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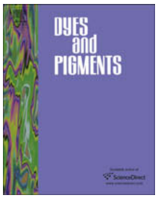

Blue organic light-emitting diodes using novel spiro[fluorene-benzofluorene]-type host materials

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ABSTRACT

Deep blue colored, fluorescent, spiro-type host materials, 5-[4-(1-naphthyl)phenyl]-spiro[fluorene-7,9′-benzofluorene] and 5,9-bis[4-(1-naphthyl)phenyl]-spiro[fluorene-7,9′-benzofluorene] were designed and successfully prepared by the Suzuki reaction. The electroluminescence characteristics of the two compounds as blue host materials doped with blue dopant materials, diphenyl[4-(2-[1,1;4,1]terphenyl-4-yl-vinyl)phenyl]amine and 1,6-bis[(*p*-trimethylsilylphenyl)amino]pyrene (SPP) were evaluated. The device used comprised ITO/*N*,*N*′-bis-[4-(di-*m*-tolylamino)phenyl]-*N*,*N*′-diphenylbiphenyl-4,4′-diamine)/bis[*N*-(1-naphthyl)-N-phenyl]benzidine/Host:5% dopant/tris(8-hydroxyquinolinato)aluminium/Al–LiF. The device obtained from 5-[4-(1-naphthyl)phenyl]-spiro[fluorene-7,9′-benzofluorene] doped with 1,6-bis[(*p*-trimethylsilylphenyl)amino]pyrene displayed high color purity (0.138, 0.138) and high efficiency (3.70 cd/A at 7 V).

1. Introduction

Organic light-emitting diodes (OLEDs) are the subject of intense research owing to their promising full-color display application. Although a large number of valuable red and green emitters that satisfy the requirements for OLEDs have been developed, efficient and stable blue emitters are still rare and have been the subject of considerable interest [1,2]. Major focus has been on various material systems, including anthracene [3–5], distyrylarylene [6–11], fluorene [12,13] and tetra(phenyl)silyl derivatives [14,15].

Spiro compounds with specific steric configurations have attracted attention as organic functional materials on account of their specific physical properties. An important class of spiro compounds with high glass transition temperatures was recently developed for use as optoelectric materials [16]. In particular, the most recent research into blue light-emitting materials has centered on spiro-based derivatives on account of their high solution and solid state photoluminescence quantum yield. Evidence suggests that amorphous, thin film OLEDs which have a high glass transition temperature (T_g) are less vulnerable to heat damage and, hence, are more stable during use. Thus, high T_g materials are always desirable in OLED applications. Spirobifluorenes can be functionalized easily with a variety of substituents as blue host and dopant materials, such as aryl, alkyl and diarylamine. Many attempts have been made to develop new amorphous spirobifluorenes with high morphological stability. The introduction of the spiro linkage has resulted in the generation of spiro-type host and dopant materials with higher T_g values [5,17–20]. Recently, spiro[fluorene-benzofluorene] derivatives with various aromatic and aromatic amine groups on the 5-position were synthesized and their electroluminescence (EL) characteristics were investigated as both host and dopant materials for blue OLEDs [21–24].

This paper reports the synthesis two new deep blue-emitting host materials consisting of 5-[4-(1-naphthyl)phenyl]-spiro[fluorene-7,9′-benzofluorene] (BH–1PN) and 5,9-bis[4-(1-naphthyl)phenyl]-spiro[fluorene-7,9′-benzofluorene] (BH–1DPN) along with their characterization using the following techniques: ^{1}H nuclear magnetic resonance (NMR), ^{13}C NMR, Fourier transform infrared (FT-IR), Mass spectroscopy, thermal analysis, and ultraviolet (UV)–visible and photoluminescence (PL) spectroscopy. The EL properties of multilayered OLEDs fabricated using a BH–1PN and BH–1DPN host and BD-1 and SPP as the dopant were evaluated.

