



A novel, bipolar host based on triazine for efficient solution-processed single-layer green phosphorescent organic light-emitting diodes



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ARTICLE INFO

Article history:

Received 1 July 2013

Received in revised form

16 August 2013

Accepted 11 September 2013

Available online 25 September 2013

Keywords:

OLEDs

Solution-processed

Electrophosphorescence

Triazine

Bipolar

Host material

ABSTRACT

A novel N-phenyl carbazole substituted 2, 4,6-trisphenyl-triazine host material (TPCPZ) for solution processed green phosphorescent organic light-emitting devices (PhOLEDs) was synthesized by a Suzuki-cross coupling reaction. The optical, electrochemical and thermal properties of TCPZ have been characterized. TCPZ exhibits a high glass transition temperature of 165 °C and a triplet energy of 2.63 eV. The appropriate HOMO energy level (−5.39 eV) and LUMO energy level (−2.16 eV) matching with the HOMO energy level of PEDOT:PSS (−5.35 eV) and the LUMO energy level of Cs₂CO₃/Al bilayer cathode (−2.2 eV), facilitate the transfer of holes and electrons. The solution-processed single-layer device using TCPZ as the host for fac-tris(2-(4-phenylpyridine)iridium (Ir(ppy)₃) exhibited a low turn-on voltage of 3.5 V, a maximum current efficiency of 20.8 cd A^{−1} and a maximum luminance of 18,000 cd m^{−2}. These results demonstrated that TCPZ as a host material is advantageous for fabrication of highly efficient single-layer green PhOLEDs.

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1. Introduction

Since efficient phosphorescent emitters for organic light-emitting diodes (PhOLEDs) was discovered by Forrest's group, tremendous efforts have been made in the development of highly efficient devices [1–5]. In PhOLEDs, the emitting layer consists of a phosphorescent dye which is doped into a host material to avoid concentration quenching and to optimize the charge balance. High efficiencies are obtained only when the energy is efficiently transferred from the host to the phosphorescent dye. To achieve efficient electrophosphorescence, the choice of host is of vital importance [6]. Generally, the triplet energy of the host material is higher than that of the guest. For efficient OLEDs, well balanced charge carrier transport and a broad recombined zone are desirable. In general, the electron mobility of many host materials is much lower than the hole mobility because of the fact that they consist electron donors such as aromatic amines, carbazoles. This task is mostly solved by a design of a bipolar material [7,8]. Introduction of strong electron donors such as aromatic amines or carbazoles [9] to electron accepting N-heterocycles like pyridine [10–

12], oxadiazole [13–16], phenanthroline [17–19], benzimidazole [20–24], is well described. Triazines, are known to be good electron conductors and their derivatives have been used as electron transport layers in OLEDs [4,25–28]. Recently, some bipolar host materials based on triazines for PhOLEDs are reported [29–34]. A series of donor substituted 1,3,5-triazine derivatives as host for blue or green PhOLEDs show good performance.

In recent years, many groups are devoted to improve the performance of solution-processed PhOLEDs which are highly desirable to simplify the fabrication process and reduce the cost of larger area displays [35–44]. Even though polymer light-emitting diodes can be easily fabricated by solution casting, they are generally difficult to synthesize and purify for electronic devices. Therefore, it is a good strategy to develop OLEDs based on solution-processed small molecules. However, the largest challenge for small molecules lies in the poor solubility accompanied by the tendency to crystallize and failure to form high quality films. Consequently, it is of significant importance to design and synthesize amorphous small molecules with high purity and solubility. To date, few works reported on bipolar host based on 1, 3,5-triazine for solution-processed PhOLEDs.

In this work, a bipolar host composed of electron-transporting 2, 4,6-trisphenyl-triazine and hole-transporting N-phenyl carbazole,

