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Conjugated Polymer as Host for High Efficiency Blue and White Electrophosphorescence

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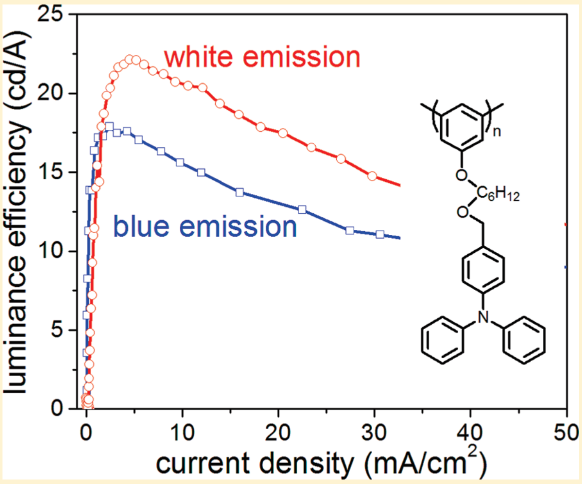

ABSTRACT: We demonstrate that conjugated polymers are able to efficiently host blue and white electrophosphorescence if the conjugated polymer has both high triplet energy level (E_T) and high-lying HOMO energy level. A novel conjugated polymer host (**PmPTPA**) is developed by attaching triphenylamine unit to poly(*m*-phenylene) backbone. The poly(*m*-phenylene) backbone endows **PmPTPA** an E_T as high as 2.65 eV, which is sufficiently high to prevent triplet energy back transfer. The tethering triphenylamine unit leads to the HOMO energy level of −5.35 eV for **PmPTPA** and facilitates hole injection. As the result, blue phosphorescent polymer light-emitting diodes (PPLEDs) based on **PmPTPA** exhibit the luminance efficiency of 17.9 cd/A and external quantum efficiency of 9.3%. White PPLEDs with blue, green and red phosphorescent dopants dispersed in **PmPTPA** show the luminance efficiency of 22.1 cd/A and external quantum efficiency of 10.6%. For both the blue and white PPLEDs based on the conjugated polymer host **PmPTPA**, the EL performance are fairly comparable to those of the state-of-the-art nonconjugated polymer host, poly(vinyl−carbazole) (PVK). These results indicate that conjugated polymers are suitable host materials for PPLEDs with all emission colors.

INTRODUCTION

Polymer light-emitting diodes (PLEDs) have been intensively investigated for important applications such as flat-panel displays and solid-state lighting with low-cost solution processing.[1] Phosphorescent PLEDs (PPLEDs) employing phosphorescent dopants dispersed in a polymer host have the potential to harvest both singlet and triplet excitons and obtain 100% internal electron-to-photon quantum efficiency.[2−22] Among variable emission colors, blue and white are the most difficult to achieve in PPLEDs:[3−7,17−22] the efficiencies of blue and white PPLEDs are rather low compared to red and green devices, largely due to lack of a suitable polymer host for blue phosphorescent dopants. An ideal polymer host should have a triplet energy level (E_T) higher than that of the phosphorescent dopants. Otherwise, triplet energy back transfer from the phosphorescent dopants to the host will quench triplet excitons and leads to low electroluminescence (EL) efficiency.[23] Meanwhile, the polymer host should have proper LUMO/HOMO energy levels to facilitate the injection and transport of both electrons and holes. Currently, the most widely used polymer host for blue PPLEDs is a nonconjugated polymer, poly(vinyl−carbazole) (PVK).[3−7] However, PVK has a low lying HOMO energy level of −5.9 eV, which mismatches work function of common anode and results in difficult hole injection. Moreover, PPLEDs using PVK all exhibit short lifetime. Therefore, it is strongly required to develop polymer host with both high E_T and proper LUMO/HOMO energy levels.