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2. Experimental

2.1. Materials and measurements

Tetrakis(triphenylphosphine)palladium(0) and bromine (Aldrich Chem. Co.) were used without further purification. 4-(Naphthalene-1-yl)phenylboronic acid and 5-[4-(1-naphthyl)phenyl]-spiro[fluorene-7,9′-benzofluorene] (BH–1PN) were prepared using the method reported elsewhere [25]. Tetrahydrofuran and toluene were distilled over sodium and calcium hydride. Diphenyl-[4-(2-[1,1;4,1]terphenyl-4-yl-vinyl)-phenyl]-amine (BD-1, band gap, 2.87 eV; HOMO 5.44 eV; λ_{max} (Absorption) = 383 nm; λ_{max} (Emission) = 472 nm) [6] and 1,6-bis[(*p*-trimethylsilylphenyl)amino]pyrene (SPP; band gap, 2.73 eV; HOMO 5.47 eV; T_{sub}, 340 °C; λ_{max} (Absorption) = 426 nm; λ_{max} (Emission) = 460 nm) [26] were used as dopant materials. 5-Bromo-spiro[fluorene-7,9′-benzofluorene] (**1**) was prepared using a previously reported method [21].

The FT-IR spectra were obtained using a Biorad Excaliber FTS-3000MX spectrophotometer, and the ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker, Avance 500 (500 MHz for ^{1}H and 125 MHz for ^{13}C) spectrometer. The photoluminescence (PL) spectra were recorded on a fluorescence spectrophotometer (Jasco FP-6500) and the UV–vis spectra were obtained using a UV–vis spectrophotometer (Shimadzu, UV-1601 PC). Elemental analyses were performed using a CE Instrument (EA1110), and the differential scanning calorimetry (DSC) measurements were performed using a Mettler DSC 822^e under nitrogen at a heating rate of 10 °C/min. The low and high resolution mass spectra were recorded using a mass spectrometer (JEOL, JMS-AX505WA) in FAB mode. The energy levels were measured using a low-energy photoelectron spectrometer (Riken-Keiki AC-2). The current–voltage characteristics of the encapsulated devices were measured using a programmable electrometer with current and voltage sources (Keithley 237 model). The luminance and EL spectra were measured using a PR650 system (Photo Research Co. Ltd.).

2.2. Synthesis of 5,9-dibromo-spiro[fluorene-7,9′-benzofluorene] (2)

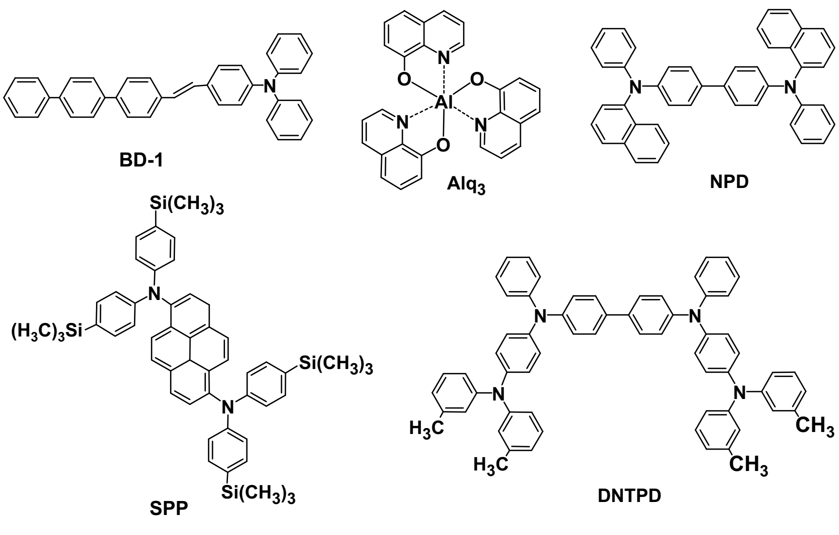
Compound **1** (4.43 g, 10 mmol) was dissolved in chloroform (100 mL) in a two-necked flask. Bromine (1.6 g, 0.2 mmol) was then added dropwise over a period of 20 min. The mixture was stirred at room temperature for 24 h. The precipitated solid was filtered and dried in vacuo to give the crude product, which was then purified by recrystallization from acetonitrile to give a white powder.

Yield 76%. Mp 243 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.78–8.76 (d, 1H, Ar–CH–benzene), 8.42–8.40 (d, 1H, Ar–CH–naphthalene), 8.35–8.33 (d, 1H, Ar–CH–benzene), 7.88–7.87 (s, 1H, Ar–CH–benzene), 7.75–7.72 (t, 2H, Ar–CH–fluorene), 7.65–7.63 (t, 2H, Ar–CH–fluorene), 7.47–7.44 (t, 2H, Ar–CH–fluorene), 7.41–7.37(t, 2H, Ar–CH–fluorene), 7.17–7.15 (d, 1H, Ar–CH–benzene), 6.89–6.87 (d, 1H, naphthalene–CH–Br), 6.69–6.67 (d, 1H, Ar–CH–Br), 6.71–6.69 (d, 1H, Ar–CH–benzene). FT-IR (KBr, cm^{-1}) 3057, 3040, 3012 (aromatic C–H), 745 (aromatic C–Br). MS (FAB) *m/z* 523.96 [(M+1)$^+$]. UV–vis (THF): λ_{max} (Absorption) = 356 nm, λ_{max} (Emission) = 422 nm.

