

Multifunctional electroluminescent material based on dimesitylboron and α -naphthylamino fluorene bridge

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ARTICLE INFO

Article history:

Received 26 May 2011

Received in revised form 26 August 2011

Accepted 28 August 2011

Available online 22 September 2011

Keywords:

Multicolor emission

Naphthylamino

Dimesitylboron

Fluorene bridge

Multifunctional

Electroluminescent device

ABSTRACT

By introducing a triarylamino and three-coordinate boron groups into a fluorene bridge, an asymmetric and bipolar compound, 2-dimesitylboron-7-(*N*-phenyl- α -naphthyl-amino)-9,9-diethylfluorene (BNPEF), was prepared. Light emissions of BNPEF were different in solvents of different polarities. A series of electroluminescent devices was fabricated. Device A ITO/BNPEF/LiF/Al, Device B ITO/NPB/BNPEF/LiF/Al, Devices C and D ITO/NPB/BNPEF/BCP/AlQ₃/LiF/Al, Device E ITO/BNPEF/AlQ₃/LiF/Al and Device F ITO/NPB/BNPEF/AlQ₃/LiF/Al, were fabricated. Device A exhibited a blue emission at 456 nm. Device B showed a green emission at 512 nm with current efficiency of 0.67 cd/A. The blue EL peaks of Devices C and D were at 488 and 484 nm with current efficiencies of 2.92 and 5.03 cd/A, respectively. Devices E and F emitted green lights at 520 and 528 nm with current efficiencies of 4.03 and 2.35 cd/A, respectively. The characteristics of the devices demonstrated that BNPEF was a multifunctional molecule acting as a blue emitting, or hole- or electron-transporting materials.

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1. Introduction

Organic light-emitting devices (OLEDs) typically comprise an electron-transport layer, an emitter and a hole-transport layer [1]. To facilitate charge transport and charge recombination in order to enhance the overall efficiency, a device with multi-layered structures or doped emitter layer is often used [2]. However, this often results in degradation, leading to poor stability of OLEDs. It is therefore desirable to fabricate devices with a single and/or undoped layer to simplify the fabrication process and minimize the problems due to interfacial diffusion. An effective way to reduce the number of layers in a device is to use a multi-functional molecule as both emitter and charge-transport materials. AlQ₃ is a typical bifunctional compound which is a green emitter as well as an electron-transport materials in OLEDs [3,4]. Stable blue emitters remain to be one of the greatest challenges in OLEDs due to the poor stability of the devices, and thus has been the focus of researches and development [5–8]. A recent work has demonstrated a way to improve color stability in a multilayered OLED for a blue primary using a multifunctional material (PAQ5). Its super light-emitting and electron-transporting properties played an important role [9].

Three-coordinate boron compounds have been reported as electron-transport/hole-blocking materials for OLEDs [10–13].

2,2'-Dipyridylamino or 7-azaindolyl functional groups are introduced into three-coordinate boron compounds, which are used as blue-emitter and electron-transport materials [14]. Devices based on Mes₂B(p-4,4'-biphenyl-NPh(1-naphthyl)) (BNPB), a molecule which has having part of a 4,4'-bis(*N*-1-naphthylphenylamino)biphenyl (NPB) molecule and a boron centre, produce a bright blue electroluminescence (EL). A two-layered structure with BNPB for blue and AlQ₃ for green produces white (or bluish white) emission due to dual emission from BNPB and AlQ₃. BNPB serves as a hole-transporting material, AlQ₃ as electron-transporting material and both as light emitting material [15]. A fluorene-bridge compound with a fused-ring system greatly enhanced the thermal stability and charge-transporting capability of the material. Recently fluorene-bridge triarylamines have been widely investigated as hole-transport materials or emitters [16–18].

In this paper triarylamino and three-coordinate boron groups were incorporated into a fluorene bridge in the preparation of an asymmetric and bipolar compound, 2-dimesityl-boron-7-(*N*- α -naphthylphenylamino)-9,9-diethylfluorene (BNPEF). The solvent-dependent emission, thermal stability and electrochemical properties of BNPEF were investigated. Electroluminescent devices having different structures incorporating BNPEF, Device A ITO/BNPEF/LiF/Al, Device B ITO/NPB/BNPEF/LiF/Al, Devices C and D ITO/NPB/BNPEF/BCP/AlQ₃/LiF/Al, Device E ITO/BNPEF/AlQ₃/LiF/Al and Device F ITO/NPB/BNPEF/AlQ₃/LiF/Al were fabricated and characterized.