Although conjugated polymers have been demonstrated to be suitable host materials for green and red PPLEDs,[8−16] the reports of conjugated polymers in blue and white PPLEDs have been scare due to the generally low E_T of conjugated polymers. The state-of-the-art blue phosphorescent dopant, iridium(III) [bis(4,6-difluorophenyl)pyridinato-*N*,C^2]picolinate (FIrpic), has an E_T of 2.62 eV. However, only until very recently, we reported the first conjugated polymer, poly(*m*-phenylene) containing carbazole unit as a side group (**PmPCz**), that has an $E_T >$ 2.62 eV.[24] Owing to the absence of triplet energy back transfer, PPLEDs employing **PmPCz** doped with FIrpic exhibit a luminance efficiency of 4.7 cd/A, which is a 10 times improvement over previously reported blue PPLEDs based on a conjugated polymer host.[25] However, this efficiency is still lower than that of the blue PPLEDs based on the blend of nonconjugated polymer PVK and an electron-transport material (15−20 cd/A).[3−7] The low efficiency is caused by the mismatch of the

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HOMO energy level of **PmPCz** with the work function of the anode.[24]

White PPLEDs are of particular importance because of their great potential for energy saving solid-state lighting and low cost flat-panel display.[17−22] The development of white PPLEDs is also hampered by the lack of a polymer host for blue phosphorescence. Until now, all efficient (>20 cd/A) white PPLEDs use PVK as the host, which suffers from difficult hole injection and short lifetime as mentioned above.[17−19] There is no report of high efficiency (>20 cd/A) white PPLEDs based on a conjugated polymer host.

Here, we report a novel conjugated polymer host (**PmPTPA**) based on poly(*m*-phenylene) backbone containing triphenylamine tethered side groups. The poly(*m*-phenylene) backbone and triphenylamine moiety impart **PmPTPA** a high E_T (2.65 eV) as well as a high-lying HOMO energy level (−5.35 eV), which prevents triplet energy back transfer and facilitates hole injection, respectively. Blue PPLEDs based on **PmPTPA** doped with FIrpic exhibit a luminance efficiency of 17.9 cd/A and an external quantum efficiency of 9.3%. When doped with blue, green and red emissive Ir complexes with appropriate ratio, white PPLEDs are obtained with the CIE coordinates of (0.37, 0.47), luminance efficiency of 22.1 cd/A and external quantum efficiency of 10.6%. For both the blue and the white PPLEDs, the EL performance of this conjugated polymer host **PmPTPA** are fairly comparable to those of the state-of-the art nonconjugated polymer host PVK.[3−7,17−22] These results indicate that conjugated polymers are suitable host materials for PPLEDs with all emission colors.

EXPERIMENTAL SECTION

Materials. All chemicals and reagents for synthesis were purchased from Aldrich Chemical Co. and used as received without further purification. 4-(Diphenylamino)benzaldehyde was synthesized according to the literature procedures.[26] All the reactions were carried out under argon atmosphere. The three phosphorescent dopants, iridium (III) [bis(4,6-difluorophenyl)pyridinato-*N*,C^2]picolinate (FIrpic), iridium (III) tris[2-(p-tolyl)pyridine] (Ir(mppy)$_3$) and iridium(III) tris(1-phenyl isoquinoline) (Ir(piq)$_3$), were purchased from American Dye Source.

Characterization. ^{1}H NMR spectra were conducted on a Bruker arx-400 spectrometer. Elemental analysis was performed with a Perkin-Elmer 2400 elemental analyzer. Molecular weights of the polymer were measured by gel permeation chromatography (GPC) method using tetrahydronfuran (THF) as the eluent and polystyrene as the standard. Cyclic voltammetry (CV) was carried out in a solution of Bu_4NBF_4 (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 electroluminescent devices were recorded by a computer-controlled Keithley 2400/2002 source unit calibrated with a Photoresearch PR-655 spectrophotometer. Electroluminescence spectra were measured by the Photoresearch PR-655 spectrophotometer. Luminance efficiencies, power efficiencies and external quantum efficiencies of the devices were calculated according to the voltage−current-brightness curves and the electroluminescence spectra.

