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PAPER

# An ambipolar poly(*meta*-phenylene) copolymer with high triplet energy to host blue and green electrophosphorescence

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An ambipolar polymer host for electrophosphorescence is developed by incorporating a hole transporting carbazole unit and electron transporting oxadiazole unit into a poly(*meta*-phenylene) backbone with a high triplet energy level.

#### Introduction

Polymer light-emitting diodes (PLEDs) promise new flat-panel display and solid-state lighting technologies fabricated by lowcost solution processing.<sup>1</sup> The materialization of this potential largely hinges on phosphorescent PLEDs (PPLEDs), in which phosphorescent dopants are dispersed in a polymer host and used as the emissive layer. PPLEDs can harvest both singlet and triplet excitons and achieve an internal electron-to-photon quantum efficiency of 100%.<sup>2-11</sup> Ideally, owing to solution processing in fabrication, PPLED devices have a single-layer configuration with the emissive layer sandwiched between the cathode and anode. During device operation, electrons and holes are injected from electrodes to the polymer host and generate singlet and triplet excitons. The excitons are transferred to the triplet states of the phosphorescent dopants and generate light. An ideal polymer host, on the one hand, should possess proper LUMO/HOMO energy levels matching with the cathode/anode to facilitate charge carrier injection.<sup>12,13</sup> On the other hand, a polymer host should have a high triplet energy level  $(E_T)$  to prevent triplet energy back transfer from the dopant to the host and the consequent quenching of triplet excitons.14 Unfortunately, polymer host materials with both high E<sub>T</sub> and proper LUMO/HOMO energy levels are scarce.

PPLEDs typically use a non-conjugated polymer, poly (vinylcarbazole) (PVK), as the host because of its high  $E_T$ . However, PVK is a good hole transporter but a poor electron transporter. Therefore, PVK has to be admixed with electron transporting materials for charge carrier balance, which raises the probability of phase separation during long-term device operation.<sup>2–8</sup> Conjugated polymers, such as poly(2,7-fluorene)s (PFs), are excellent host materials because of their suitable

LUMO/HOMO energy levels and high conductivity.15-17 However, PFs have an E<sub>T</sub> of only about 2.2 eV, which is lower than those of green (2.4 eV) and blue (2.6 eV) phosphorescent dopants. Therefore, PFs cannot efficiently host blue and green electrophosphorescence.<sup>18-20</sup> Recently, several high  $E_T$  conjugated polymers ( $E_T = 2.5-2.6$  eV) featuring a meta-linkage in the backbone were reported as promising host materials for green PPLEDs, such as poly(3,6-carbazole),<sup>13,14,21,22</sup> poly(3,6-silafluorene)<sup>23-25</sup> and poly(3,6-fluorene).<sup>26,27</sup> We reported that poly(meta-phenylene) had an  $E_T$  as high as 2.64 eV and could be used as a conjugated polymer host for efficient blue electrophosphorescence.28,29 While the meta-linkages in the backbone of these polymers increase the  $E_{T}$ , they also increase the bandgap of the polymers and lead to larger barriers for charge carrier injections. The resulting single-layer devices exhibit poor electroluminescence (EL) performance. Although insertion of an additional charge carrier injection layer boosts EL efficiency, the device fabrication becomes more complicated.

Herein, we improve charge carrier injection/transporting abilities of high-E<sub>T</sub> conjugated polymer hosts by incorporating both hole transporting unit and electron transporting unit to the conjugated polymer backbone. Poly(meta-phenylene) has been proved by us to exhibit  $E_T$  high enough to host blue and green electrophosphorescence.<sup>28</sup> Carbazole and oxadiazole are well-known hole transporting materials and electron transporting materials, respectively. They both have been introduced to conjugated polymers to improve charge carrier transporting.<sup>30-33</sup> In this manuscript, we develop a novel ambipolar high-E<sub>T</sub> conjugated polymer host (PmPCz-Ox, see Scheme 1) with a carbazole unit and oxadiazole unit incorporated to a poly(meta-phenylene) backbone. This ambipolar polymer host retains high  $E_T$  (2.60 eV) of the poly (meta-phenylene) backbone and possesses suitable LUMO (-2.43 eV) and HOMO (-5.57 eV) energy levels owing to the oxadiazole unit and the carbazole unit, respectively. Therefore, PmPCz-Ox is expected to be a suitable host for blue and green PPLEDs with high efficiencies and low driving voltages.