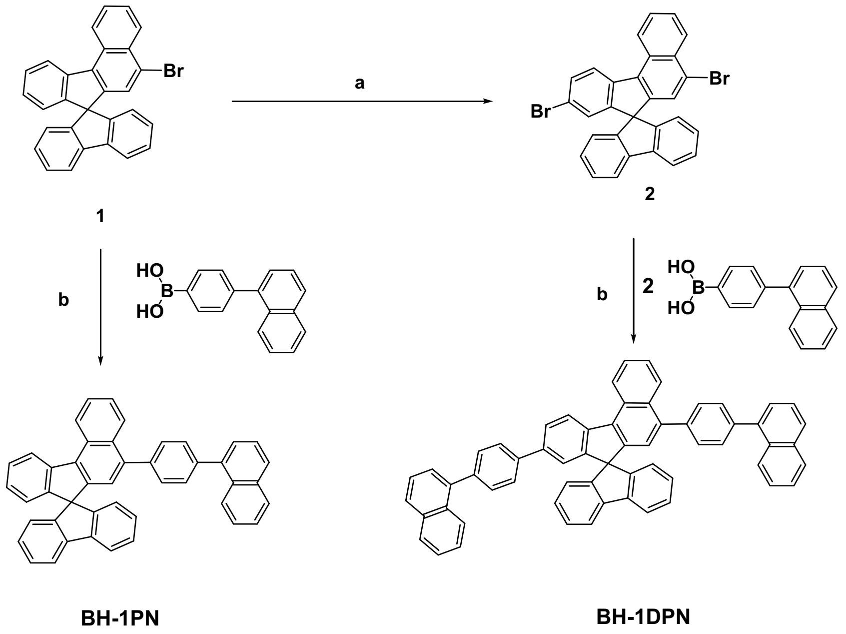
2.3. Synthesis of 5,9-bis[4-(1-naphthyl)phenyl]-spiro[fluorene-7,9′-benzofluorene] (BH–1PN)

Compound **2** (5.23 g, 10 mmol), 4-(naphthalene-1-yl)phenylboronic acid (4.87 g, 20 mmol) and tetrakis(triphenyl-phosphine)palladium(0) (0.81 g, 0.70 mmol) were dissolved in THF (150 mL) in a two-necked flask with constant stirring under nitrogen for 1 h. A solution of potassium carbonate (2 M, 100 mL) was added dropwise to the above reaction mixture over a period of 30 min, and the reaction mixture was heated under reflux for 24 h in a nitrogen atmosphere. After cooling to ambient temperature, the reaction mixture was extracted with methylene chloride and water. The organic layer was evaporated using a rotary evaporator, and the residue was subjected to column chromatography using methylene chloride/*n*-hexane (1/3) as the eluant. A yellow powdery product was obtained.

Yield: 65%. Mp 371.8 °C. ^{1}H NMR (500 MHz; $CDCl_3$) δ 9.03–9.01 (d, J = 8.43 Hz, 1H, Ar–CH–benzene), δ 8.60–8.58 (d, J = 8.19 Hz, 1H, Ar–CH–benzene), δ 8.19–8.17 (d, J = 8.44 Hz, 1H, Ar–CH–benzene), δ 7.97–7.95 (d, J = 8.40 Hz, 1H, Ar–CH–benzene), δ 7.90–7.88 (t, 4H, Ar–CH–naphthalene), δ 7.86–7.83 (t, 4H, Ar–CH–naphthalene), δ 7.81–7.78 (t, 1H, Ar–CH–benzene), δ 7.62–7.60 (d, 1H, Ar–CH–benzene), δ 7.59–7.57 (d, 2H, Ar–CH–fluorene), δ 7.54–7.51 (d, 4H, Ar–CH–benzene), δ 7.50–7.47 (d, 4H, Ar–CH–benzene), δ 7.47–7.45 (d, 4H, Ar–CH–naphthalene), δ 7.43–7.40 (d, 2H, Ar–CH–naphthalene), δ 7.40–7.39 (d, 2H, Ar–CH–fluorene), δ 7.16–7.14 (d, 2H, Ar–CH–fluorene), δ 7.14–7.13 (d, 1H, Ar–CH–benzene), δ 6.89–6.88 (d, 2H, Ar–CH–fluorene), δ 6.88 (d, 1H, Ar–CH–benzene). ^{13}C NMR ($CDCl_3$) δ 150.6, 147.9, 147.5, 142.2, 142.1, 140.7, 139.8, 139.7, 139.6, 139.5, 135.9, 133.9, 132.3, 131.6, 130.5, 130.2, 129.9, 128.3, 128.0,