Device Fabrication. Indium tin oxide (ITO) glass substrates were ultrasonically cleaned for 30 min each sequentially with detergent, deionized water, acetone and 2-propanol. Then they were dried in a heating chamber at 70 °C for 20 min. The poly(3,4-ethylenedioxythiophene) doped with poly(styrenesulfonate) (PEDOT:PSS, Clevios VP Al 4083 from H. C. Starck Inc.) layer was spin-coated on ITO 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 (approximate 70 nm) was then spin-coated at 1500 rpm for 60 s from the solution of **PmPTPA** (20 mg/mL) and the phosphorescent dopants in chlorobenzene. After the spin-coating, the devices were transferred to a vacuum chamber. At a pressure of 10^{-6} Torr, 1,3-bis[(4-*tert*-butylphenyl)-1,3,4-oxadiazolyl]phenylene (OXD-7, 30 nm), CsF (1.0 nm) and aluminum (100 nm) were sequentially deposited by thermal evaporation through a shadow mask. The active area of the devices was 0.12 cm^2.

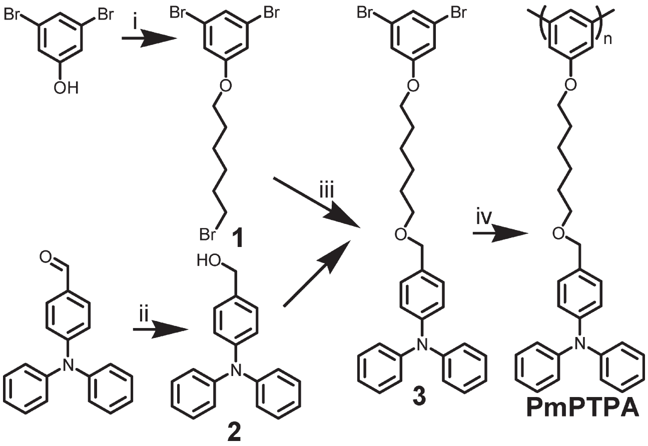
Synthesis of 1-(6-Bromohexyloxy)-3,5-dibromobenzene (1). A mixture of 3,5-dibromophenol (2.58, 10.24 mmol), 1,6-dibromohexane (3.2 mL, 20.85 mmol), NaOH (aq., 50 wt %, 7 mL), toluene (30 mL) and tetrabutylammonium bromide (0.32 g, 1.00 mmol) was stirred at 80 °C for 24 h. After work-up, the organic layer was separated and washed with water for three times. The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated. Column chromatography on silica gel with CH_2Cl_2/hexane = 1/4 as eluent afforded the title compound as a white solid. Yield: 3.21 g (75.5%). ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 7.23 (s, 1H), 6.98 (s, 2H), 3.92 (t, 2H), 3.43 (t, 2H), 1.89 (m, 2H), 1.80 (m, 2H), 1.50 (m, 4H). Anal. Calcd for $C_{12}H_{15}Br_3O$: C, 34.73; H, 3.64; Found: C, 35.52; H, 3.38.

Synthesis of (4-(Diphenylamino)phenyl)methanol (2). To a dispersion of $LiAlH_4$ (0.76 g, 20.00 mmol) in THF (20 mL) at room temperature was added dropwise a solution of 4-(diphenyl amino)benzaldehyde (2.50 g, 9.16 mmol) in THF (20 mL). After finished, the mixture was stirred at room temperature overnight. Several drops of water were carefully added to the mixture to destroy the excessive $LiAlH_4$, followed by removal of the solvent. The residual was dissolved in CH_2Cl_2, washed with water for three times, dried with anhydrous Na_2SO_4, and then filtered. Removal of the solvent gave the title compound as a white solid. Yield: 1.90 g (75.4%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.24 (m, 6H), 7.07 (m, 6H), 7.01 (m, 2H), 4.64 (s, 2H). Anal. Calcd for $C_{19}H_{17}NO$: C, 82.88; H, 6.22; N, 5.09; Found: C, 82.50; H, 6.22; N, 4.94.