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namely 2, 4,6-tris (3-((9-phenyl) carbazol-3-yl)-phenyl) -triazine(TPCPZ) is designed and synthesized. The dipolar nature of TPCPZ can promote the strong π - π intermolecular stacking of molecules in the solid state, which can facilitate high charge transport. Moreover, the electron-donating N-phenyl carbazole moieties are conjugated to the electron accepting 2, 4,6-trisphenyl-triazine core; the donor-acceptor intramolecular interaction tends to decrease the energy gap. TPCPZ has a triplet energy of 2.62 eV, which make it suitable as host material for green phosphorescent emitters. The material exhibits high thermal stability and good sublimation properties. The glass transition temperature of 165 °C ensures morphological stability of the host-guest emission layer during the operation of the device. Additionally, TPCPZ has good solubility in common solvents such as CH_2Cl_2 , CHCl_3 , 1,2-dichloroethane et al., which make it suitable for the fabrication of solution-processed PhOLEDs. The single-layer device with TPCPZ as host has low turn-on voltage of 3.5 V, a high maximum luminance efficiency of 20.8 cd A^{-1} and maximum luminance of 18,000 cd m^{-2} in a solution-processed green phosphorescent OLED using an emitter of fac-tris(2-(4-phenylpyridine)iridium ($\text{Ir}(\text{ppy})_3$)).

2. Experimental

2.1. General

All reactants and solvents were purchased from commercial sources and used without further purification. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker ARX300 NMR spectrometer with $\text{Si}(\text{CH}_3)_4$ as the internal standard. Elemental analysis was performed on an Elementar Vario EL CHN elemental analyzer. Mass spectra were obtained using a Thermo Electron Corporation Finnigan LTQ mass spectrometer. UV-vis absorption spectra were recorded with a spectrophotometer (Agilent 8453) and PL spectra were recorded with a fluorospectrophotometer (Jobin Yvon, FluoroMax-3). Thermogravimetric analysis (TGA) was performed using a Netzsch simultaneous thermal analyzer (STA) system (STA 409PC) under dry nitrogen atmosphere at a heating rate of 10 °C min^{-1} . Glass transition temperature was recorded by differential scanning calorimetry (DSC) at a heating rate of 10 °C min^{-1} with a thermal analysis instrument (DSC 2910 modulated calorimeter). Cyclic voltammetry measurements were 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 a constant argon flow for 10 min before measurements.

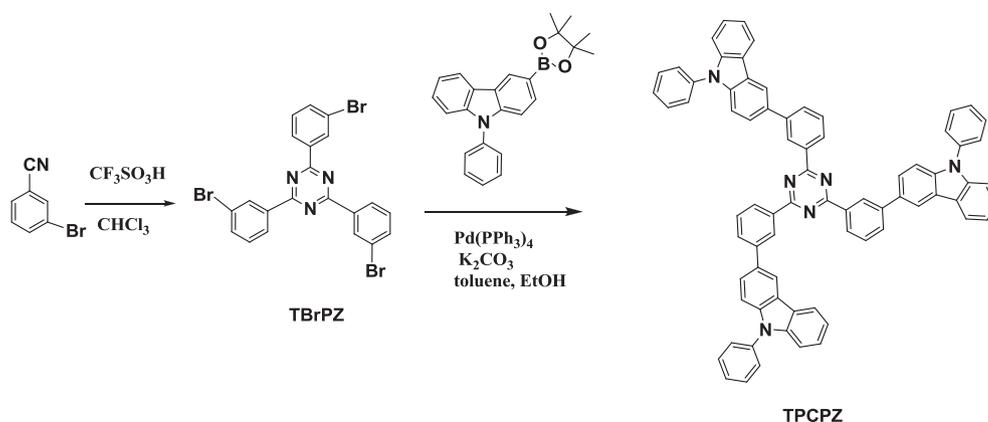
DFT calculations of TPCPZ were performed using the Gaussian 03 program package. The calculation was optimized at the B3LYP/6-31G(d) level of theory. The molecular orbitals were visualized using Gaussview [45].

