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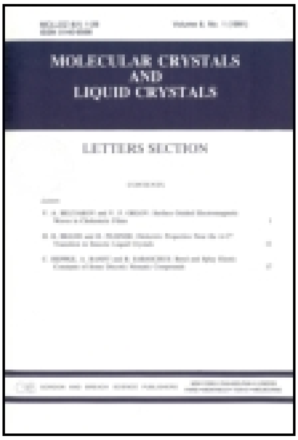

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4-(2-(4′-(2,2-diphenylvinyl)biphenyl-4-yl)vinyl)-N,N-diphenylaniline Derivatives for Blue Organic Light-Emitting Diodes

Sung Min Kwon [a] , Kum Hee Lee [a] , Seok Jae Lee [b] , Young Kwan Kim [b] & Seung Soo Yoon [a]

[a] Department of Chemistry , Sungkyunkwan University , Suwon , 440-746 , Korea

[b] Department of Information Display , Hongik University , Seoul , 121-791 , Korea

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4-(2-(4′-(2,2-diphenylvinyl)biphenyl-4-yl)vinyl)-N,N-diphenylaniline Derivatives for Blue Organic Light-Emitting Diodes

SUNG MIN KWON,[1] KUM HEE LEE,[1] SEOK JAE LEE,[2]
YOUNG KWAN KIM,[2] AND SEUNG SOO YOON[1,*]

[1]Department of Chemistry, Sungkyunkwan University, Suwon 440-746, Korea
[2]Department of Information Display, Hongik University, Seoul 121-791, Korea

This paper reports the synthesis and electroluminescent properties of the new blue fluorescent diphenylaniline derivatives end-caped with various aryl groups. Multilayered OLEDs were fabricated using these materials as dopants in ADN host, with the configuration of ITO/DNTPD (40 nm)/NPB (20 nm)/ADN: Blue dopants ***1–4*** *(20 nm)/Alq*$_3$ *(40 nm)/Liq (1.0 nm)/Al. All devices showed the efficient blue emissions. Among these devices, the devices* ***C*** *as a dopant exhibited blue emission with the luminance of 18020 cd/m*^{2} *at 11v, the luminous efficiency of 9.6 cd/A at 20 mA/cm*^{2}*, the power efficiency of 3.91 lm/W at 20 mA/cm*^{2}*. In addition, the device* ***D*** *exhibited deep blue emisson with CIE coordinates of (0.151, 0.134) at 8 V showed a luminous efficiency of 5.3 cd/A and power efficiency of 2.3 lm/W at 20 mA/cm*^{2}*.*

Keywords Blue OLEDs; fluorescent dopant; styrylarylene derivatives; unsymmetrical structure

Introduction

Since the pioneering work of multilayered organic light-emitting devices (OLEDs) by Tang, this research field has attracted extensive attention because of its huge market potential in the next-generation flat-panel display technology [1]. For full color OLED display applications, a set of primary blue, green, and red emitters is required. Red and green OLEDs with high efficiency and stability have been achieved [2,3]. However, the development of efficient blue-emitting materials remains an important issue for full color display. Therefore, there were focused on the development of high-performance blue electroluminescent materials with desirable properties, because the improved efficiency, raised color purity, and extended operation lifetime are difficult due to their wide energy band gap [4–6]. For this reason, several efficient blue emitters have been developed, including metal-complexes, distyrylarylenes, oligofluorenes, spirobifluorenes, carbazoles, and others [7–13]. Among the blue emitters developed thus far, an unsymmetrical mono(styryl)amine-based blue dopant known as BD-1 has high efficiency and a pure blue color. A current efficiency of 5.4 cd/A with CIE coordinates of (0.14, 0.13) and external quantum efficiency of 5.1%

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[*] Address correspondence to Prof. Seung Soo Yoon, Department of Chemistry, Sungkyunkwan University, Cheoncheon-dong, Jangan-gu, Suwon, Gyeonggi-do 440-746, Korea (ROK). Tel.: (+82)31-290-7071; Fax: (+82)31-290-5971. E-mail: ssyoon@skku.edu