Synthesis of *N*-(4-((6-(3,5-Dibromophenoxy)hexyloxy)methyl)phenyl)-*N*-phenylbenzenamine (3). A mixture of 1-(6-bromohexyloxy)-3,5-dibromobenzene (**1**) (0.89 g, 2.15 mmol), (4-(diphenylamino)phenyl)methanol (**2**) (0.60 g, 2.18 mmol), NaOH (aqueous, 20 wt %, 4 mL), toluene (2 mL), and tetrabutylammonium bromide (0.06 g, 0.20 mmol) was stirred at 80 °C for 24 h. After work-up, the organic layer was separated and washed with water for three times. The organic layer was dried with anhydrous Na_2SO_4, filtered, and concentrated. Column chromatography on silica gel with CH_2Cl_2/hexane = 1/4 as eluent afforded the title compound as a viscous liquid. Yield: 0.90 g, (68.7%). ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 7.24 (m, 6H), 7.06 (m, 11H), 4.44 (s, 2H), 3.92 (t, 2H), 3.51 (t, 2H), 1.78 (m, 2H), 1.66 (m, 2H), 1.50 (m, 4H). Anal. Calcd for $C_{31}H_{31}Br_2NO_2$: C, 61.10; H, 5.13; N, 2.30; Found: C, 60.78; H, 5.21; N, 2.15.

Synthesis of PmPTPA. To a mixture of bis(1,5-cyclooctadiene) nickel(0) (0.33 g, 1.20 mmol), 1,5-cyclooctadiene (0.13 g, 1.20 mmol), bipyridine (0.19 g, 1.20 mmol), *N*,*N*-dimethylformamide (5 mL) and toluene (3 mL) in argon at 80 °C was added a solution of *N*-(4-((6-(3,5-dibromophenoxy)hexyloxy)methyl)phenyl)-*N*-phenylbenzenamine (**3**) (0.60 g, 1.00 mmol) in toluene (2 mL). The mixture was stirred in dark at 80 °C for 2 days. After work-up, the mixture was poured to methanol. The solid was collected and dissolved in CH_2Cl_2. The resulting solution was washed with water for three times, dried over Na_2SO_4, filtered and concentrated. The residual was poured into methanol to give white solid. Reprecipitation in CH_2Cl_2/CH_3OH twice afforded the title polymer as white powder. Yield: 0.13 g. (35.2%) ^{1}H NMR (400 MHz, $CDCl_3$), δ (ppm): 7.28−7.13 (br, 6H), 7.05−6.90 (br, 11H), 4.34 (br, 2H), 3.94

Scheme 1. Chemical Structure and Synthetic Route of PmPTPA[a]



[a] Reagents and conditions: (i) 1,6-dibromohexane, NaOH(aq), toluene, $(n\text{-}C_4H_9)_4NBr$, 80 °C; (ii) $LiAlH_4$, THF, room temperature; (iii) NaOH(aq), toluene, $(n\text{-}C_4H_9)_4NBr$, 80 °C; (iv) bis(1,5-cyclooctadiene)nickel(0), 1,5-cyclooctadiene, bipyridine, DMF, toluene, 80 °C.

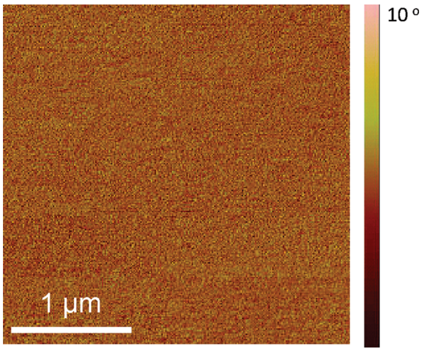


Figure 1. AFM phase image of the film spincoated from the blend of **PmPTPA** and FIrpic (15 wt %).

(br. 2H), 3.50 (br, 2H), 1.84 (br, 4H), 1.30 (br, 4H). Anal. Calcd for $C_{31}H_{31}NO_2$: C, 82.82; H, 6.95; N, 3.12; Found: C, 82.75; H, 7.23; N, 2.89.

RESULTS AND DISCUSSION

Scheme 1 outlines the synthetic route for the polymer host **PmPTPA**. Reduction of 4-(diphenylamino)benzaldehyde with $LiAlH_4$ readily afforded **2**. The key monomer **3** was readily synthesized via two etherification reactions of 1,6-dibromohexane with 3,5-dibromophenol leading to ether **1** and **1** with **2**. The overall yield was 54%. The monomer **3** was polymerized to afford **PmPTPA** using the Yamamoto condition with bis(1,5-cyclooctadiene)nickel(0) as the catalyst. The yield of the polymerization was 35% because of the loss in the purification process.