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Scheme 1 Chemical structures and synthetic routes of PmPCz-Ox.

#### **Experimental section**

#### Materials

9-(6-(3,5-Dibromophenoxy)hexyl)-9*H*-carbazole (1)<sup>28</sup> and 2-(3,5dibromophenyl)-5-phenyl-1,3,4-oxadiazole (3)<sup>13</sup> were prepared according to the reported procedures. All the reactions were carried out under argon gas protection. The phosphorescent dopants, *fac*-tris[2-(2-pyridyl-kN)-5-mehylphenyl]iridium(III) (Ir(mppy)<sub>3</sub>) and bis[(4,6-difluorophenyl)pyridinato-N,C<sup>2</sup>]-(picolinato)iridium(III) (FIrpic), were purchased from American Dye Source.

### 9-(6-(3,5-Bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) phenoxy)hexyl)-9*H*-carbazole (2)

A mixture of 9-(6-(3,5-dibromophenoxy)hexyl)-9H-carbazole (1) (0.54 g, 1.08 mmol), Pd(dppf)<sub>2</sub>Cl<sub>2</sub> (0.048 g, 0.065 mmol), potassium acetate (0.64 g, 6.53 mmol), bis(pinacolato)diboron (0.61 g, 2.40 mmol) and DMSO (15 mL) was stirred at 80 °C overnight. After being cooled down, the mixture was poured into brine and extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic layer was washed with water three times, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated. The residual was purified by column chromatography with  $CH_2Cl_2$ /hexane = 1/1 as the eluent to give the title compound as a white solid. Yield: 0.47 g (73.0%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm): 8.10 (d, 2H), 7.86 (s, 1H), 7.46 (m, 6H), 7.23 (td, 2H), 4.32 (t, 2H), 3.97 (t, 2H), 1.91 (m, 2H), 1.74 (m, 2H), 1.53 (m, 4H), 1.33 (s, 24H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ (ppm): 150.07, 140.42, 133.50, 125.62, 123.50, 122.82, 120.33, 118.71, 108.66, 83.77, 67.56, 42.97, 29.28, 28.92, 27.05, 25.94, 24.88. HR-MS (m/z (%)): 596.37 (100) [M + H]<sup>+</sup>. Anal. Calcd. for C<sub>36</sub>H<sub>47</sub>B<sub>2</sub>NO<sub>5</sub>: C, 72.62; H, 7.96; N, 2.35. Found: C, 72.93; H, 8.05; N, 2.35.

#### PmPCz-Ox

To a mixture of 9-(6-(3,5-bis(4,4,5,5-tetramethyl-1,3,2-dioxa-borolan-2-yl)phenoxy)hexyl)-9H-carbazole (2) (0.2827 g,

0.500 mmol), 2-(3,5-dibromophenyl)-5-phenyl-1,3,4-oxadiazole (3) (0.1907 g, 0.500 mmol), Pd(PPh<sub>3</sub>)<sub>4</sub> (0.0101 g, 0.0100 mmol) and Aliquat 336 (1 drop) under argon atmosphere was added K<sub>2</sub>CO<sub>3</sub> (2.0 mL 2.0 M aqueous solution) and toluene (6 mL). The mixture was stirred at 90 °C for 48 h. After being cooled down to room temperature, the mixture was poured into CHCl<sub>3</sub> and washed with water three times, followed by drying over anhydrous Na<sub>2</sub>SO<sub>4</sub> and filtration. The filtrate was concentrated and precipitated in methanol to give the crude product as a white powder, which was further purified by re-precipitation twice in CHCl<sub>3</sub>/CH<sub>3</sub>OH. The powder was collected and dried in vacuum overnight. Yield: 0.17 g (51.5%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 8.14–7.93 (br, 7H), 7.55–7.27 (br, 8H), 7.10 (br, 4H), 4.15 (br, 2H), 3.91 (br, 2H), 1.80 (br, 4H), 1.30 (br, 4H). Anal. Calcd. for C<sub>38</sub>H<sub>31</sub>N<sub>3</sub>O<sub>2</sub>: C, 81.26; H, 5.56; N, 7.48. Found: C, 80.33; H, 5.60; N, 7.21.