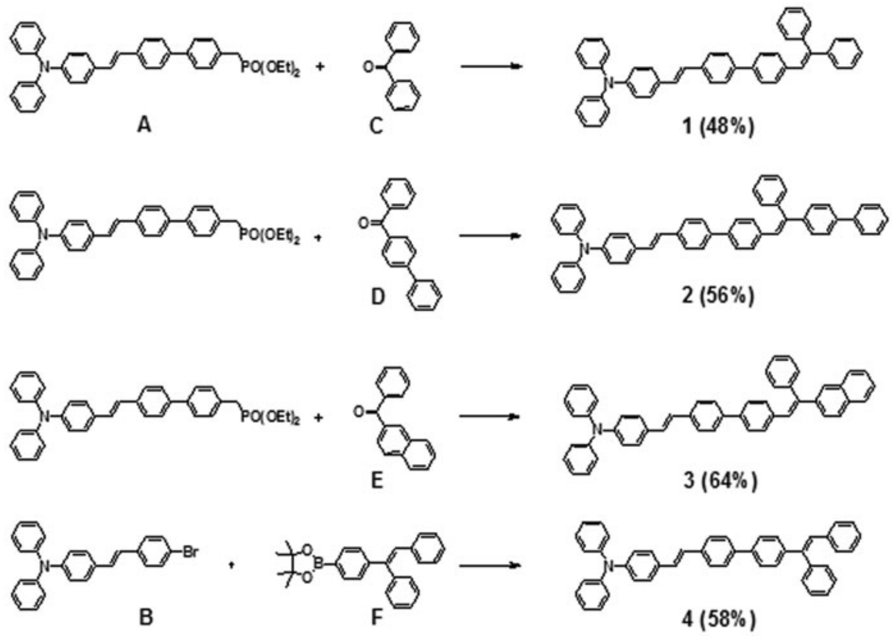

Figure 1. Synthetic routes of blue fluorescent materials **1**–**4**. Conditions: (a) KOtBu, THF, 0°C to room temperature, 30 min. (b) Pd(PPh_3)$_4$, K_2CO_3 (2M), Aliquat 336, Toluene, reflux, 3h.

were demonstrated by doping BD-1 in 2-methyl-9,10-di(2-naphthyl)anthracene (MADN). Unfortunately, blue dopant materials based on aromatic amine units suffer color-shifting problems at high doping concentrations due to intermolecular interactions between the dopant materials.

In this paper, we report the synthesis, characterization, and EL properties of the blue fluorescent materials based on unsymmetrical diphenylaminostyrylarylene derivatives for OLEDs. The blue fluorescent materials (**1–4**) were based on a 4,4′-distyrylbiphenyl with a diphenylamine unit and various end-capped groups (Fig. 1). In these materials, the 4,4′-distyrylbiphenyl cores were combined with diphenylamine units, which have the enhanced hole-transporting properties [14–17]. Furthermore, the bulky diaryl end-capping groups could prevent the close-packing of these materials in solid state and improve the EL efficiencies by the suppression of concentration quenching. Also, the unsymmetrical structures of these materials would have less tendency to pack into crystal lattice and hence favor amorphous morphology [18–20]. As described herein, blue OLEDs using these novel blue materials showed the high efficiencies and deep blue color chromaticities.

Experimental

General Procedure for the Synthesis of Compounds 1–4

General procedure for the Horner-Wadsworth-Emmons reaction (**1-3**): To a mixture of phosphonate intermediate (0.11 mmol) and the corresponding aldehyde (0.13 mmol) in 10 mL of anhydrous THF at 0°C was added dropwise 0.13 mL of 1.0 M KOtBu (0.13 mmol) in THF under nitrogen. The reaction mixture was stirred for 10 min at 0°C, followed by