For a host material, the compatibility of the host with the dopants is very important for the dispersion of the dopants in the host and for the energy transfer from the host to the dopants. The compatibility of **PmPTPA** and FIrpic is investigated by atom force microscopy (AFM). As shown in Figure 1, the phase image of the film of the blend of **PmPTPA** and FIrpic (15 wt %) shows smooth and uniform surface without phase separation, indicating the good compatibility of the host and the dopant.

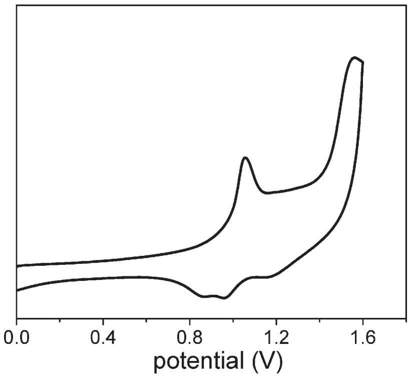


Figure 2. CV of **PmPTPA** in solid film.

PmPTPA is readily soluble in common organic solvents, such as toluene, chlorobenzene, chloroform and tetrahydrofuran. Its chemical structure is verified by ^1H NMR and elemental analysis. The number-average molecular weight (M_n) is 5400 with a polydispersity of 1.42, as determined by GPC against poly(styrene) standards. The reason for the moderate molecular weight of **PmPTPA** is not clear yet. Thermogravimetric analysis (TGA) of **PmPTPA** shows a decomposition temperature (T_d, corresponding to 5% weight loss) of 380 °C, indicative of good thermal stability.

Cyclic voltammetry was employed to estimate the LUMO/HOMO energy levels of the conjugated polymer host. As shown in Figure 2, cyclic voltammogram of **PmPTPA** shows two oxidation peaks with the onset potentials at 0.95 and 1.45 V (vs SCE), respectively. The first peak is attributed to the triphenylamine unit and the second to the poly(*m*-phenylene) backbone. No reduction wave is detected. According to the first onset oxidation potential and the empirical formula, $E_{HOMO} = -e(E_{ox} + 4.4)$ [eV], the HOMO energy level of **PmPTPA** is estimated to be −5.35 eV. On the basis of the absorption onset of the polymer in film, the optical bandgap of **PmPTPA** is 3.49 eV. Therefore, the LUMO energy level is calculated to be −1.86 eV. The HOMO of **PmPTPA** is much higher than that of our previous polymer host **PmPCz** (HOMO: −5.56 eV)[24] and PVK (HOMO: −5.90 eV) due to the strong electron donating ability of triphenylamine unit in **PmPTPA**. The HOMO energy level of **PmPTPA** is close to the Fermi level of PEDOT:PSS (−5.2 eV), which can lead to a small hole-injection barrier and improved hole injection for **PmPTPA**. Thus, low driving voltage and high device efficiency can be expected.

Figure 3 shows the absorption spectrum and fluorescence spectrum of **PmPTPA** in dilute solution and in thin film. The absorption band at 309 nm in film is attributed to the triphenylamine unit.[24] The fluorescence spectrum of **PmPTPA** in solution exhibits two bands at 365 and 428 nm, which are assigned to the emission from triphenylamine unit itself and excimer, respectively. In thin film, the fluorescence spectrum shows emission predominantly from the excimer due to the close packing of triphenylamine unit in solid state. The phosphorescence spectrum of **PmPTPA** at 77 K (see Figure 4) is characteristic of poly(*m*-phenylene) backbone.[24] Deconvolution of the broad phosphorescence spectrum gives the higher energy band at 469 nm. Therefore, the E_T of **PmPTPA** is determined to be 2.65 eV. This value is higher than that of the state-of-art blue phosphorescent dopant, FIrpic (2.62 eV), indicating that **PmPTPA** is able to host FIrpic without detrimental triplet energy back transfer.