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2. Experimental

2.1. Materials

Fluorene, *N*-phenyl- α -naphthylamine, *n*-butyllithium (*n*-BuLi, 1.6 M), 18-crown-6, dimesitylboron fluoride ((Mes)₂BF), 4,4'-bis(*N*-1-naphthylphenylamino)biphenyl (NPB), 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP), tris(8-hydroxy-quinoline)aluminum (AlQ₃), LiF and Al wires were purchased from Acros Organics. Chemicals and solvents were used as received in the highest commercially available grade unless described otherwise. Diethyl ether (Et₂O) was refluxed with sodium and distilled. 1,2-Dichlorobenzene and dimethyl sulfoxide (DMSO) were distilled at reduced pressure.

2.2. Synthetic procedures

2.2.1. 9,9-Diethylfluorene (**1**) [19], 2,7-diiodo-9,9-diethylfluorene (**2**) [20], 2-iodo-7-(*N*- α -naphthylphenylamino)-9,9-diethylfluorene (**3**) [20,21]

These were synthesized using methods described in the literatures with modifications.

2.2.2. 2-Dimesitylboron-7-(*N*-phenyl- α -naphthylamino)-9,9-diethylfluorene (BNPEF) [15,21]

A mixture of 2-iodo-7-(*N*-phenyl- α -naphthylamino)-9,9-diethylfluorene (0.28 g, 0.5 mmol) in Et₂O (30 ml) was purged with nitrogen, then *n*-BuLi (1.6 M, 0.55 mmol, 0.35 ml) in hexane was added at -78°C in one portion. After stirring for 1.5 h, a solution of dimesitylboron fluoride (0.17 g, 0.55 mmol) in Et₂O (10 ml) was added dropwise to the above reaction flask. The mixture was stirring for another 1 h at -78°C . It was then allowed to cool slowly to room temperature and kept stirring overnight. The solvent was removed under reduced pressure. The crude product was purified by column chromatography on silica gel using light petroleum/dichloromethane (v/v, 10/1) as eluant, a green-yellow solid (0.22 g) was obtained (yield, 63%).

IR (KBr pellet, cm⁻¹): 2960, 2916, 1595, 1573, 1492, 1461, 1419, 1392, 1376, 1338, 1288, 1271, 1240, 1213, 1193, 1168, 1079, 1027, 1020, 962, 937, 910, 890, 881, 848, 820, 767, 744, 715, 694, 681, 654, 647.

¹H NMR (DCCl₃, 400 MHz): δ = 0.24–0.27 (t, 6H), 1.74–1.77 (m, 2H), 1.83–1.86 (m, 2H), 2.01 (s, 12H), 2.31 (s, 6H), 6.81 (s, 4H), 6.89–6.98 (m, 2H), 6.99–7.03 (d, 1H), 7.04–7.09 (d, 2H), 7.17–7.24 (t, 2H), 7.29–7.33 (t, 2H), 7.40–7.48 (m, 4H), 7.51–7.54 (m, 2H), 7.75–7.77 (d, 1H), 7.86–7.91 (m, 2H).

MS (CH₃OH, molecular weight: 687.7) *M/e* (%): 688.4 (*M*⁺+1). Elemental analysis: calcd. for C₅₁H₅₀BN: C, 89.06; H, 7.33; N, 2.04; found C, 88.93; H, 7.21; N, 1.90.

2.3. Instruments

The synthetic compounds were characterized by elemental analysis (Carlo Erba 1106), FT-IR (Shimadzu FT-IR 8400), and ¹H NMR spectroscopy (Bruker DMX-300), respectively. Mass spectrometry analysis was performed on a Shimadzu LC-MS 2010A. Cyclic voltammetry (CV) analyses were carried out by using a Zahner Zennium. Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were performed on a Netzsch DSC-200PC and a Netzsch TG-209. The UV-vis absorption spectrum was measured by using a GBC Cintra 303 spectrometer. The photoluminescent (PL) spectrum was recorded with a Perkin Elmer LS-55 luminescence spectrometer.

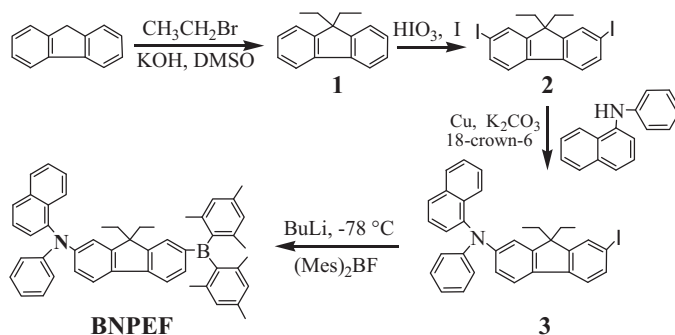


Fig. 1. A schematic diagram for chemical structure and synthetic route of BNPEF. Reagents and conditions are: (i) CH₃CH₂Br, KOH, DMSO. Cold water bath, over 2.5 h; (ii) H₂SO₄, HIO₃, I₂, 85 °C; (iii) activated Cu powder, 18-crown-6, K₂CO₃, *N*-phenyl- α -naphthylamine, 200 °C; and (iv) *n*-BuLi, -78°C , (Mes)₂BF.