2.2. Synthesis of 2,4,6-tris(3-((9-phenyl)carbazol-3-yl)-phenyl)-triazine(TPCPZ)

To a solution of 2,4,6-tris(3-bromophenyl)-triazine (TBrPZ) (0.546 g, 1.0 mmol) and N-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolane-2-yl)-6H-carbazole (1.107 g, 3.0 mmol) in 20 mL of toluene and 4 mL of ethanol was added 2 mL of 2.0 M aqueous K_2CO_3 solution. The reaction mixture was then purged with nitrogen for 10 min before adding tetrakis(triphenylphosphine) palladium(0) (0.055 g, 0.048 mmol). After refluxing for 24 h under nitrogen, the resulting mixture was cooled to room temperature and then poured into water and extracted with 60 mL (3×20 mL) CH_2Cl_2 . The combined organic phase was then washed with 20 mL (2×10 mL) saturated aqueous NaCl solution and dried with anhydrous Na_2SO_4 . After removal of the solvent by rotary evaporation, the residue was purified by silica gel column chromatography to afford TPCPZ as a white solid. Yield: 0.60 g (58.08%). ^1H NMR (300 MHz, CDCl_3): δ (ppm) 9.17 (s, 3H), 8.81–8.78 (d, 3H), 8.50 (s, 3H), 8.16–8.19 (d, 3H), 7.96–7.98(d, 3H), 7.80–7.83(m, 3H), 7.67–7.73 (t, 3H), 7.65–7.56 (m, 12H), 7.52–7.43 (m, 6H), 7.40–7.35 (m, 6H), 7.16–7.20 (m, 3H). ^{13}C NMR (75 MHz, CDCl_3) δ (ppm): 109.50, 109.78, 118.67, 119.71, 120.03, 123.06, 123.62, 125.18, 125.80, 126.71, 126.97, 127.14, 127.39, 128.78, 129.54, 131.16, 132.68, 136.51, 137.29, 140.18, 141.02, 142.18, 171.57. MS (MALDI-TOF) [m/z]: calcd for $\text{C}_{75}\text{H}_{48}\text{N}_6$, 1033.22; found, 1033.48. Anal. Calcd. for $\text{C}_{75}\text{H}_{48}\text{N}_6$ (%): C, 87.18; H, 4.68; N 8.13. Found: C, 87.34; H, 4.72; N 8.38.

2.3. Device fabrication and performance measurements

The solution-processed single-layer device using TPCPZ as host with a configuration ITO/PEDOT:PSS (40 nm)/TPCPZ: $\text{Ir}(\text{ppy})_3$ (90 wt%:10 wt%,100 nm)/ Cs_2CO_3 (2 nm)/Al (120 nm) has been fabricated by spin-coating. 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 Poly(3,4-ethylenedioxythiophene) doped with Poly(styrene-4-sulfonate)(PEDOT:PSS) aqueous solution was spin coated onto the ITO substrate and baked at 210 °C for 10 min. The substrates were then taken into a nitrogen glove box, where $\text{Ir}(\text{ppy})_3$ -doped TPCPZ layer was spin coated onto the



Scheme 1. Synthetic route of TPCPZ.

PEDOT:PSS layer from 1,2-dichloroethane solution and annealed at 120 °C for 30 min. The substrate was then transferred into an evaporation chamber, where the Cs₂CO₃/Al bilayer cathode was evaporated at evaporation rates of 0.2 and 10 Å/s for Cs₂CO₃ and Al, respectively, under a pressure of 1×10^{-3} Pa. The current-voltage-brightness characteristics of the device 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.

3. Results and discussion

3.1. Synthesis and characterization

Scheme 1 shows the synthetic routes and structure of the newly synthesized TPCPZ. 2,4,6-tris(3-bromophenyl)-triazine (TBrPZ) was synthesized according to the literature procedure⁴. N-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolane-2-yl)-6H-carbazole was synthesized according to the literature procedure with carbazole as start material [46]. Subsequently, Suzuki-cross coupling reaction of TBrPZ with N-phenyl-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolane-2-yl)-6H-carbazole led to TPCPZ with a yield of 58.08%. Finally, the product was purified by the silica column method and recrystallization from n-hexane/CH₂Cl₂, yielding the very pure white powders. ¹H NMR, ¹³C NMR, matrix-assisted laser desorption ionization time-of-flight (MALDI-TOF) mass spectrometry, and elemental analysis were employed to confirm the chemical structure of TPCPZ in the experimental section.

3.2. Thermal properties of the host materials

The thermal properties TPCPZ were investigated by thermal gravimetric analyses (TGA) and differential scanning calorimetry (DSC) under nitrogen atmosphere at a heating rate of 10 °C min⁻¹. As shown in Fig.1, TGA measurement reveals its high thermal decomposition temperature (Td), corresponding to 5% weight-loss of 559 °C. The DSC trace exhibits a clear glass transition temperature (Tg) of 165 °C during the second heating scans. The thermal analysis results clearly demonstrate the very high thermal stability of TPCPZ, which facilitates the forming of amorphous films through solution processing.