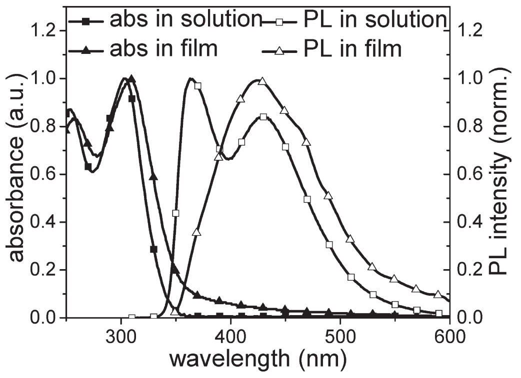


Figure 3. Absorption and fluorescence spectra of **PmPTPA** in solution and in solid film.

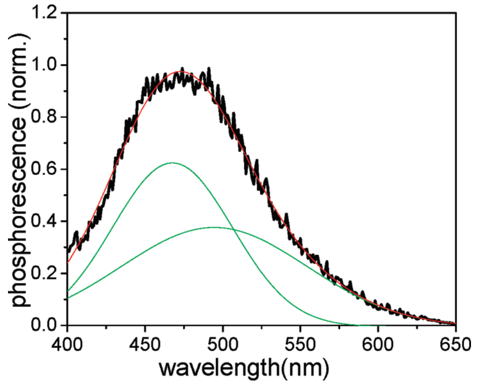


Figure 4. Deconvolution of the phosphorescence spectrum of **PmPTPA** in dilute solution.

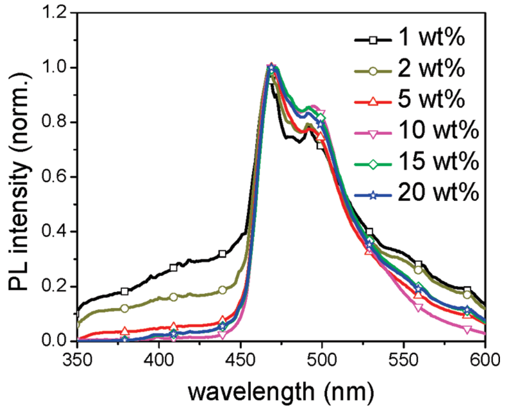


Figure 5. PL spectra of the blend of **PmPTPA** and FIrpic in thin film with specified weight ratios of FIrpic.

Figure 5 shows the photoluminescence (PL) spectra of the blends of **PmPTPA** and FIrpic at different FIrpic weight ratios in solid film. The PL spectra are dominated by FIrpic's emission even at a FIrpic concentration as low as 1 wt %, indicative of efficient energy transfer from **PmPTPA** to FIrpic. The relative intensity of the host emission decreases with increasing FIrpic concentration. When the content of FIrpic is more than 5 wt %, the emission from **PmPTPA** is almost completely quenched, which indicates absence of triplet energy back transfer from FIrpic to **PmPTPA** and is consistent with the higher E_T of **PmPTPA** than that of FIrpic.[23]