2.4. Device fabrication and testing

Indium tin oxide (ITO) glass substrate with a sheet resistance of 25 Ω /square was (1) cleaned with detergents, deionized water, ethanol, and isopropanol in an ultrasonic bath, (2) dried under nitrogen flow, and (3) treated with oxygen plasma (Emitech K1050X) for eight minutes before use. A series of devices were fabricated by sequential vacuum deposition of organic materials, LiF and Al on pre-treated ITO with device structures as follows: Device A ITO/BNPEF/LiF/Al, Device B ITO/NPB/BNPEF/LiF/Al, Devices C and D ITO/NPB/BNPEF/BCP/AlQ₃/LiF/Al, Device E ITO/BNPEF/AlQ₃/LiF/Al and Device F ITO/NPB/BNPEF/AlQ₃/LiF/Al, where NPB was used as a hole-transporting and electron-blocking layer, and BCP as an electron-transporting and hole-blocking layer. AlQ₃ served as an electron-transporting layer or emitter, and LiF/Al as a complex cathode. BNPEF was used as a multifunctional material.

The luminance–current–voltage characteristics were measured by a computer controlled Keithley 2420 source meter unit with a Minolta LS 110 luminescence meter. EL spectra and Commission Internationale de l'Eclairage (CIE) coordinates were recorded by using a SpectraScan PR 655 photometer (Photo Research). The measurements of the devices were performed at room temperature under ambient atmosphere.

3. Results and discussion

3.1. Synthesis and characterization

A schematic diagram of the chemical structure and synthetic route of BNPEF is outlined in Fig. 1. The intermediate 2,7-diiodo-9,9-diethylfluorene (**2**) was prepared by reacting of 9,9-diethylfluorene (**1**) in the presence of H₂SO₄, HIO₃ and I₂ at 85 °C. Then **2** was reacted with *N*-phenyl- α -naphthylamine in the equivalent molar ratio to produce asymmetric 2-iodo-7-(*N*-phenyl- α -naphthylamino)-9,9-diethylfluorene (**3**) via a copper-activated Ullmann coupling. The target compound 2-dimesitylboron-7-(*N*-phenyl- α -naphthylamino)-9,9-diethylfluorene (BNPEF) was synthesized by reacting **3** with *n*-BuLi and (Mes)₂BF at -78°C with a high yield.

3.2. Thermal properties

The thermal properties of BNPEF were determined by differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) under a nitrogen atmosphere, both at a heating rate of 10 °C/min as shown in Fig. 2. After a fast quenching process, a relatively high glass transition temperature (*T*_g) at 126 °C and a melting point at 226 °C were observed. From the TGA curve, it can be seen

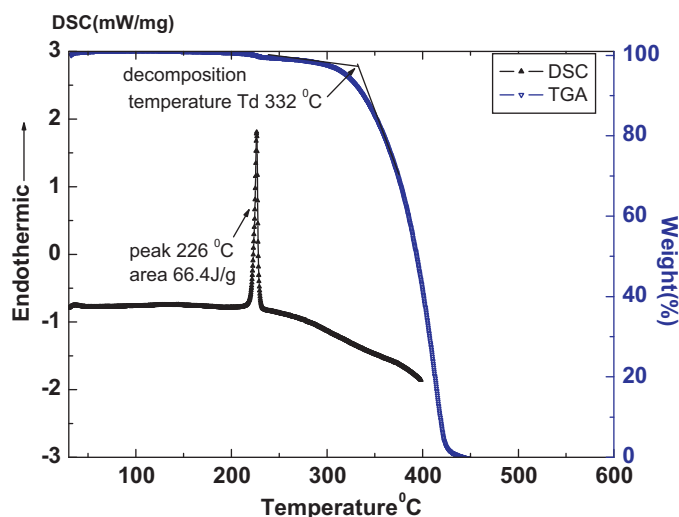


Fig. 2. DSC and TGA curves of BNPEF.

that the BNPEF was stable up to 300 °C with a thermal decomposition temperature (T_d) of around 332 °C.