Scheme 1. Chemical structure of HTL, ETL, HIL and dopant materials.



Scheme 2. (a) Br_2, $CHCl_3$, 25 °C, 24 h, (b) Tetrakis(triphenylphosphine)palladium(0), K_2CO_3, THF, 18 h, 80 °C.

126.9, 126.2, 125.9, 125.5, 124.4, 123.4, 120.3, 77.9, 77.3, 76.7 ppm. FT-IR (KBr, cm^{-1}) 1600 (aromatic C=C) 3120,889 (aromatic C–H). MS (FAB) *m*/*z* 772.0 [(M+1)$^+$]. Anal. Calcd. for $C_{61}H_{38}$ (770.95) C, 94.94; H, 5.06. Found: C, 95.02; H, 4.98. UV–vis (THF): λ_{max} (Absorption) = 368 nm, λ_{max} (Emission) = 460 nm.

2.4. OLED fabrication

The organic layers were deposited sequentially onto a substrate at a rate of 1.0 Å/s by thermal evaporation from heated alumina crucibles. The thicknesses of the *N*,*N*′-bis-[4-(di-*m*-tolylamino)-phenyl]-*N*,*N*′-diphenylbiphenyl-4,4′-diamine (DNTPD, hole injection layer (HIL)), bis[*N*-(1-naphthyl)-N-phenyl]benzidine (α-NPD, hole transport layer (HTL)), host:5% dopant (emission layer (EML)) and Alq_3 layers were *ca.* 400, 200, 300 and 200 Å, respectively, as shown in Scheme 1. Diphenyl-[4-(2-[1,1;4,1]terphenyl-4-yl-vinyl)phenyl]-amine (BD-1) and 1,6-bis[(*p*-trimethylsilylphenyl)amino]pyrene (SPP) were used as the conventional dopant. Before depositing the metal cathode, a 10 Å thick LiF layer was deposited onto the organic layers. A high-purity aluminum cathode was then deposited as the top layer at a rate of 1–5 Å/s to a thickness of 2000 Å.

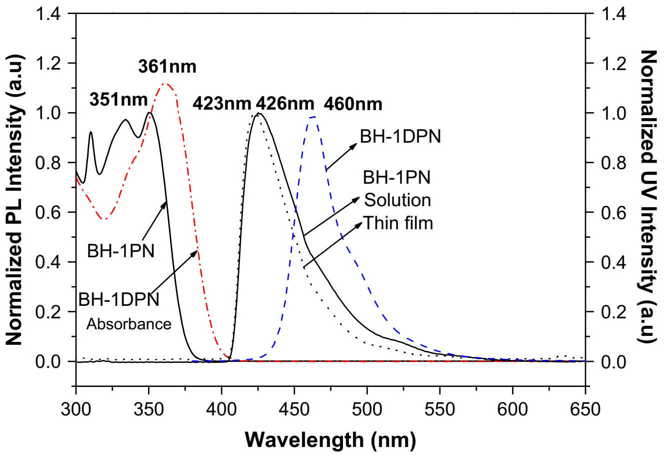


Fig. 1. UV–vis and PL spectra of BH–1PN and BH–1DPN.

3. Results and discussion

3.1. Synthesis and characterization

The BH–1PN and BH–1DPN host materials were prepared using the Suzuki reaction of compounds **1** and **2** with 4-(naphthalene-1-yl)phenylboronic acid in the presence of a palladium catalyst. The synthetic routes to BH–1PN and BH–1DPN are described in Scheme 2. The chemical structures and compositions of the resulting precursor and spiro compounds were characterized by ^{1}H NMR, ^{13}C NMR, FT-IR, GC–MS and elemental analysis.