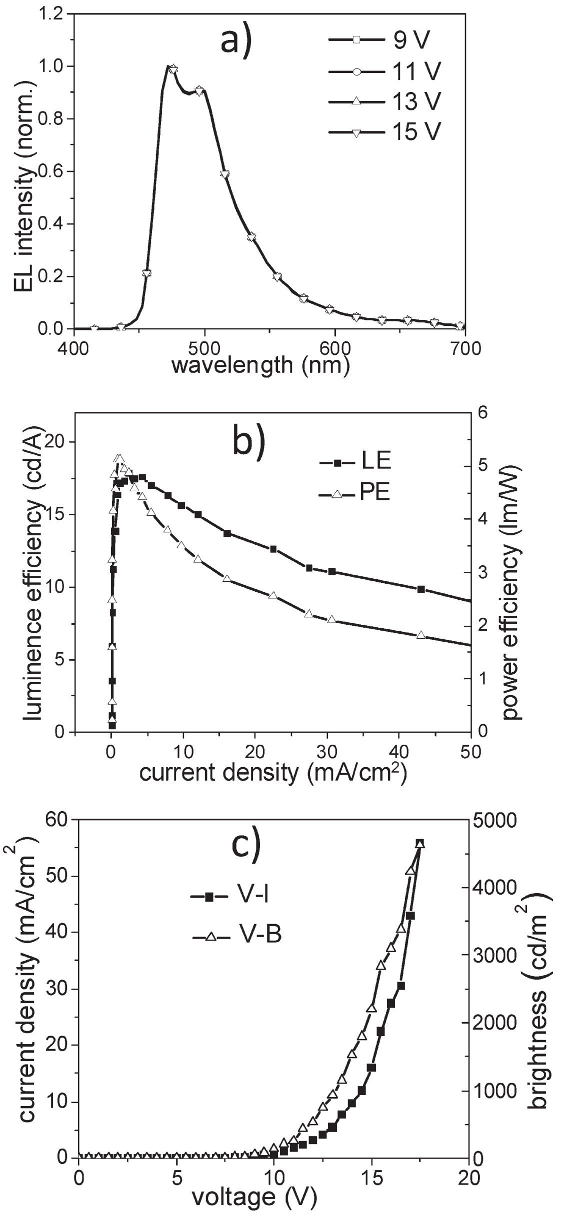


Figure 6. EL spectra (a), current density—luminance efficiency—power efficiency curve (b), and voltage—current density—brightness curve (c) of the blue PPLEDs made from **PmPTPA** and 15 wt % FIrpic.

To evaluate **PmPTPA** as a host material for blue electrophosphorescence, blue PPLEDs were fabricated with the configuration of ITO/PEDOT:PSS (40 nm)/**PmPTPA**: FIrpic (15 wt %, 70 nm)/OXD-7 (30 nm)/CsF (1 nm) /Al (100 nm). OXD-7 was used as an electron transporting layer to boost electron injection. As shown in Figure 6a, the EL spectra of the device exhibit sky-blue emission exclusively from FIrpic and no emission from the polymer host. The absence of the polymer host emission is owing to the energy transfer and charge trapping from **PmPTPA** to FIrpic in the EL process. Figure 6b presents the current density-luminance efficiency—power efficiency characteristic and Figure 6c shows the voltage—current density-brightness curve of the blue PPLEDs. The devices exhibit a turn-on voltage of 6.0 V. At a brightness of 1000 cd/m^2, the luminance efficiency, power efficiency and external quantum efficiency of the device are 17.9 cd/A, 5.2 lm/W, and 9.3%, respectively, with the CIE