In combination with its solubility, BNPEF was suitable for thin film preparation by either a solution casting or a vacuum evaporation for OLED application.

3.3. Photophysical properties

UV-vis absorption and photoluminescence (PL) spectra of BNPEF in various solvents and solid film are shown in Fig. 3. It can be observed that the shapes of UV-vis absorption spectra are similar. The main absorption at long wavelength range peaked at about 403 nm in solvents of relatively weak polarities, such as hexane, dioxane, toluene, and tetrahydrofuran and at 397 nm in solvents of relatively strong polarities (methanol and methyl cyanide). In the short wavelength range, absorption peaked at about 287 nm, for a wide range of solvents of different polarities.

Photoluminescence from BNPEF is bright and strongly solvent-dependent. Emission peaks are found at 458 nm in solid film, 422 nm in non-polar hexane, 443 nm in toluene, 450 nm in dioxane, 470 nm in tetrahydrofuran, 480 nm in methanol and 507 nm in methyl cyanide. As solvent polarities were increased, the PL emissions from BNPEF shifted to red, that is, from blue to green. Solvent-dependent emission had been frequently observed for three-coordinate organoboron compounds and attributed to the presence of a highly polarized excited state [15].

3.4. Electrochemical properties

The energy levels, that is, the highest occupied molecular orbit (HOMO) and the lowest unoccupied molecular orbit (LUMO), of BNPEF were estimated from redox properties measured by cyclic voltammetry (CV) (Fig. 4), using an Ag/AgCl electrode as the reference electrode, a dichloromethane solution as a medium in the presence of *tetra-n*-butylammonium hexafluorophosphate (TBAPF₄) (0.1 M) as the supporting electrolyte with a BNPEF concentration of 1×10^{-3} M and a scanning rate of 50 mV/s. The onset oxidation potential (E_{onset}) of BNPEF was measured from the first anodic oxidation wave at 0.81 eV. The HOMO energy level (E_{HOMO}) was calculated according to the following equation: $E_{\text{HOMO}} = -(E_{\text{onset}} + 4.44)$ eV, to be -5.25 eV for BNPEF. From the spectroscopy absorption edge ($\lambda_{\text{a,edge}}$, 446 nm) the value of the energy gap (E_g , eV) of BNPEF was found to be 2.78 eV. So

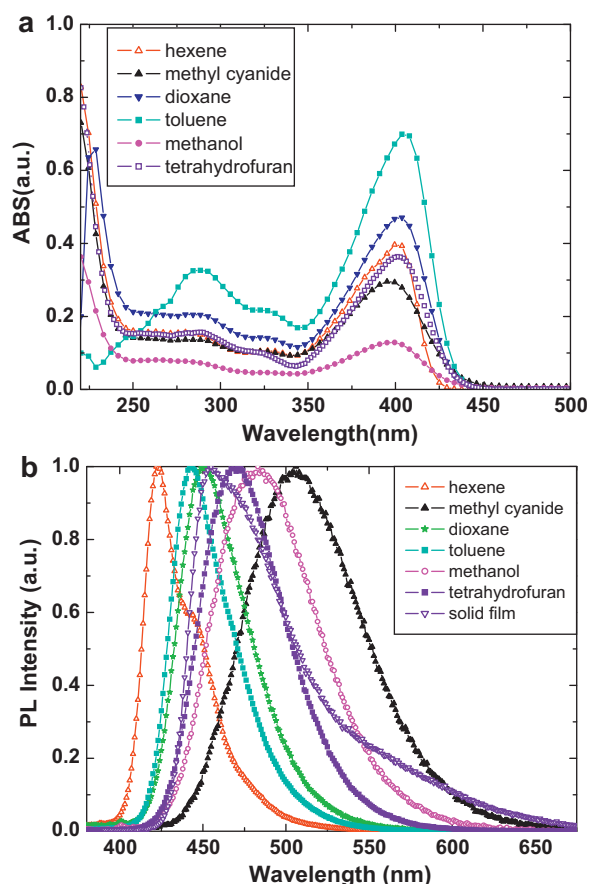


Fig. 3. (a) UV-vis absorption and (b) emission spectra of BNPEF in different solvents.

the LUMO energy level (E_{LUMO}) was calculated from the equation: $E_{\text{LUMO}} = E_{\text{HOMO}} + E_g = -2.47$ eV [22].