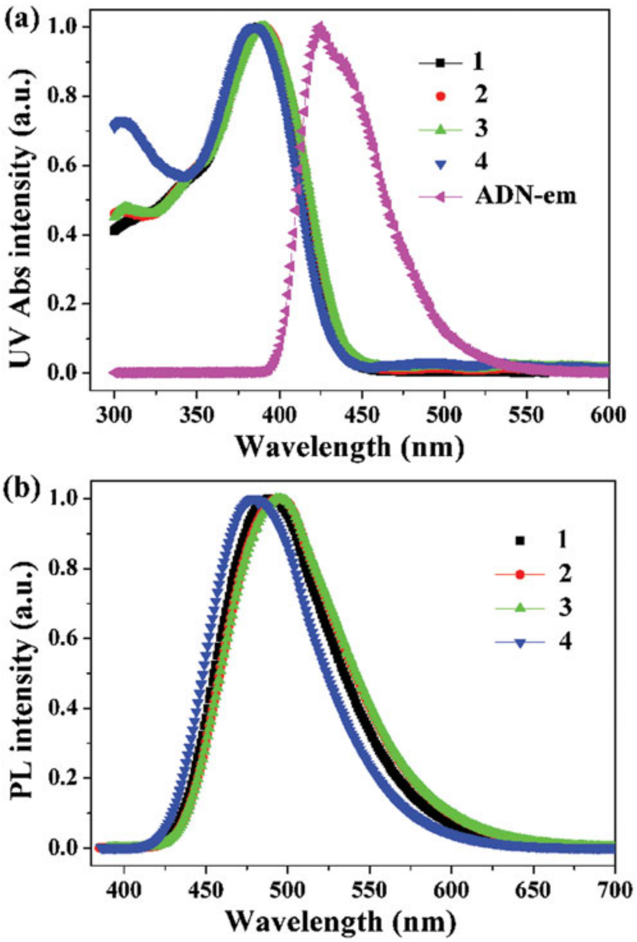

Figure 2. UV absorption spectra (a) and PL spectra of materials (**1**–**4**).

1 hr at room temperature and quenched with water. The solution mixture was filtered with water. The filtered solid was dissolved in CH_2Cl_2, dried over Na_2SO_4, filtered with silica gel, and the solvent removed under reduced pressure. The crude mixture was purified through column chromatography using silica-gel and crystallization to give the desired compound.

Table 1. Physical properties of materials **1–4**

Compound	UV[a]	PL[a]	FWHM	HOMO	LUMO	E_g	Q.Y.[b]
1	388	488	79	5.58	2.78	2.80	0.21
2	390	495	83	5.59	2.76	2.83	0.60
3	389	495	82	5.58	2.75	2.83	0.72
4	385	479	75	5.60	2.72	2.88	0.89

[a] CH_2Cl_2 solution (10^{-5} M). [b] Using BDAVBi as a standard; $\lambda_{ex} = 360$nm ($\Phi = 0.86$ in CH_2Cl_2).

Table 2. Electroluminescent characteristics of devices **A-D**

Device	EL [nm][a]	J [mA/cm^2][b]	L [cd m^{-2}][d]	LE [cd A^{-1}][c,d]	PE [lm W^{-1}][c,d]	CIE [x,y][e]
A	458, 60	128	16800	8.08/ 8.17	3.26/ 3.77	0.159, 0.176
B	460, 56	86	15970	8.81/ 8.82	3.59/ 3.85	0.157, 0.185
C	462, 56	102	18020	9.57/ 9.59	3.92/ 4.28	0.157, 0.197
D	454, 55	128	5257	5.29/ 5.30	2.26/ 2.35	0.151, 0.134

[a]FWHM. [b]Current density at 10V. [c]At 20 mA cm^{-2}. [d]Maximum value. [e]Value collected at 8.0 V.