#### Characterization

<sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker arx-400 spectrometer. Elemental analysis was performed with a Perkin-Elmer 2400 elemental analyzer. Molecular weights of the polymer were measured with gel permeation chromatography (GPC) method using mono-disperse polystyrene standards and tetrahydrofuran (THF) as the eluent. Thermal gravimetic analysis (TGA) was carried out on a Perkin-Elmer TGA-7 system with a heating rate of 20 °C/min. Differential scanning calorimetry (DSC) was performed on a Perkin Elmer DSC-6 system with a heating rate of 20 °C/min. Cyclic voltammetry (CV) was conducted in a solution of Bu<sub>4</sub>NBF<sub>4</sub> (0.1 M) in acetonitrile with Pt wire, Pt plate and saturated calomel electrode (SCE) as the working electrode, counter electrode and reference electrode, respectively. The polymer film was dip-coated on the working electrode from its solution in methylene chloride. Absorption spectra were obtained from a Shimadzu UV-1700 UV/Vis spectrophotometer. Fluorescence spectra at room temperature and phosphorescence spectra at 77 K were measured with a PTI QuantaMaster 30 spectrofluorometer. Current-voltage and brightness-voltage curves of PPLED devices were recorded by a computer-controlled Keithley 2400/2002 source unit calibrated with a Photoresearch PR-655 spectrophotometer. Electroluminescence spectra and CIE coordinates were recorded with the Photoresearch PR-655 spectrophotometer.

#### **Device fabrication**

Indium tin oxide (ITO) glass substrates were ultrasonically cleaned for 30 min each sequentially with detergent, de-ionized water, acetone and isopropanol. Then they were dried in a heating chamber at 70 °C. PEDOT:PSS (Clevios VP Al 4083 from H. C. Starck Inc.) was spin-coated on the ITO glass substrates at 3000 rpm for 60 s and then baked at 120 °C for 15 min to give an approximate thickness of 40 nm. The emissive layer (70 nm) was then spin-coated from the solution of the polymer host (20 mg mL<sup>-1</sup>) and the dopants in chlorobenzene. Finally, a thin layer of CsF (1.0 nm) followed by a layer of aluminum (100 nm) were deposited in a vacuum thermal evaporator through a shadow mask at a pressure of  $10^{-6}$  Torr. The active area of the devices is 0.126 cm<sup>2</sup>.

#### **Results and discussion**

#### Design and synthesis of PmPCz-Ox

The chemical structure of the polymer host PmPCz-Ox is shown in Scheme 1. The hole-transporting carbazole unit is connected to the poly(*meta*-phenylene) backbone through an alkyloxy spacer and the electron-transporting oxadiazole unit is in conjugation with the poly(meta-phenylene) backbone. As shown in Scheme 1. The diboronate monomer 2 was synthesized from carbazole-tethering dibromobenzene 1 and bis(pinacolato) diboron under palladium catalysis. The 2,5-diphenyl-1,3,4-oxadiazole dibromide monomer 3 was obtained in a four-step synthesis following the literature procedures.<sup>13</sup> With the two monomers in hand, we synthesized the alternating polymer **PmPCz-Ox** by Suzuki polycondensation with palladium catalysis.34 Its chemical structure is verified by 1H NMR and elemental analysis. PmPCz-Ox is readily soluble in common organic solvents, such as chloroform, toluene and chlorobenzene. The weight average molecular weight (Mw) and polydispersity (PDI) of PmPCz-Ox, as determined by GPC with polystyrene standards, are 5700 and 1.59, respectively.