Under the same experimental conditions two reversible oxidation peaks for NPB were found at 0.76 and 1.03 eV, which may be attributed to the sequential oxidation of two triaryl amino groups. Having only one triaryl amino group in the molecular structure, a only single reversible oxidation peak was found for BNPB (Mes₂B(*p*-4,4'-biphenyl-NPh(1-naphthyl))) or BNPEF. The oxidation potential of BNPB was higher than the first oxidation potential of NPB. The reversible oxidation potential of BNPEF in CH₂Cl₂ was 0.86 eV (vs Ag/AgCl), while that of BNPB was 1.03 eV. E_{HOMO} , E_{LUMO} and E_g of BNPB were reported at -5.30 , -2.44 and 2.86 eV [15]. In comparison with NPB and BNPB, the planar fluorene core in the BNPEF

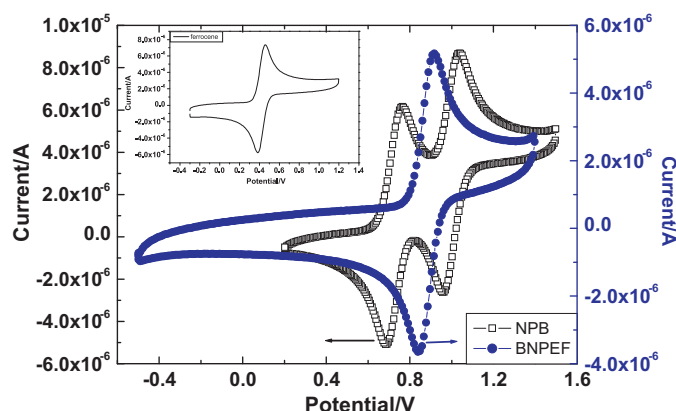


Fig. 4. Cyclic voltammograms of BNPEF and NPB (ferrocene shown in the inset).

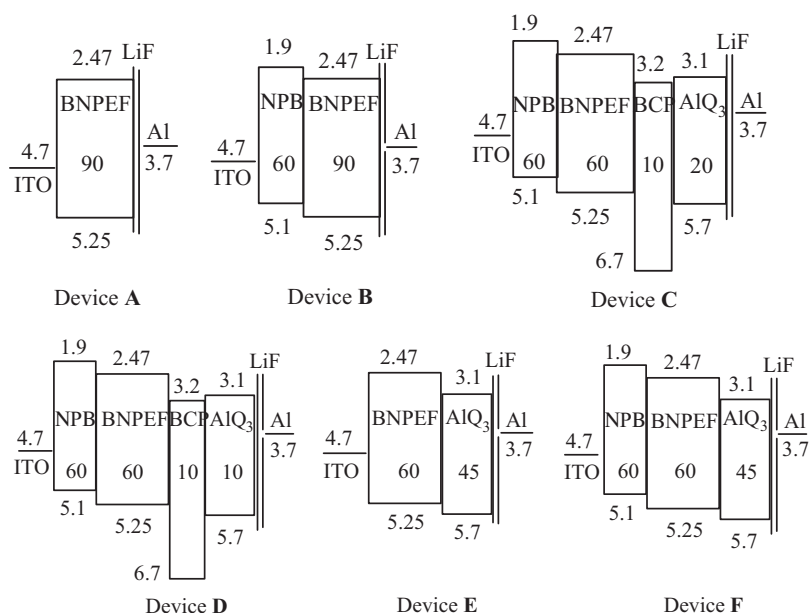


Fig. 5. The configurations and energy level diagrams of the devices.

structure enhanced molecular conjugation and resulted in a red shift in the absorption spectrum, which played an important role in reducing E_g (2.78 eV). The LUMO energy levels of BNBP (−2.44 eV) and BNPEF (−2.47 eV) were higher than that of AlQ₃ (−3.1 eV), but three-coordinate boron compounds were demonstrated to be good electron-transport materials, possibly due to the empty *p*-orbital on the boron centre.

3.5. Electroluminescent devices using BNPEF

In order to evaluate the use of BNPEF as a multifunctional compound in OLED application, a single-layer Device A, two double-layer Devices B and E, a three-layer Device F, and two four-layer Devices C and D were fabricated. Configurations and energy level diagrams of Devices A, B, C, D, E and F are shown in Fig. 5. EL spectra and CIE coordinates of the devices are presented in Fig. 6.

Current density–voltage and luminance–voltage characteristics of the devices were measured as shown in Fig. 7. At a fixed current density of 20 mA/cm², the corresponding driving voltages of Device E (13.5 V) and Device F (13.5 V) were obviously lower than those of Device B (24.1 V), Device C (20.1 V) and Device D (21.9 V).

The current efficiency or luminance efficiency (LE) and power efficiency (PE) vs current density characteristics of the devices are presented in Fig. 8.