1: 4-(2-(4′-(2,2-diphenylvinyl)biphenyl-4-yl)vinyl)-*N*,*N*-diphenylaniline: Yield 48%. ^{1}H-NMR (300MHz, $CDCl_3$): δ ppm 7.54-7.47 (m, 4H), 7.40-7.30 (m, 11H), 7.29-7.28 (m, 1H), 7.26-7.20 (m, 6H), 7.11-7.09 (d, $J = 7{,}5$Hz, 5H), 7.06-6.95 (m, 8H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ ppm 147.5, 147.4, 143.4, 142.7, 140.5, 139.3, 138.8, 136.7, 136.450, 131.5, 130.4, 130.0, 129.3, 128.8, 128.3, 128.2, 127.7, 127.6, 127.6, 127.5, 127.4, 127.0, 126.7, 126.5, 126.3, 124.5, 123.6, 123.1. APCI-MS (m/z): 602 [M$^+$]. HRMS [EI$^+$] calcd for $C_{46}H_{35}N$: 601.2770, found: 601.2765.

2: 4-(2-(4′-(2-(biphenyl-4-yl)-2-phenylvinyl)biphenyl-4-yl)vinyl)-*N*,*N*-diphenylaniline; Yield 56%. ^{1}H-NMR (300MHz, $CDCl_3$): δ ppm 7.68-7.66 (d, $J = 6.9$Hz, 1H), 7.62-7.60 (d, $J = 8.1$ Hz, 2H), 7.57-7.52 (m, 5H), 7.45-7.28 (m, 15H), 7.23 (s, 2H), 7.17-7.14 (d, $J = 8.4$Hz, 1H), 7.12-7.10 (d, $J = 8.1$Hz, 5H),7.06-7.02 (m, 7H). ^{13}C-NMR (75 MHz, $CDCl_3$): δ ppm 147.5, 147.4, 143.5, 142.3, 140.6, 140.1, 139.4, 139.3, 138.8, 136.7, 136.5, 131.5, 130.9, 130.4, 130.1, 129.3, 128.9, 128.8, 128.3, 128.2, 128.0, 127.8, 127.6, 127.4, 127.3, 127.0, 126.9, 126.7, 126.5, 126.4, 126.3, 124.5, 123.6, 123.1. APCI-MS (m/z): 678 [M$^+$]. HRMS [EI$^+$] calcd for $C_{52}H_{39}N$: 677.3083, found: 677.3081.

3: 4-(2-(4′-(2-(naphthalen-2-yl)-2-phenylvinyl)biphenyl-4-yl)vinyl)-*N*,*N*-diphenylaniline: Yield 64 %. ^{1}H-NMR (300MHz, $CDCl_3$): δ ppm 7.86-7.71 (m, 4H), 7.55-7.46 (m,

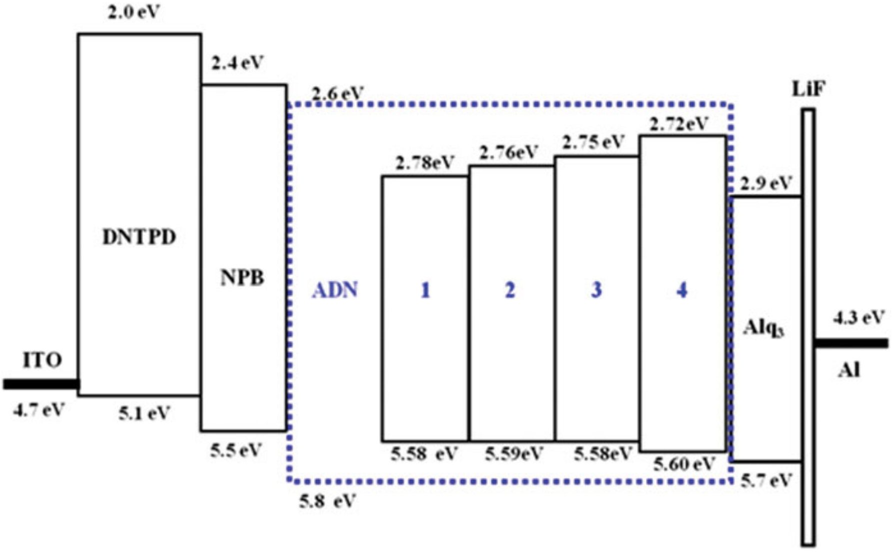

Figure 3. Device structure and energy level diagram used in this study.