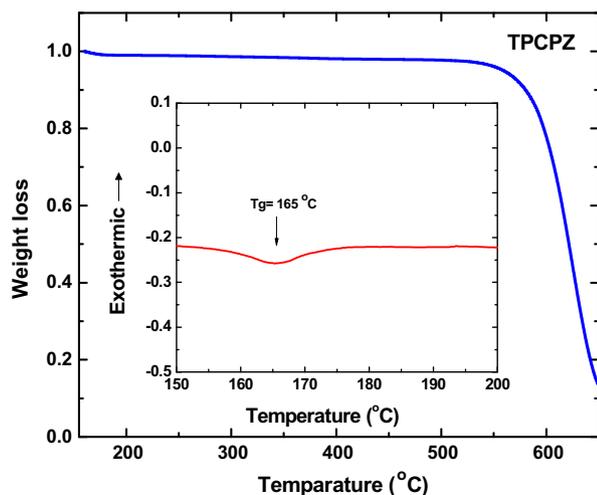


Fig. 1. TGA traces (blue curve) of TPCPZ recorded at a heating rate of 10 °C min⁻¹, DSC (red curve) measurement recorded at a heating rate of 10 °C min⁻¹.

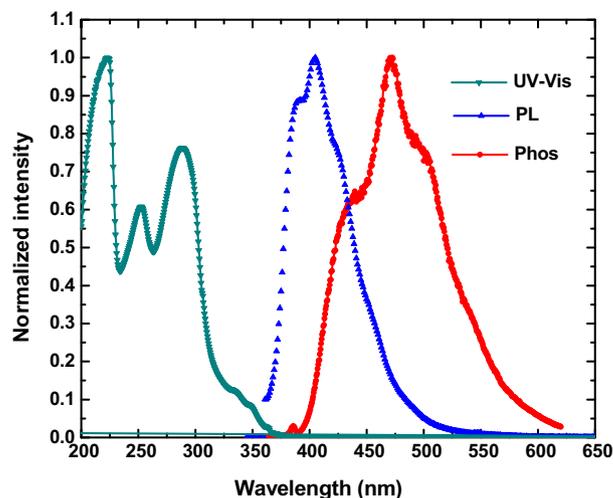


Fig. 2. Normalized UV-vis absorption (Abs, green curve), photoluminescence (PL, blue curve) and phosphorescence (77 K) (PH, red curve) spectra of TPCPZ.

3.3. Optical properties of the host materials

Fig. 2 depicts the UV-vis absorption and photoluminescence (PL) spectra of TPCPZ in hexane. Four major absorption peaks locate at 223, 252, 289, 335 nm in the absorption spectra, while the emission peaks appear at 405 nm in the PL spectra. The absorption peaks at 223, 252 nm can be attributed to the $\pi-\pi^*$ and $n-\pi^*$ transitions of the central 2,4,6-trisphenyl-triazine and outer N-phenyl. The absorption peaks at 289 nm, 335 nm can be attributed to the $\pi-\pi^*$ and $n-\pi^*$ transitions of the outer carbazole of TPCPZ. From the absorption edge of the UV-vis absorption, the optical bandgap (E_g) of TPCPZ can be estimated to be 3.23 eV. The phosphorescence spectra measured from a frozen 2-methyltetrahydrofuran matrix at 77 K are also shown in Fig. 2. The triplet energy of TPCPZ was determined to the value of 2.63 eV by the highest energy 0–0 phosphorescent emission, which is sufficiently high enough to serve as the appropriate host for Ir(ppy)₃.

3.4. Electrochemical analysis and theoretical calculations of the host material

The electrochemical properties TPCPZ were studied in solution through cyclic voltammetry (CV) using tetrabutylammonium