The thermal properties of **PmPCz-Ox** are investigated using TGA and DSC under nitrogen atmosphere. **PmPCz-Ox** exhibits good thermal stability with 5% weight loss temperature ( $T_d$ ) at 404 °C. The polymer does not show a glass transition, crystallization, or melting behavior in the temperature range 25 °C–250 °C, implying good morphology stability at normal device operation conditions.

The compatibility of **PmPCz-Ox** and phosphorescent dopants is explored by atom force microscopy (AFM). Fig. 1 shows the phase images of the film spin-coated from the blends of **PmPCz-Ox** with FIrpic or Ir(mppy)<sub>3</sub>. The phase images exhibit a uniform surface without pinholes, particles or phase separation, indicating excellent compatibility of **PmPCz-Ox** with FIrpic or Ir (mppy)<sub>3</sub>. This compatibility ensures good dispersion of the phosphorescent dopants in the host and is necessary for excellent device performance.

#### Photophysical properties

Fig. 2 shows the absorption and photoluminescence (PL) spectra of **PmPCz-Ox** in dilute  $CH_2Cl_2$  solution and in a thin film. **PmPCz-Ox** in dilute solution has a broad absorption band spanning from 308 nm to 255 nm, attributed to the oxadiazoleincorporated poly(*meta*-phenylene) backbone. There is a small



**Fig. 1** AFM phase images of the films spin-coated from the blends of **PmPCz-Ox** (20 mg mL<sup>-1</sup> solution in chlorobenzene) with (a) FIrpic (10 wt%) or (b) Ir(mppy)<sub>3</sub> (1 wt%).



Fig. 2 Absorption spectra and fluorescence spectra of PmPCz-Ox in dilute solution and in solid film.

peak at 337 nm stemming from the carbazole unit. The absorption spectrum in the thin film is red-shifted by 10 nm compared to that in solution because of the interaction between the polymer chains in the solid state. The PL spectrum of PmPCz-Ox in solution shows two vibronic peaks at 352 nm and 368 nm with a broad band at 410 nm. The former two peaks are characteristic of the carbazole unit while the latter broad band comes from the exciplex between the carbazole unit and the oxadiazole unit.35 In the thin film, the emission from the carbazole unit disappears and the exciplex emission dominates the PL spectrum owing to the close packing of the carbazole unit and the oxadiazole unit. The exciplex between the carbazole unit and oxadiazole unit has always been observed in blends of PVK and 2-(4-biphenylyl)-5-(4-tert-butylphenyl)-1,3,4-oxadiazole (PBD).<sup>2-8</sup> Such blends are frequently used as hosts for PPLEDs. The formation of the exciplex has been shown not to affect EL performance of PPLEDs. Fig. 3 shows the overlap of the fluorescent spectrum of **PmPCz-Ox** along with the absorption spectra of FIrpic and Ir (mppy)<sub>3</sub>. The broad overlap is beneficial for efficient Förster energy transfer from the polymer host to the dopants.

The phosphorescence spectrum of **PmPCz-Ox** dissolved in THF was measured at 77 K (see Fig. 4). Deconvolution of the phosphorescence spectrum gives three peaks at 474 nm, 501 nm and 542 nm. Using peak energy at 474 nm, the  $E_T$  of **PmPCz-Ox** 



**Fig. 3** PL spectrum of **PmPCz-Ox** in thin film and absorption spectra of FIrpic and Ir(mppy)<sub>3</sub> in dilute solution.



**Fig. 4** Deconvolution of the phosphorescence spectrum of **PmPCz-Ox** in THF at 77 K.

is calculated to be 2.60 eV. This value is slightly lower than that of the blue phosphorescent dopant, FIrpic (2.62 eV), and higher than that of green phosphorescent dopant,  $Ir(mppy)_3$  (2.41 eV).<sup>36</sup> Therefore, when **PmPCz-Ox** is hosting green electrophosphorescence, triplet excitons can be confined in  $Ir(mppy)_3$ without triplet energy back transfer. However, for hosting blue electrophosphorescence, the confinement of triplet excitons in FIrpic is questionable. The  $E_T$  of **PmPCz-Ox** is slightly lower than that of the previously reported conjugated polymer host based on poly(*meta*-phenylene) (2.64 eV),<sup>28,29</sup> probably due to the conjugation of the oxadiazole unit with the poly(*meta*-phenylene) backbone.