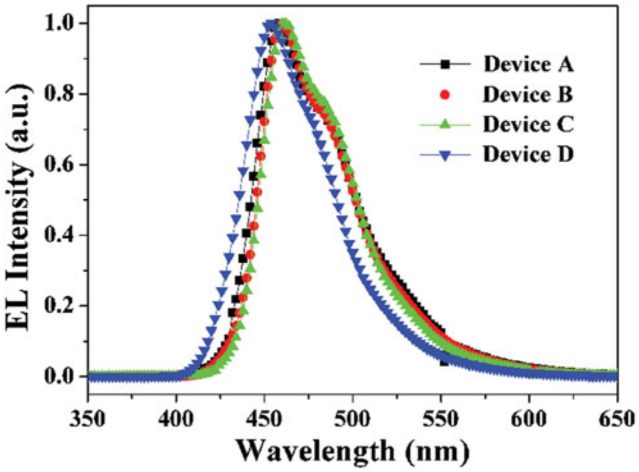

Figure 4. EL spectra of devices **A-D**.

5H), 7.44-7.41(m, 2H), 7.39-7.27 (m, 9H), 7.25-7.22 (m, 4H), 7.12-7.01 (m, 12H), 6.99 (s, 1H). ^{13}C-NMR (75 MHz, CDCl$_3$): δ ppm 147.6, 147.4, 138.8, 131.5, 130.5, 130.1, 129.3, 128.8, 128.7, 128.4, 128.3, 128.2, 127.8, 127.8, 127.6, 127.4, 127.0, 127.0, 126.9, 126.8, 126.7, 126.5, 126.3, 126.2, 126.1, 126.1, 124.6, 123.6, 123.1. APCI-MS (m/z): 652 [M$^+$]. HRMS [EI$^+$] calcd for $C_{50}H_{37}N$: 651.2926, found: 651.2922.

General Procedure for the Suzuki cross-coupling reaction, synthesis of **4**: **A** (0.48 mmol) the corresponding **F** (0.72 mmol), Pd(PPh$_3$)$_4$ (0.024 mol), aqueous 2.0 M K_2CO_3 (6.0 mmol), Aliquat 336, and toluene were mixed in a flask. The mixture was refluxed for 2.5 hr. After the reaction had finished, the reaction mixture was extracted with ethyl acetate and washed with water. The organic layer was dried with anhydrous $MgSO_4$ and filtered with silica gel. The crude mixture was purified through column chromatography using silica-gel and crystallization to give the desired compound.

4: 4-(2-(4′-(1,2-diphenylvinyl)biphenyl-4-yl)vinyl)-*N*,*N*-diphenylaniline: Yield 58%. ^{1}H-NMR (300MHz, CDCl$_3$): δ ppm 7.65-7.55 (m, 4H), 7.41-7.24 (m, 12H), 7.13-6.98 (m, 17H). ^{13}C-NMR (75 MHz, CDCl$_3$): δ ppm 147.6, 147.3, 139.5, 139.4, 137.4, 136.8, 131.5, 131.0, 130.4, 129.6, 129.3, 128.7, 128.4, 128.2, 128.1, 128.0, 127.8, 127.6, 127.5, 127.4, 127.1, 126.9, 126.8, 126.6, 126.5, 124.5, 123.6, 123.1. APCI-MS (m/z): [M$^+$]. HRMS [EI$^+$] calcd for $C_{46}H_{35}N$: 601.2770, found: 601.2767.

Optical Measurements

The UV-vis absorption and photoluminescence spectra of the new dopant materials were measured in dichloromethane (10^{-5} M) using a Sinco S-3100 and Aminco-Browman series 2 luminescence spectrometer, respectively. The fluorescence quantum yields of the blue materials were determined in dichloromethane at 293 K against BDAVBi as a reference ($\Phi = 0.86$) [21]. HOMO (highest occupied molecular orbital) energy levels were determined with a low energy photoelectron spectrometer (Riken-Keiki, AC-2). The energy band gaps were determined from the intersection of the absorption and photoluminescence spectra. LUMO (lowest unoccupied molecular orbital) energy levels were calculated by subtracting the corresponding optical band gap energies from the HOMO energy values.