3.2. Optical properties and energy levels

Fig. 1 and Table 1 show the UV–vis and photoluminescence (PL) spectra and data of the spiro compounds, respectively. BH–1PN showed a UV–vis absorption maximum at 357 nm, whereas BH–1DPN showed a UV absorption maximum at 368 nm in film. The PL absorption maximum of BH–1PN and BH–1DPN were located at 423 and 472 nm in film, respectively. The introduction of a naphthylphenyl group to both the 5- and 9-positions in spiro[fluorine-benzofluorene] caused a significant red-shift in the PL spectrum of BH–1DPN to 472 nm as a result of increased electron delocalization.

Table 1
UV absorption, PL, Energy levels and thermal properties of BH–1PN and BH–1DPN.

Sample	Properties								Φ_f[a]
	Abs (nm)		PL (nm)		Energy level (eV)		Thermal properties (°C)		
	Solution	Film	Solution	Film	HOMO	LUMO	T_g	T_m	
BH–1PN	351	357	426	423	5.95	2.84	171.0	339.3	0.58
BH–1DPN	361	368	454	472	5.85	2.72	200.2	371.8	0.69

[a] Fluorescence quantum efficiency, relative to 9,10-diphenylanthracene in cyclohexane ($\Phi_f = 0.90$).

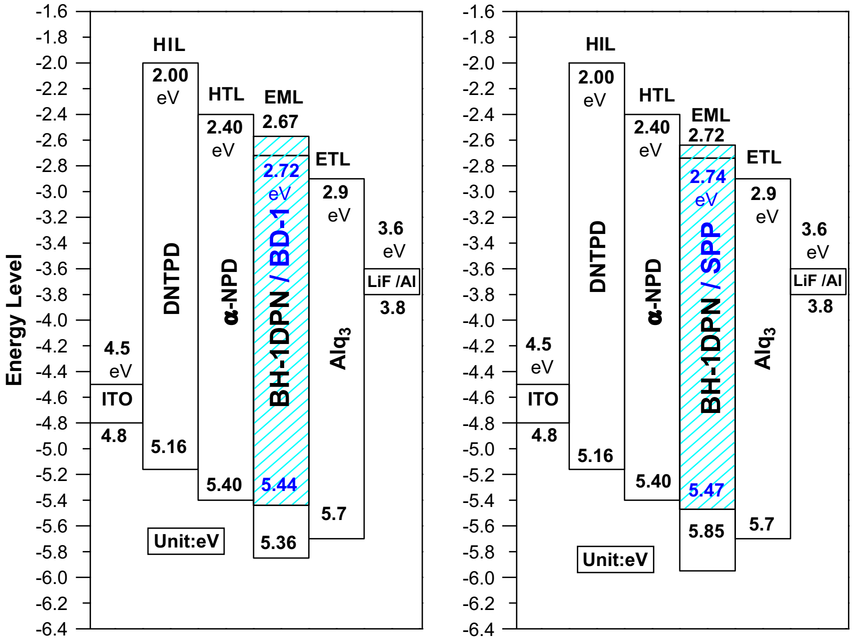


Fig. 2. Energy level of BH–1PN and BH–1DPN doped with BD-1 and SPP.

The solution fluorescence quantum efficiency (Φ_f) of host materials are 0.58 and 0.72, were determined relative to 9,10-diphenylanthracene in cyclohexane ($\Phi_f = 0.90$).

A low-energy photoelectron spectrometer was used to obtain information on the HOMO energies of the host materials and to examine the charge injection barriers. The HOMO energy levels of host materials were determined to be 5.95 and 5.85 eV for BH–1PN and BH–1DPN by AC-2, respectively. The optical energy band gaps of BH–1PN and BH–1DPN were estimated to be 3.31 and 3.13 eV, respectively, based on the energy threshold of its electronic absorption spectrum. The LUMO energy levels of BH–1PN and BH–1DPN were calculated to be 2.64 and 2.72 eV, respectively. Table 1 and Fig. 2 show the energy levels of devices using BH–1DP and BH–1DPN host materials.

3.3. Thermal properties

Table 2 summarizes the DSC data of BH–1PN and BH-1DPN. The purified samples of BH–1PN and BH–1DPN showed a melting point (T_m) of 339.3 and 371.8 °C after sublimation, respectively. After the sample had cooled to room temperature, a second DSC scan performed at 10 °C/min revealed a high glass transition temperature (T_g) of 171.0 and 200.2 °C, respectively. This suggests that thermal stability was improved significantly by the introduction of a naphthylphenyl moiety. BH–1DPN is more thermally stable than BH–1PN because it has a more symmetric structure and a higher molecular weight due to the presence of an additional naphthylphenyl group.