To investigate the potential of PmPCz-Ox as a phosphorescence host, we blended PmPCz-Ox with FIrpic or Ir(mppy)<sub>3</sub> and measured their PL spectra in thin films. The results are shown in Fig. 5. When **PmPCz-Ox** is doped with FIrpic, the PL spectra show emission from both PmPCz-Ox and FIrpic. With increasing FIrpic content, the relative intensity of FIrpic emission increases while the relative intensity of PmPCz-Ox emission decreases. These results indicate no serious triplet energy back transfer from FIrpic to PmPCz-Ox and are consistent with the similar  $E_T$  of **PmPCz-Ox** and FIrpic. For the blends of the polymer host with Ir(mppy)<sub>3</sub>, the same trend is observed. When the Ir(mppy)<sub>3</sub> content is higher than 5 wt%, the emission from PmPCz-Ox is almost completely quenched. The energy transfer from host to phosphorescent dopant is more complete in the case of Ir(mppy)<sub>3</sub> compared to that of FIrpic. Two possible factors may be responsible for the difference. One is the more efficient Förster energy transfer from the host to  $Ir(mppy)_3$  than that from the host to FIrpic. This would be consistent with the observed larger spectral overlap in the case of Ir(mppy)<sub>3</sub> (see Fig. 3). The other one is the possible slight triplet energy back transfer from FIrpic to **PmPCz-Ox** due to their  $E_T$  alignment.

#### **Electrochemical properties**

Cyclic voltammetry of **PmPCz-Ox** film was employed to investigate the redox behavior and to estimate the LUMO/HOMO energy levels of the polymer host. The cyclic voltammogram exhibits one irreversible reduction wave with an onset potential at -1.97 V, due to the electron-deficient oxadiazole unit. There are two irreversible oxidation waves with the onset potentials at



**Fig. 5** PL spectra of **PmPCz-Ox** blends with FIrpic (a) or  $Ir(mppy)_3$  (b) with specified phosphorescent dopant contents.

1.17 V and 1.50 V, respectively. The former is attributed to the carbazole unit, while the latter the poly(*meta*-phenylene) backbone. According to the empirical formula ( $E_{HOMO} = -e$  ( $E_{ox} + 4.4$ ) [eV],  $E_{LUMO} = -e$  ( $E_{red} + 4.4$ ) [eV]),<sup>37</sup> the HOMO and LUMO energy levels of **PmPCz-Ox** are calculated to be -5.57 eV and -2.43 eV, respectively. The  $E_{HOMO}$  matches well with the work functions of the anode (ITO/PEDOT:PSS: -5.2 eV), and the  $E_{LUMO}$  with the cathode (CsF/Al: -2.2 eV). These results indicate that the incorporation of carbazole unit and oxadiazole unit would facilitate both hole injection and electron injection into **PmPCz-Ox**.

#### **Electroluminescent properties**

Polymers with high  $E_T$  generally have large bandgaps and suffer from difficult charge injections due to misalignment of their HOMO/LUMO with Fermi energy levels of electrodes. Therefore, their single-layer devices always exhibit very low EL efficiencies.<sup>13,24,26</sup> A charge carrier transporting material has to be blended in the emissive layer<sup>3,4,6</sup> or inserted as an additional charge injection layer<sup>21,25,39-42</sup> to improve charge carrier balance for high EL efficiency. The proper LUMO/HOMO energy levels of **PmPCz-Ox** are expected to facilitate charge carrier injections and enable fabrication of single-layer devices. The single (emissive) layer would employ a blend of **PmPCz-Ox** with 10 wt% of FIrpic or 1 wt% of Ir(mppy)<sub>3</sub>. Accordingly, single-layer devices were fabricated with a sandwich structure of ITO/PEDOT:PSS (40 nm)/emissive layer (70 nm)/CsF (1 nm)/Al (100 nm).