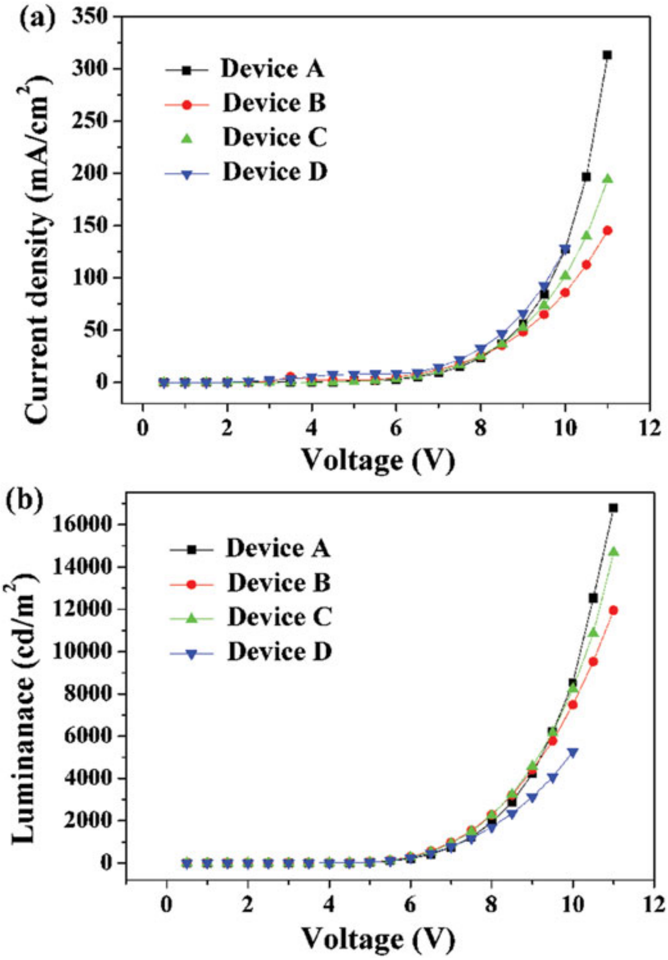

Figure 5. Current density and luminance versus applied electric voltage characteristics of devices **A-D**.

Fabrication of OLED

For fabricating OLEDs, indium-tin-oxide (ITO) thin films coated on glass substrates were used, 30 Ω/square of the sheet resistivity with a thickness of 1000 A. The ITO-coated glass was cleaned in an ultrasonic bath by the following sequence: acetone, methyl alcohol, distilled water, storage in isopropyl alcohol for 48 h, and dried by N_2 gas gun. The substrates were treated by O_2 plasma under conditions of 2×10^{-2} Torr at 125 W for 2 min. All organic materials and metal were deposited under high vacuum (5×10^{-7} Torr). The OLEDs were fabricated in the following sequence: ITO/ DNTPD (40 nm)/ NPB (20 nm)/ ADN: Blue dopants **1–4** (20 nm)/ Alq_3 (40 nm)/ Liq (1.0 nm)/ Al. The current density (J), luminance (L), luminous efficiency (LE), power efficiency (PE), and the CIE chromaticity coordinates of the OLEDs were measured with Keithly 2400, Chroma meter CS-1000A. Electroluminescence was measured using a Roper Scientific Pro 300i.