Table 2
EL properties of the devices obtained from BH–1PN and BH–1DPN host and dopant materials.

Properties		Devices					
		BH–1PN			BH–1DPN		
		–	BD-1	SPP	–	BD-1	SPP
EL at 7 V	λ_{max} (nm)	440	444	464	440	448	464
	mA/cm^2	4.41	8.39	7.83	15.90	15.93	13.95
	cd/A	1.01	2.81	3.39	1.37	3.17	3.70
	lm/W	0.45	1.26	1.52	0.62	1.44	1.65
	cd/m^2	44.26	265.4	226.4	217.2	505.6	515.9
	CIE-x	0.160	0.153	0.137	0.159	0.153	0.138
	CIE-y	0.083	0.101	0.132	0.083	0.119	0.138
	EQE(%)	0.86	2.26	2.82	0.93	2.61	2.98

3.4. EL properties

The EL properties of the BH–1PN and BH–1DPN host materials were examined by fabricating multilayer devices with the following configuration: glass ITO anode/HIL/HTL/EML/electron transport layer (ETL)/electron injection layer (EIL)/Al cathode. DNTPD was used as the HIL, α-NPD as the HTL, BH–1PN or BH–1DPN:5% dopant as the EML and Alq3 as the ETL; A 10 Å LiF layer was used as the EIL.

Fig. 3 shows the normalized EL spectra of a device composed of BH–1PN and BH–1DPN doped with BD-1 and SPP at 7 V. All the devices showed intense blue emission in the EL spectra at 448 nm

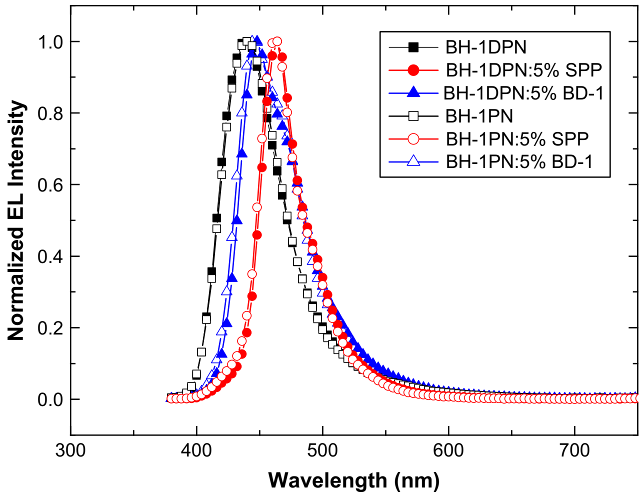


Fig. 3. EL spectra of blue OLED obtained from BH–1PN and BH–1DPN doped with 5% dopant.

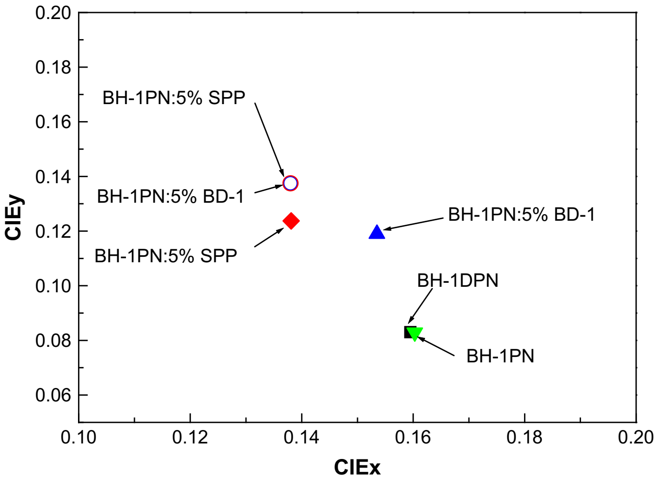


Fig. 4. The CIE 1931 coordinates diagram of devices at 7 V.

and 462 nm for BH–1PN and BH–1DPN, respectively, which does not resemble that of the PL spectra. The maximum wavelength of the EL spectra varied significantly according to the dopant material. The devices doped with SPP showed 20 and 16 nm longer wavelength in the EL spectra than those of BD-1, respectively. Based on the EL spectrum, the CIE coordinates of the emitting layer BH–1PN and BH–1DPN doped with 5% SPP were measured to be (0.139, 0.119) and (0.139, 0.133), respectively, as shown in Fig. 4. The device composed of BH–1PN without the dopant showed an EL spectrum at 440 nm, while the other EL spectra were shifted 8 or 22 nm toward longer wavelengths.