As shown in Fig. 6, the EL spectra of the two devices exhibit emission exclusively from FIrpic ( $\lambda_{max} = 472 \text{ nm}$ ) or Ir(mppy)<sub>3</sub> ( $\lambda_{max} = 512 \text{ nm}$ ). No contribution from **PmPCz-Ox** is observed in the EL spectra. Note the presence of emission from the polymer in the PL spectra of the blends (see Fig. 5). The absence of the host emission in the EL spectra can be attributed to charge trapping effect by FIrpic and Ir(mppy)<sub>3</sub>.<sup>38</sup>

The primary PPLED device performance data are listed in Table 1. Fig. 7 shows the luminance efficiencies as a function of current densities. A typical blue electrophosphorescent device has a turn-on voltage of 7.6 V, luminance efficiency of 0.50 cd  $A^{-1}$  and maximum brightness of 160 cd m<sup>-12</sup>. This performance is somewhat worse than that of poly(meta-phenylene) tethered with carbazole.<sup>28</sup> The reason is unknown. Possibly, PmPCz-Ox requires optimized device structure for good EL performance. The green electrophosphorescent device has a turn-on voltage of 6.5 V, luminance efficiency of 20.9 cd A<sup>-1</sup> and maximum brightness of 6050 cd  $m^{-12}$ . This performance is among the highest reported for green PPLEDs based on conjugated polymer host, indicating that PmPCz-Ox is an excellent host for green electrophosphorescence. For comparison, we use carbazole-tethering poly(meta-phenylene) without an oxadiazole unit<sup>28</sup> as the host to fabricate control green PPLED. The control device exhibits a luminance efficiency of 12.8 cd A<sup>-1</sup>, which is lower than of PmPCz-Ox. Therefore, the excellence of PmPCz-



Fig. 6 EL spectra of single layer devices, ITO/PEDOT:PSS/emissive layer/CsF/Al, wherein the emissive layer is a blend of **PmPCz-Ox** with 10 wt% FIrpic (a) or 1 wt%  $Ir(mppy)_3$  (b).

Table 1 EL performance of the blue and green PPLED devices

Emissive layer	<b>PmPCz-Ox</b> :10 wt% FIrpic	<b>PmPCz-Ox</b> :1 wt% Ir(mppy) <sub>3</sub>
Turn-on voltage (V)	7.6	6.5
Maximum brightness (cd m <sup>-2</sup> )	160	7650
Luminance efficiency (cd $A^{-1}$ )	0.50	20.9
Power efficiency (lm/W)	0.14	6.4
EL maximum (nm)	472	512
CIE coordinates (x,y)	(0.16, 0.32)	(0.24, 0.65)



**Fig. 7** Dependence of luminance efficiency on current density of the blue and green PPLEDs.

**Ox** is attributed to the high triplet energy level due to the poly (*meta*-phenylene) backbone, and the proper LUMO/HOMO energy levels owing to the oxadiazole/carbazole units. This result demonstrates the molecular design strategy of ambipolar polymer host materials with electron–hole transporting units incorporated into a high- $E_T$  conjugated polymer backbone.

#### Conclusion

In summary, we have developed an ambipolar conjugated polymer host material PmPCz-Ox with both a hole transporting carbazole unit and electron transporting oxadiazole unit incorporated into a poly(meta-phenylene) backbone. The poly(metaphenylene) backbone ensures high  $E_T$  (2.60 eV) of PmPCz-Ox. The carbazole unit raises the polymer's HOMO energy level to enhance hole injection, while the oxadiazole unit lowers the LUMO energy level, to boost electron injection. The PPLED devices with PmPCz-Ox doped with green or blue phosphorescent dopants as the active layer emit exclusive phosphorescent light from the dopants. The preliminary devices show a luminance efficiency of 20.9 cd A<sup>-1</sup> for green electrophosphorescence and 0.5 cd  $A^{-1}$  for blue emission. The excellent EL performance of the green PPLED demonstrates our molecular design of ambipolar polymer hosts, in which the electron transporting moiety and hole transporting moiety are incorporated into a polymer backbone with high  $E_T$ . We believe that through further molecular design, ambipolar conjugated polymer hosts can be developed with sufficiently high E<sub>T</sub> to host blue phosphorescent dopants.

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