Device A ITO/BNPEF (90 nm)/LiF (0.5 nm)/Al (120 nm) was fabricated by vacuum evaporation, which exhibited a blue emission at 456 nm near to the PL emission (458 nm) of BNPEF in solid film. Maximum luminance was measured as 11 cd/m² (17 V), and the CIE coordinate was located at (0.245, 0.256). These indicated that BNPEF was capable of being a multifunctional material acting as an emitter, and hole-transporting or electron-transporting materials, which may be attributed to the dimethylboron group acting as an electron acceptor and the triaryl amino group as an electron donor in BNPEF. So BNBP and BNPEF can also be used as electron-transporting materials in OLEDs. Device A showed lower luminance, instability and poor efficiency due to the poor matching of energy levels and poor interfacial stability between BNPEF and electrodes (cathode and anode).

To improve the luminance and efficiency, two double-layer devices, Device B ITO/NPB (60 nm)/BNPEF (90 nm)/LiF (0.5 nm)/Al

(120 nm) and Device E ITO/BNPEF (60 nm)/AlQ₃ (45 nm)/LiF (0.5 nm)/Al (120 nm) were fabricated, including either an additional hole injection layer (NPB) or an electron injection layer (AlQ₃), respectively. However, the electroluminescence from Device B is a green emission centred at 512 nm with a CIE coordinate of (0.283, 0.398). It was unexpected that Device B emitted a different EL spectrum from Device A, which may arise from the formation of an interface state of NPB/BNPEF. The luminance of

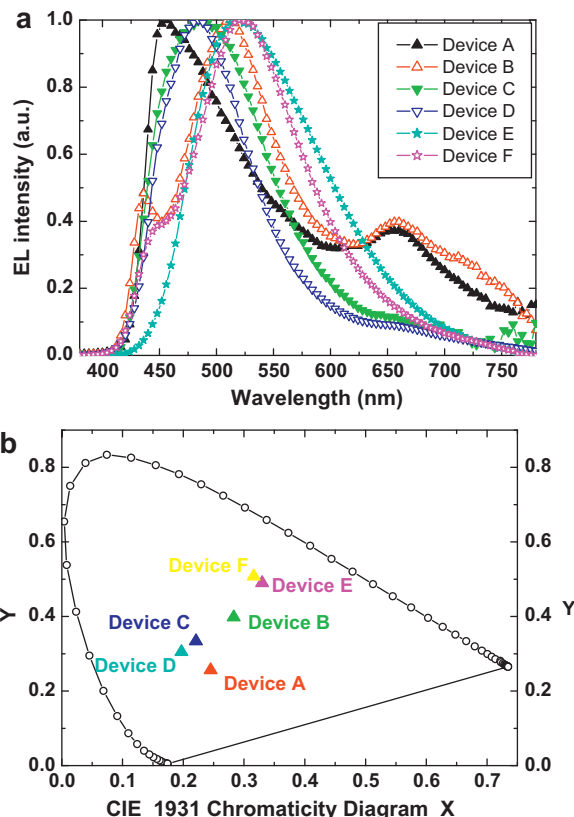


Fig. 6. (a) EL spectra and (b) CIE coordinates of the devices.

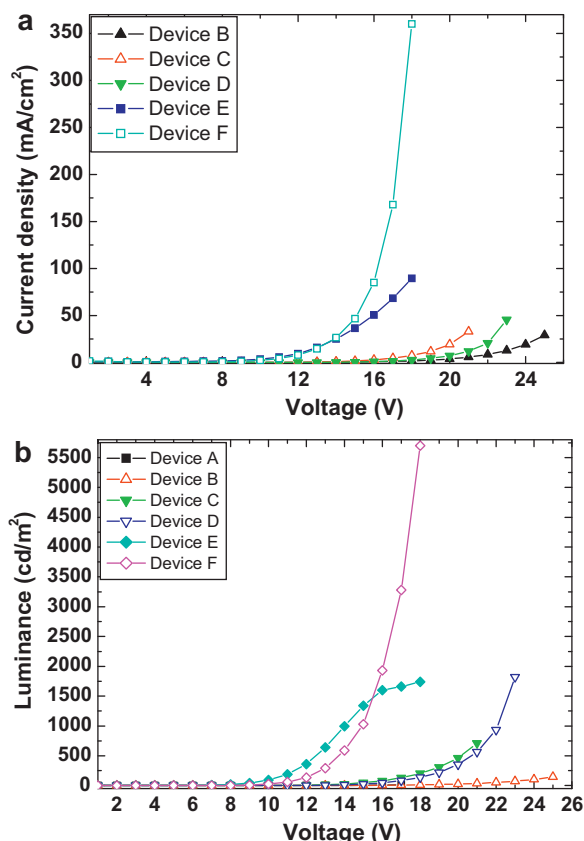


Fig. 7. (a) Current density–voltage and (b) luminance–voltage characteristics of the devices.

the Device B was measured as 144 cd/m^2 at 25 V and the current efficiency was 0.67 cd/A .