The devices consisting of BH–1PN:5% BD-1 and BH–1DPN:5% BD-1 in the emitting layer showed a deep blue emission (x,y) = (0.153, 0.101) and (0.153, 0.119), respectively, in terms of the CIE chromaticity coordinates. A comparison of these chromaticity coordinates with those of optimal devices containing MADN:5% BD-1 confirmed that the devices containing BH–1PN and BH–1DPN had better color saturation, i.e., the CIE coordinates of BH–1PN and BH–1DPN were better than those of MADN (0.154, 0.123) [27].

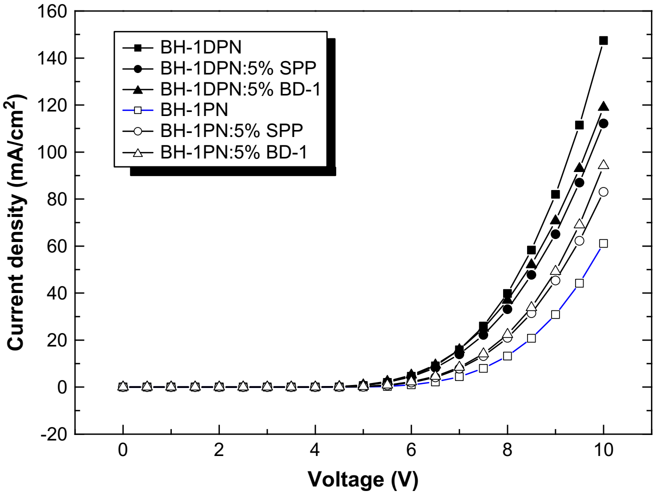


Fig. 6. Current density–voltage characteristics of BH–1PN and BH–1DPN host materials.

3.5. OLED device properties

Fig. 5 shows the luminance–voltage characteristics of the OLEDs with BH–1PN and BH–1DPN as the EML. In the case of the device using BH–1DPN doped with 5% SPP, light emission occurs at approximately 4.5 V with a brightness of 3684 cd/m^2 at 10 V. The luminance and efficiency of the devices made using BH–1DPN as the host and 5% BD-1 as the dopant were more than 5 times higher those of the device without the dopant. This suggests that the dopant is effective in enhancing the EL properties of the spiro-type emitting layer. Table 2 summarizes the device characteristics at 7 V.

The BH–1DPN:5% BD-1/Alq3 device also showed a maximum brightness of 3684 cd/m^2 at a current density of 119.05 mA/cm^2, as shown in Figs. 5 and 6. The luminescent efficiency of BH–1DPN:5% SPP device increased abruptly to 4.03 cd/A as the current density was increased gradually. This was attributed to the formation of excitons required to emit light. The maximum power efficiency of the device obtained from BH–1DPN doped with 5% SPP was 1.65 lm/W, which is double that of the device without the dopant (0.65 lm/W). The device shows the maximum EQE of 2.97% and the efficiency increased rapidly to a maximum of approximately 3.70 cd/A at a low current density of 13.95 mA/cm^2 at 7 V.

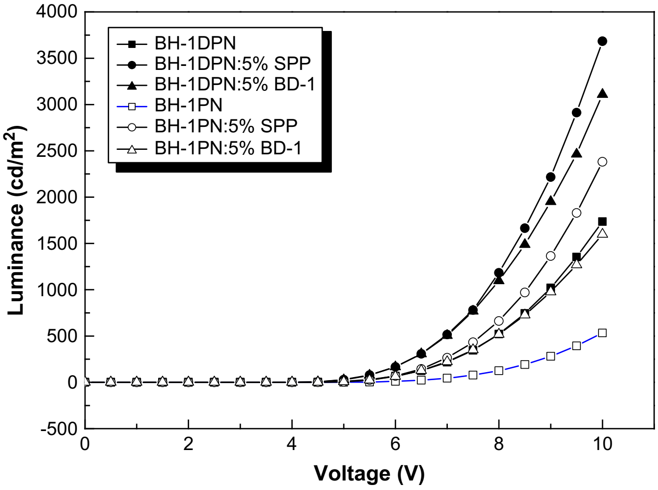


Fig. 5. Brightness–voltage characteristics of the device composed of BH–1PN and BH–1DPN hosts.