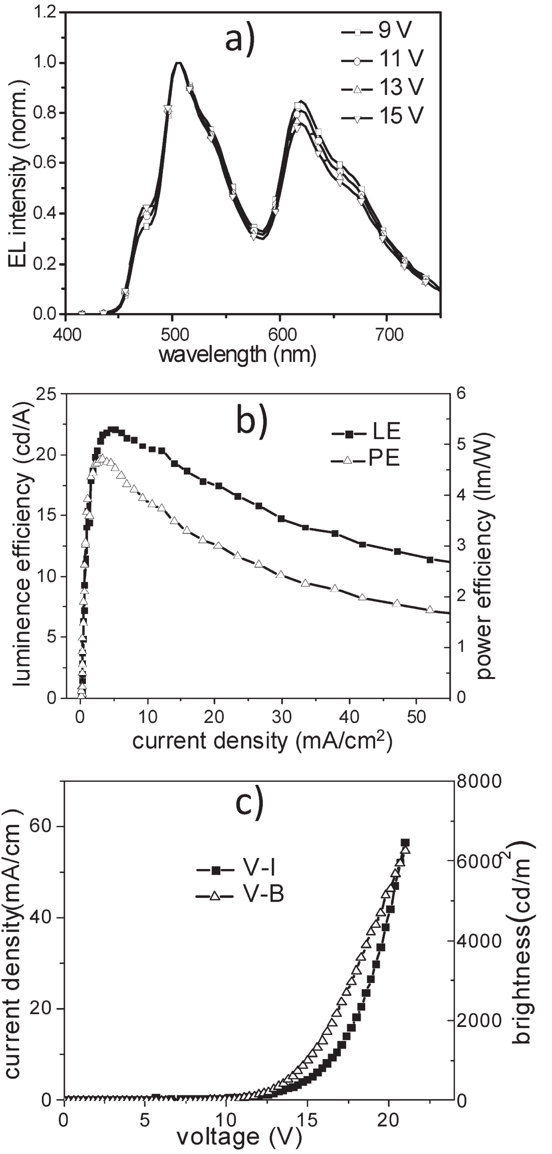


Figure 7. EL spectra (a), current density—luminance efficiency—power efficiency curve (b) and voltage—current density—brightness curve (c) of the white PPLEDs made from **PmPTPA**, 15 wt % FIrpic, 0.2 wt % Ir(mppy)$_3$ and 0.5 wt % Ir(piq)$_3$.

coordinates of (0.16, 0.33). The EL efficiencies are much higher than those of previous blue PPLEDs based on conjugated polymer hosts, such as carbazole-tethering poly(*m*-phenylene) (4.7 cd/A)[24] and poly(3,6-fluorene) (0.4 cd/A).[25] Moreover, the device efficiencies of **PmPTPA** are fairly comparable to those of nonconjugated polymer host PVK (about 15—20 cd/A at practical brightness).[3−7] The high device efficiencies of **PmPTPA** are attributed to two factors. First, the high E$_T$ of **PmPTPA** prevents the detrimental triplet energy back transfer from the dopant to the host. Second, owing to the high-lying HOMO energy level of **PmPTPA**, the hole injection barrier from PEDOT:PSS to **PmPTPA** is small, enhancing hole injection and improving balance of electrons and holes.

On the basis of the successful blue PPLEDs, we fabricated white PPLEDs with **PmPTPA** host. Emissive layer of ideal white PPLEDs involved blue, green, and red phosphorescent dopants dispersed in a polymer host. Here, we selected commercially available FIrpic, Ir(mppy)$_3$, and Ir(piq)$_3$ as the blue, green, and red dopants, respectively. The concentrations of the three phosphorescent dopants were optimized for balanced blue, green and red emission to give white light. As the result, high quality warm white emission was obtained at FIrpic, Ir(mppy)$_3$, and Ir(piq)$_3$ contents of 15, 0.2, and 0.3 wt %, respectively. The EL spectra of the white PPLEDs (see Figure 7a) exhibit three bands at 475, 506, and 619 nm, which are attributed to the emissions from FIrpic, Ir(mppy)$_3$, and Ir(piq)$_3$, respectively. No emission from the host is observed. It is worthy to mention the excellent bias independence of the EL spectra. When the bias is increased from 9 to 15 V, the EL spectra remain almost unchanged (see Figure 7a) with the CIE coordinates vary slightly from (0.398, 0.480) to (0.378, 0.470). The bias independent EL spectra are essential for white PPLEDs in lighting and display applications.[27−29] The white PPLEDs have a turn-on voltage of 6.5 V. At a brightness of 1000 cd/m^2, the device gives a luminance efficiency of 22.1 cd/A, power efficiency of 4.7 lm/W and external quantum efficiency of 10.6%. Figure 7b displays the dependence of luminance efficiency and power efficiency on current density of the white light emitting device and Figure 7c shows the voltage—current density-brightness curve of the device. Similar to the blue PPLEDs, the white PPLEDs based on **PmPTPA** also exhibit efficiencies comparable to those of three-color white PPLEDs using PVK as the host.[19]This result further confirms that **PmPTPA** is an excellent host polymer for electrophosphorescence.

■ CONCLUSION

In summary, we develop a conjugated polymer host by covalently attaching triphenylamine unit to the side chain of poly(*m*-phenylene) backbone. The polymer can efficiently host blue and white electrophosphorescence thanks to its E_T as high as 2.65 eV and high-lying HOMO energy level of −5.35 eV. The blue and white PPLEDs show external quantum efficiency of 9.3% and 10.6%, respectively, which are both fairly comparable to those of nonconjugated polymer hosts. Work is underway to hopefully improve the lifetime of blue and white PPLEDs. The prior work in achieving extremely long lifetime PLEDs using conjugated polymers[30] provides insights and encouragement in developing PPLEDs based on poly(*m*-phenylene) derivative host with variable emission colors, high efficiency, and long lifetime.

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