Device E showed a green light at 520 nm and CIE coordinate of (0.330, 0.490), which mainly originated from the emission of AlQ_3 . The luminance and current efficiency were measured to be 1739 cd/m^2 (18 V), and 4.03 cd/A , respectively. The turn-on voltage ($V_{\text{turn-on}}$, 5.4 V) for Device E was the lowest one among these devices. The efficient emission and low $V_{\text{turn-on}}$ for Device E were mainly due to the fact that BNPEF showed efficient hole injection and transport from the anode and that AlQ_3 enhanced electron injection and transport from the cathode compared to Devices A and B. But the current or power efficiency of Device E decreased dramatically when the current density was increased from 20 to 100 mA/cm^2 .

Based on the above findings regarding Devices B and E, a three-layer device, Device F ITO/NPB (60 nm)/BNPEF (60 nm)/ AlQ_3 (45 nm)/LiF (0.5 nm)/Al (120 nm) was fabricated with both NPB as a hole-transporting and electron-blocking layer and AlQ_3 as an electron injection layer. Device F also exhibited a green emission from AlQ_3 centred at 528 nm with a CIE coordinate of (0.316, 0.508). The luminance and current efficiency were 5710 cd/m^2 (at 18 V) and 2.35 cd/A , respectively. $V_{\text{turn-on}}$ was 7.5 V. The current or power efficiency remained relatively constant within a wide range of current densities of between 20 and 100 mA/cm^2 .

Considering that E_{HOMO} of BNPEF (-5.25 eV) was much higher than that of BCP (-6.7 eV) and that E_{LUMO} of BNPEF (-2.47 eV) was located between those of NPB (-1.9 eV) and BCP (-3.2 eV), it is expected that there will be barriers to the migration of holes from BNPEF to BCP and to the movement of electrons from BNPEF to NPB. This structure may confine the electrons and holes within the BNPEF emitting layer and improve recombination [23]. A four-layer

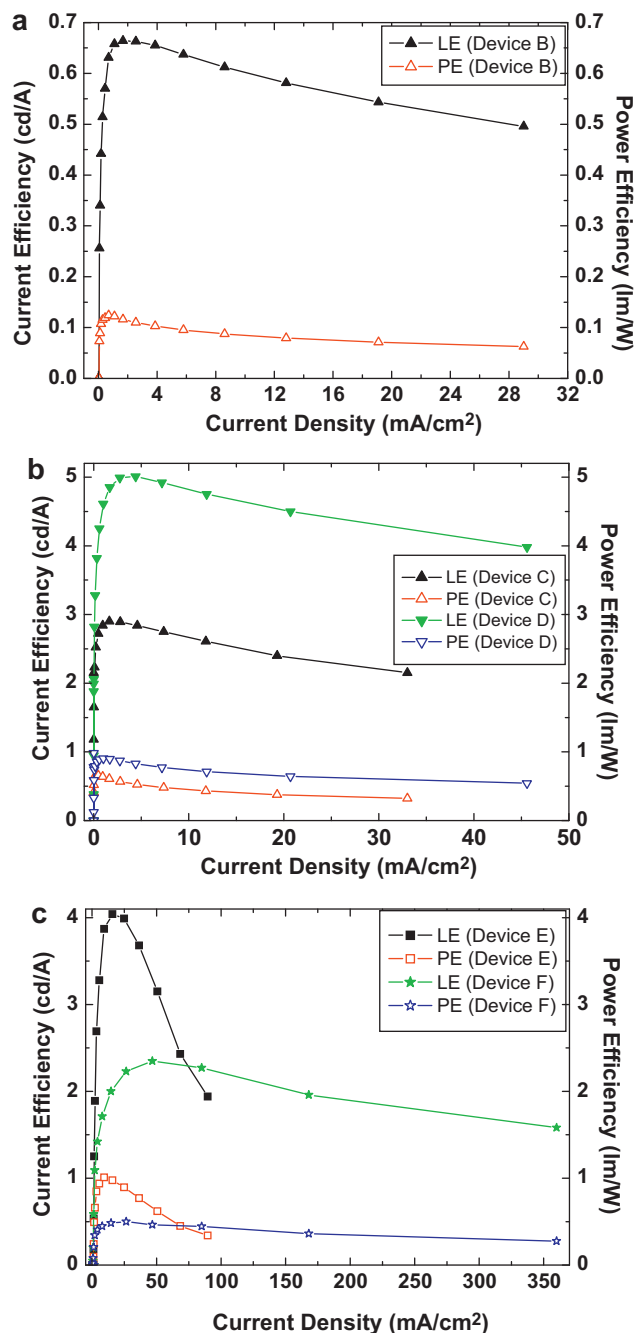


Fig. 8. Current efficiency and power efficiency vs current density curves of Devices B, C, D, E and F.