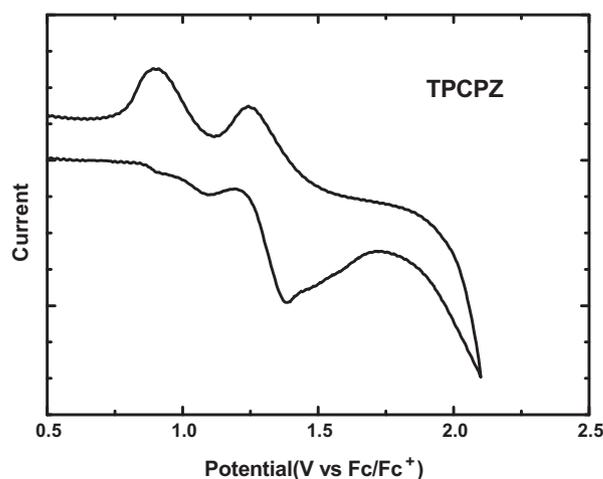


Fig. 3. Oxidation part of the CV curves of TPCPZ in CH₂Cl₂ solutions (10^{-3} M).

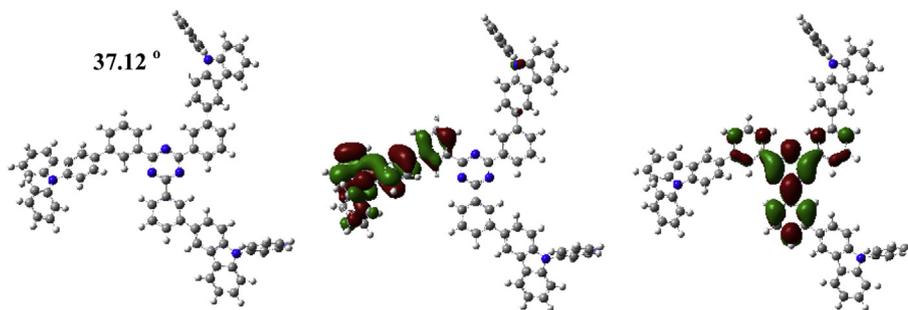


Fig. 4. Optimized geometries and calculated HOMO and LUMO density maps for TPCPZ according to DFT calculations at B3LYP/6-31* level.

hexafluorophosphate (TBAPF₆) as the supporting electrolyte and ferrocene as the internal standard. The highest occupied molecular orbital (HOMO) energy level of TPCPZ was characterized by the electrochemical cyclic voltammetry (CV) (Fig. 3). During the anodic scan in CH₂Cl₂, TPCPZ exhibited reversible oxidation process, which can be assigned to the oxidation of electron-donating carbazole moiety, with the onset potential of 1.18 eV. No reduction wave was detected. On the basis of the onset potential for oxidation, the HOMO energy level was estimated to be -5.39 eV, the HOMO energy level approach the work function of PEDOT (-5.2 eV), which allows in a low barrier of hole injection. The LUMO energy level of TPCPZ estimated from the HOMO energy level and E_g is -2.16 eV, which matches the LUMO energy level of Cs₂CO₃/Al bilayer cathode (-2.2 eV), facilitates electrons injection.

DFT calculations were performed to understand the physical properties of the TPCPZ at thermo-molecular level. As shown in Fig. 4, the outer layer N-phenyl carbazole units are significantly twisted

with inner 2, 4,6-trisphenyl-triazine unit, resulting in a non-planar structure in the molecule. These geometrical characteristics can effectively prevent intermolecular interactions between systems and thus suppress molecular recrystallization and limit the extent of conjugation between the central core and branches, which improves the morphological stability of thin film of TPCPZ. The LUMO level of TPCPZ is localized predominantly on the 2, 4,6-trisphenyl-triazine unit, while the HOMO level is distributed over the outer layer N-phenyl carbazole fragments.

3.5. Electroluminescent properties

The single-layer green electrophosphorescent device with TPCPZ as the host and Ir(ppy)₃ as the dopant with the configuration of ITO/PEDOT:PSS/TPCPZ:Ir(ppy)₃/Cs₂CO₃/Al has been fabricated by spin-coating. As shown in Fig. 5, the electroluminescence (EL) spectra of TPCPZ based device is identical with the CIE