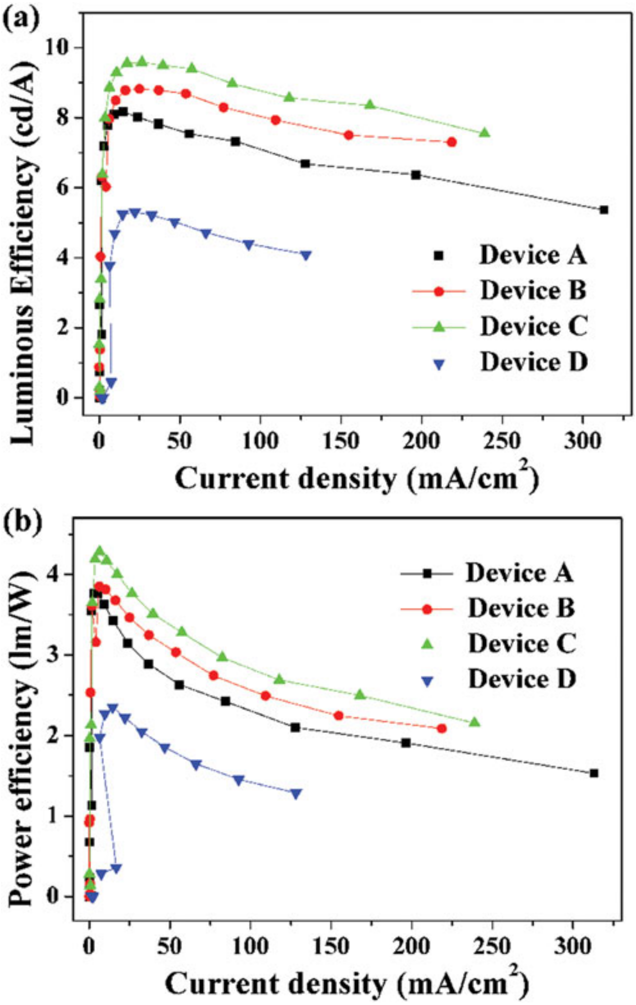

Figure 6. Luminous and power efficiencies as a function of current density for devices **A-D**.

Results and Discussion

The blue fluorescent materials (**1–4**) used for this study were synthesized by the Honor-Wadsworth-Emmons reaction and Suzuki coupling reaction, and molecular structures are shown in Fig. 1. The molecular structures of these materials were characterized by ^{1}H- and ^{13}C-NMR and low- and high-resolution mass spectroscopy.

The UV-Vis absorption spectra of these blue fluorescent materials in dichloromethane are shown in Fig. 2(a). Materials **1–4** showed the maximum absorption peaks in the 385-390 nm range. The good spectral overlap was observed between the emission of the commom blue host (ADN) and the absorptions of materials **1–4**. These observations indicate that the blue materials **1–4** can effectively accept energy from the ADN host material by Förster-type energy transfer. A particularly good spectral overlap was observed for material **3**. The efficiency of Förster-type energy transfer is highly dependent on the spectral overlap between the emission of the host and the absorption of the dopant, thus this material **3** as a dopant would be expected to have highly efficient electroluminescent properties in OLED devices using AND host.

The normalized PL emission spectra of **1–4** are shown in Fig. 2(b). All materials showed blue fluorescence with maximum emission wavelengths of 488, 495, 495 and 479 nm, respectively. Compared to **1**, the PL spectra of **2** and **3** show the red-shift of 7 nm owing to the lengthening the π-conjugation length by the additional end-capped groups such as naphthalene and biphenyl. On the other hand, compared to **1**, the PL spectrum of **4** shows the blue shift of 9 nm due to the shortening the π-conjugation length due to the steric repulsion between two end-capping phenyl groups. These observations imply that the conjugation length of **1–4** is sensitively dependent on end-capped aryl substituents. These materials **1–4**, with the exception of **1**, have high PL quantum yields of 0.60-0.89, suggesting that these materials were expected to have highly efficient electroluminescent properties in OLED devices.

The highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) energy levels of **1–4** are shown in Table 1. The HOMO energy levels were measured with an AC-2 photoelectron spectrometer and the LUMO levels calculated by subtracting the corresponding optical band gap energies from the HOMO values. The HOMO/LUMO energy levels of **1–4** are 5.58/2.78, 5.59/2.76, 5.58/2.75 and 5.60/2.72, respectively.