device, Device C ITO/NPB (60 nm)/BNPEF (60 nm)/BCP (10 nm)/ AlQ_3 (20 nm)/LiF (0.5 nm)/Al (120 nm), was therefore designed. In this device, a blue EL spectrum was obtained which was centred at 488 nm with a CIE coordinate of (0.221, 0.334). The maximum current efficiency and power efficiency of Device C were 2.92 cd/A and 0.71 lm/W . Luminance was measured to be 712 cd/m^2 at 21 V. The $V_{\text{turn-on}}$ was 10.3 V.

In this device, excellent hole injection was achieved due to a close match of E_{HOMO} of BNPEF with that of NPB. The thickness of the AlQ_3 layer was reduced to promote electron mobility to the emitting layer in a further optimization of the device. Device D ITO/NPB (60 nm)/BNPEF (60 nm)/BCP (10 nm)/ AlQ_3 (10 nm)/LiF (0.5 nm)/Al (120 nm) was prepared. Device D also showed a blue emission centred at 484 nm with a CIE coordinate of (0.197, 0.305).

Table 1

EL performances of Devices A, B, C, D, E and F.

Devices	L (cd/m ²) (V) ^a	LE (cd/A) ^a	PE (lm/W) ^a	$V_{\text{turn-on}}$ (V) ^b	EL λ_{em} (nm) (V) ^a	CIE (x,y) ^c
A	11 (17)			12.1	456 (19)	0.245, 0.256
B	144 (25)	0.67	0.13	13.4	512 (20)	0.283, 0.398
C	712 (21)	2.92	0.71	10.3	488 (16)	0.221, 0.334
D	1813 (23)	5.03	0.91	11.2	484 (20)	0.197, 0.305
E	1739 (18)	4.03	1.01	5.4	520 (13)	0.330, 0.490
F	5710 (18)	2.35	0.51	7.5	528 (15)	0.316, 0.508

^a The data for luminance (L), current efficiency (LE), power efficiency (PE) and electro-luminescent wavelength (EL λ_{em}) were the maximum values of the devices.^b Voltage needed for brightness of 1 cd/m².^c The voltages for CIE coordinates were 19 V for Device A, 20 V for Device B, 16 V for Device C, 20 V for Device D, 13 V for Device E and 15 V for Device F, respectively.

A current efficiency of 5.03 cd/A was measured with a luminance of 1813 cd/m² at 23 V. Device D showed an improved luminance and efficiency. The full width at half maximum (FWHM) of the EL spectrum from Device D became narrower than that of Device C. In both Device C and Device D, the recombination of holes and electrons was located in the BNPEF emitting layer. Therefore the EL emissions are similar to the singlet excited state of BNPEF. The EL emissions of Devices C (at 488 nm) and D (at 480 nm) appeared to be red-shifted compared to that of Device A. The EL performances of Devices A, B, C, D, E and F are summarized in Table 1.

4. Conclusions

In this work, an asymmetric and bipolar compound 2-dimethylboron-7-(*N*-phenyl, α -naphthylamino)-9,9-diethylfluorene (BNPEF) was synthesized. The photoluminescence of BNPEF depended strongly on the polarities of solvents. The single-layer Device A, double-layer Devices B and E, three-layer Device F, and four-layer Devices C and D were fabricated. Device A exhibited a blue emission at 456 nm. Device B showed a green emission at 512 nm with a current efficiency of 0.67 cd/A. The blue EL spectra of Devices C and D were centred at 488 and 484 nm and their current efficiencies were 2.92 and 5.03 cd/A, respectively. Both Device E and Device F emitted in green at 520 and 528 nm with current efficiencies of 4.03 and 2.35 cd/A, respectively. The characteristics of the devices demonstrated that BNPEF is a multifunctional optoelectronic material and can be used as a blue emitter and hole- or electron-transporting materials.

Acknowledgements

This work was funded by the Grants from NSFC (60825407, 20674004, 21174016), ISTCP (2008 DFA61420), the Beijing Commission of Education (KM201010015007), Beijing Municipal

Science & Technology Commission (Z090803044009001), and the 111 Project (B08002).

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