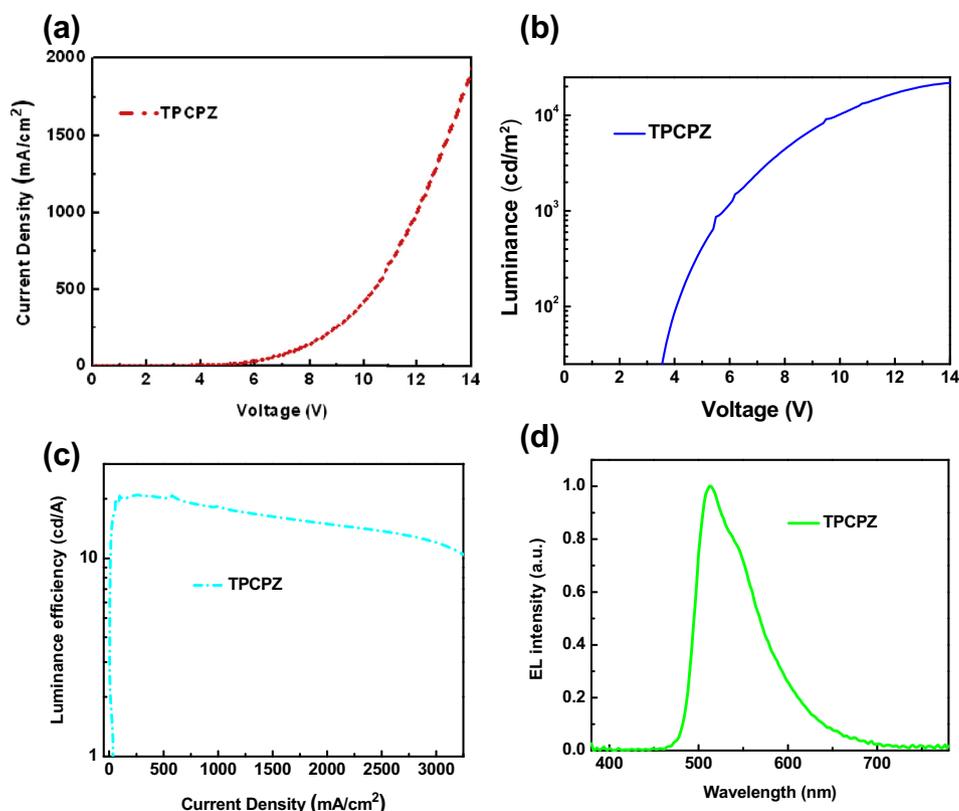


Fig. 5. (a) Current-voltage (b) Luminance-voltage characteristics (c) Luminance efficiency versus current density plots of the devices. (d) The normalized EL spectra of devices at a driving voltage of 10 V.

coordinates of (0.315, 0.605), corresponding to the emission of Ir(ppy)₃, and indicate the efficient energy transfer from the host to Ir(ppy)₃. The *J*–*V* characteristic curves of the device demonstrate the turn-on voltage is only 3.5 V. On one hand, the low turn-on voltage of TPCPZ based device is most likely due to their high-lying HOMO energy level and low-lying LUMO energy level, which matched with the HOMO energy level of PEDOT:PSS(–5.35 eV) and the LUMO energy level of Cs₂CO₃/Al bilayer cathode (–2.2 eV), thereby facilitated holes and electrons injection of the device. On the other hand, the bipolar nature of TPCPZ make it have good and balanced charge carrier transport properties for the hole–electron recombination process and confinement of the exciton formation zone in the emissive layer. As revealed in Fig. 5, the maximum LE value of this single-layer green-emitting device was 20.8 cd A^{–1} and a maximum luminance was 18,000 cd m^{–2}.

4. Conclusions

In summary, we have designed and synthesized a novel bipolar solution-processable triazine-based host material. TPCPZ exhibits high glass transition temperature and high thermal stability. Results from CV and computational calculations show that TPCPZ has high-lying HOMO energy level and low-lying LUMO energy level. The triplet energy of TPCPZ is 2.63 eV. Utilizing TPCPZ as host material, highly efficient solution-processed single-layer green PhOLED has been achieved with low turn-on voltage of 3.5 V, a maximum current efficiency of 20.8 cd A^{–1} and a maximum luminance of 18,000 cd m^{–2}.

Acknowledgments

The authors thank Dr. Zhao Yan for OLEDs fabrication and measurement. This work was supported by the National Natural Science Foundation of China (Grant No.51103023 and 21173042), the National Basic Research Program of China (Grant No.2013CB932900), the Research Fund for Graduate Innovation Project of Jiangsu Province (No. CXZZ13_0090) and the science and technology support program (industry) project of Jiangsu province (BE 2013118).

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