To explore the electroluminescent properties of these materials, devices were fabricated and the electroluminescent performance characteristics of devices are summarized in Table 2. The doping concentrations of **1–4** in ADN host were optimized to 2%, respectively. The EL emission spectra of devices **A**-**D** with emitting layers consisting of the ADN host doped with dopants **1–4** are seen in Fig. 4. All four doped devices exhibited blue emissions with the maximum emission peaks between 454-462 nm. The corresponding CIE coordinates are $x = 0.159$, $y = 0.176$ for **A**, $x = 0.157$, $y = 0.185$ for **B**, $x = 0.157$, $y = 0.197$ for **C**, and $x = 0.151$, $y = 0.134$ for **D**, and their full width at half maximum (FWHM) values are 60, 56, 56, and 55nm, respectively. The FWHM of the EL spectra of device **D** is found to be quite narrow and therefore the color purity is excellent. Compare to CIE coordinates of device **D**, those of **A**-**C** show red-shifts from 0.042 to 0.063 in the CIE_y coordinate due to the lengthening π-conjugation length of the dopants, which were in good agreement with the trends of the PL spectra of materials **1–4**.

Figure 5(a) and (b) shows the current density-voltage and luminance-voltage characteristics of devices **A**-**D** containing ADN doped with various dopant materials. The maximum current densities of these blue OLEDs varied from 86 mA/cm^2 in device **B** to as high as 128 mA/cm^2 in device **A, D**. The current density of devices **A**-**D** is determined by several factors such as the differences in HOMO/ LUMO energy levels between the adjacent organic layers and the charge carrier mobility of each materials in devices. Since materials and structure in devices **A**-**D** are under the same condition of except dopants, the charge carrier mobility of dopants **1–4** seems to be profound effects on electronic properties of devices. The maximum brightness of these blue OLEDs varied from 5257 cd/m^2 in device **D** to as high as 18020 cd/m^2 in device **3**. The luminous efficiencies and power efficiencies of the doped devices **A**-**D**, as a function of the current density, are shown in Fig. 6(a) and (b). Their power efficiency of devices **A**-**D** were 8.08, 8.81, 9.57, and 5.29 cd/A at 20 mA/cm^2, respectively. Their power efficiency of devices **A**-**D** were 3.26, 3.59, 3.92, and 2.26 lm/W at 20 mA/cm^2, respectively. Notably, the EL efficiencies of device **C** with dopant **3** was much higher than those of devices **A**, **B** and **D** with dopants **1**, **2** and **4**. Presumably, compared to dopants **1**, **2** and **4**, the superior spectral overlap between the emission of the host (ADN) and the absorption of dopant **3**, as shown in Fig. 2(a), and thus the effective energy transfer from ADN host to dopant **3** in device **C** would lead to the improved EL efficiencies of device **C** in comparison with devices **A**, **B** and **D**. This study clearly suggests that efficiencies of blue OLEDs composed of a host and dopant can be improved by a superior matched spectral overlap between the emission of the host and the absorption of the dopant.

Conclusions

We have synthesized blue fluorescent materials (**1–4**) *via* Horner-Wadsworth-Emmons reaction and Suzuki cross-coupling reaction. When doped in the ADN host in the OLED device, these materials showed the efficient blue electroluminescent properties. Particularly, the device using dopant **3** exhibited a highly efficient blue emission with the maximum luminance of 18020 cd/m^2 at 11 V, the luminous efficiency of 9.6 cd/A at 20 mA/cm^2, power efficiency of 3.9 lm/W at 20 mA/cm^2, and CIE $_{x,\,y}$ coordinates of (0.157, 0.197) at 8 V. In addition, an OLED device using dopant **4** exhibited a efficient deep-blue emission with a maximum luminanace, luminous efficiency and power efficiency of 5257 cd/m^2 at 10 V, 5.29 cd/A, and 2.26 lm/W at 20 mA/cm^2, respectively. The maximum peak wavelength of the EL spectrum of this device was 454nm with CIE $_{x,\,y}$ coordinates of (0.151, 0.134) at 8 V. These results demonstrated the superiority of these unsymmetrical diphenylamino-styrylarylene derivatives as the blue emitting materials in OLED devices.

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