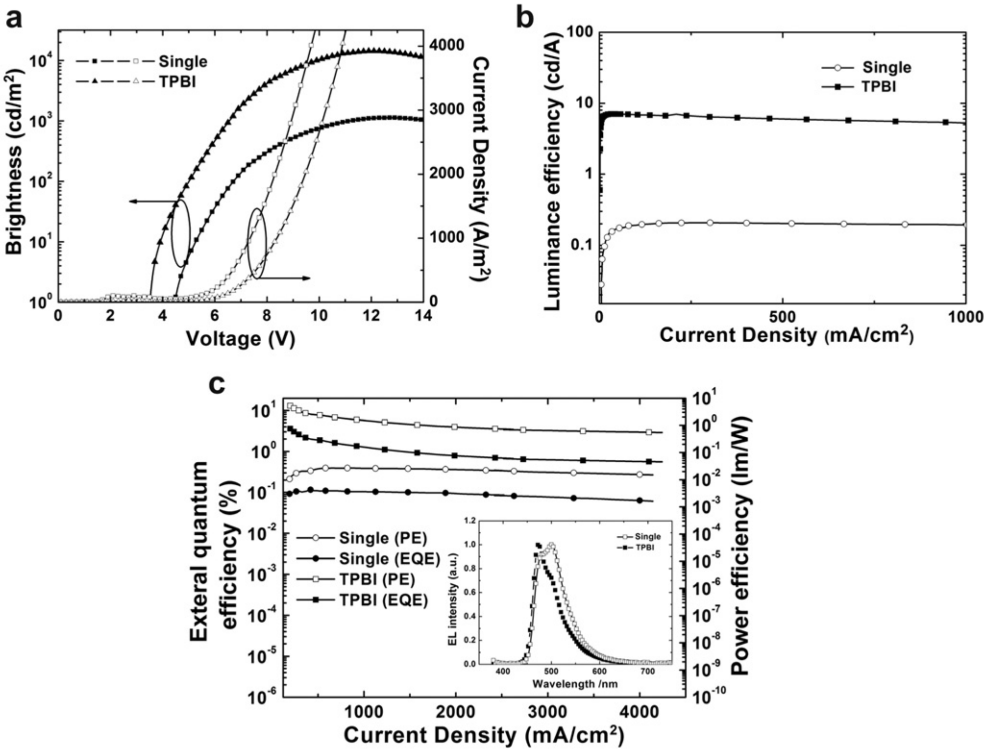


Fig. 5. Comparison of device characteristics (a) luminance–voltage–current density, (b) Luminance efficiency–current density and (c) External quantum efficiency–current density–power efficiency, inset: EL spectrum.

4. Conclusions

In summary, we have designed and synthesized a novel host material BDPPC for solution-processed blue phosphorescent organic light-emitting devices that comprises of two pyridine fragments linked to the C3 and C6 positions of the carbazole unit. The non-coplanar conformation of BDPPC provides steric bulk, resulting in a stable amorphous film, and a very high *T*g. Utilizing this new compound as host material, the solution-processed blue phosphorescence OLEDs with FIrpic as a dopant show a maximum current efficiency of 7.1 cd A^{-1}. In order to obtain high-efficiency solution-processed blue phosphorescence OLEDs, optimization of the BDPPC-based devices require further study. Our results pave the way toward reducing the complexity and cost of fabricating efficient OLEDs for use in displays and lighting applications.

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