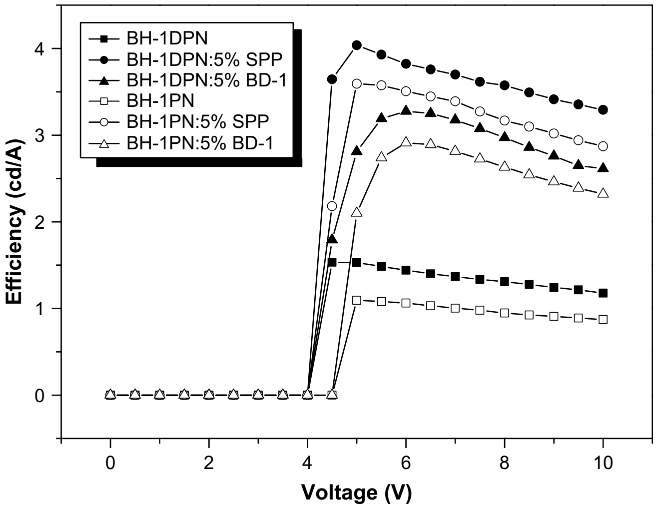


Fig. 7. Efficiency–voltage characteristics of the device using BH–1PN and BH–1DPN.

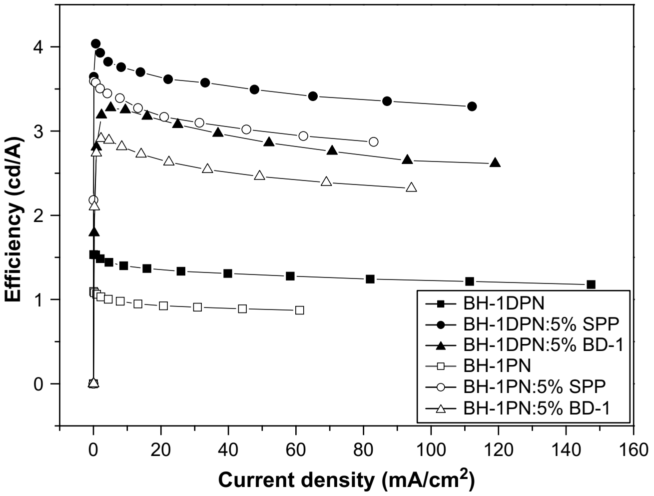


Fig. 8. Efficiency–current density characteristics of the device using BH–1PN and BH–1DPN.

Device consisting of ITO/DNTPD/NPB/BH–1DPN:5% BH–3BD/Alq$_3$/Al–LiF, exhibited a relatively higher efficiency of 4.03 cd/A at 5 V in its efficiency–voltage relation, as shown in Fig. 7.

Fig. 8 shows the dependence of efficiency on the current density of six devices. It can be seen that BH–1DPN doped with 5% SPP shows the highest efficiency and a little decrease of efficiency as the current density increased from 0 to 112 mA/cm^2, e.g., a weak current induced fluorescent quenching. It reached a current efficiency of 2.42 cd/A.

This suggests that an exciton is formed and light is emitted at specific thresholds. It should be noted that the efficiencies of these devices remained stable when the current density was increased to 112.14 mA/cm^2. The inset in the energy band diagram of BH–1DPN and SPP shows that the PL spectra of BH–1DPN overlaps with a major portion of the absorption spectrum of SPP. This indicates that SPP can effectively accept energy from the host through Förster energy transfer or function as a direct recombination center due to the higher HOMO levels.

4. Conclusion

New blue light-emitting spiro-type host and dopant materials were prepared to construct a blue, organic light-emitting diode. We have demonstrated that OLED performances are significantly affected by the introduction of various substituents into the 5- and 9-position. The EL emissions of the devices (doped with 5% BD-1 or SPP) are observed at 444 and 462 nm, respectively. In optimized structures, the device obtained using BH–1DPN: 5% SPP had greatest luminance at 516 cd/m^2, at a current density of 13.95 mA/cm^2 being 3.70 cd/A with deep blue color coordinates of (0.138, 0.138). Lifetime measurement tests are now in progress.

Acknowledgement

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