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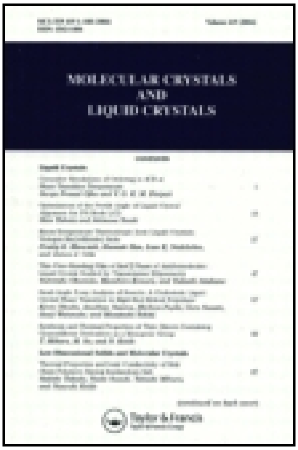

Molecular Crystals and Liquid Crystals

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Green Light-Emitting Diodes (LED) Based on Diarylethene

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Green Light-Emitting Diodes (LED) Based on Diarylethene

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A novel class of diarylethene derivatives, (diaryl = ***N****-7-azaindolylpheny, naphthyl-phenylamine,* ***m****-tolylphenylamine, ethene = perfluorocyclopentene) were synthesized and experiments were performed their usage in organic light-emitting diodes (OLEDs). Compound* ***3****, 1,2-Bis{4-[(phenyl)(m-tolyl)amino]biphenyl}-3,3,4,4,5,5-hexafluorocyclopentene, exhibits a strong green emission in both a solution and a thin film at ∼540 nm. The device with* ***3*** *as an emitting material (structure: ITO/TPD/****3****/*Alq_3 *(q = tris-(8-hydroxyquinolinolate))/Al:Li) shows high quantum efficiency with 7677 cd/*m^2 *at ∼13 V.*

Keywords: OLEDs, perflurocyclopentene; photophysical properties; X-ray structure

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1. INTRODUCTION

Luminescent compounds exerted great influence in material science because of their applicable potential in organic light-emitting diodes (OLEDs) [1,2]. High-performance organic materials emitting the three elemental colors of red, green, and blue are required to realize full-color displays. After two decades of active research, a number of red, green, and blue emitters have been reported both in small molecular and polymer systems [3]. The photochromic materials have recently attracted interest in the view of their potential applications for visible image formation, optical data storage and optical switching [4]. It has been of great interest and significance to create photochromic amorphous molecular materials containing a dithienylethene moiety. Because not only were dithienylethene derivatives regarded as promising photochromic compounds, but they also showed high thermal stability and excellent fatigue-resistant properties [5]. There are a very small number of reports on the change in the carrier injection and transport characteristics with photo-isomerization of a photochromic dithienylethene film [6], an organic bistable molecular memory using nonsymmetrical bipolar diarylethene with a triphenylamine group and an oxadiazole group [7]. Recently, Shirota *et al.* reported that the introduction of two bulky triphenylamine moieties into dithienylethene framework increases the population of the antiparallel-conformer of the molecule, leading to higher conversion of the photocyclization reaction [4,8]. Although they have very high quantum efficiency for the photocyclization reaction (Φ_{o-c}), the study on their OLEDs performance is still rare. In this paper, we report the design and syntheses of novel green emitting materials with high fluorescence quantum efficiency suitable for OLED applications based on the diarylethene-functionalized triarylamine derivatives and the investigation of their photo- and electroluminescent characteristics.

2. EXPERIMENTAL SECTION

General Considerations

All experiments were performed under dry N_2 atmosphere using standard Schlenk technique. All solvents were freshly distilled over appropriate drying reagents prior to use. All starting materials were purchased from either Aldrich or Strem and used without further purification. The starting materials, 4-Bromo-4′-(1-naphthyl-phenylamino)biphenyl, 4-bromo-4′-(1-naphthylphenylamino)benzene, 4-bromo-4′-(phenyl-*m*-tolylamino)biphenyl, 4-bromo-4′-(phenyl-*m*-tolylamino)-benzene [9], and *p*-bromo(*N*-7-azaindolyl)benzene [10] were prepared according to the literature.

Measurement

^{1}H-NMR was recorded on a Bruker avance 300 MHz spectrometer or JEOL-JMS 700 instrument, respectively. UV/Vis and photoluminescent spectra for all samples with concentrations in the range of 10–50 μM were obtained from UV/Vis spectrometer Lambda 900 and a Perkin Elmer Luminescence spectrometer LS 50B, respectively. All solutions for photophysical experiments were degassed with more than three repeated freeze-pump-thaw cycles in a vacuum line. The fluorescence quantum yields in CH_2Cl_2 using Alq_3 as the standard were determined by the previous report [11].

X-ray Crystallographic Analysis

Suitable crystals of **1** were obtained from slow vapor diffusion of benzene/hexane(2:1) into solution of **1** in CH_2Cl_2. The crystals of **1** were attached to glass fibers and mounted on a Bruker SMART diffractometer equipped with a graphite monochromated Mo Kα ($\lambda = 0.71073$ Å) radiation, operating at 50 kV and 30 mA and a CCD detector; 45 frames of two-dimensional diffraction images were collected and processed to obtain the cell parameters and orientation matrix. All data collections were performed at 173 K. The data collection 2θ range for **1** is 3.54–56.70°. No significant decay was observed during the data collection. The raw data was processed to give structure factors using the SAINT program [12]. Each structure was solved by direction methods and refined by full matrix least squares against F^2 for all data using SHELXTL software (version 5.10) [13]. All non-hydrogen atoms in compounds **1** were anisotropically refined. All hydrogen atoms were included in the calculated positions and their contributions in structural factor calculations were included. The fluorine atoms are partially disordered over two sites. However, their contributions were also included with structural factors. The crystal system in compound **1** belongs to orthorhombic and *Ccca* space group. Crystal data and structure refinement for **1** are summarized in Table 1. Selected bond lengths and angles are given in Figure 1. The refined atomic coordinates and anisotropic thermal parameters are deposited in Cambridge Crystallographic Data Center and the supporting information [14].

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TABLE 1 Crystal Data and Structure Refinement for **1**

Formula	$C_{31} H_{18} F_6 N_4$
M	560.49
T/K	173(2)
Crystal system	Orthorhombic
Space group	*Ccca*
a/Å	14.6032(10)
b/Å	23.0054(15)
c/Å	14.9591(9)
$A, \beta, \gamma/°$	90
V/Å^3	5025.5(6)
Z	8
μ(Mo-Kα)/mm^{-1}	0.120
Crystal size (mm)	0.2 × 0.15 × 0.1
Theta range for data collection(°)	1.77 to 28.35
Reflections collected	17579
Independent reflections	3129
Goodness-of-fit on F^2	1.001
$R1$ [$I > 2\sigma(I)$]	0.0497
$wR2$	0.1266

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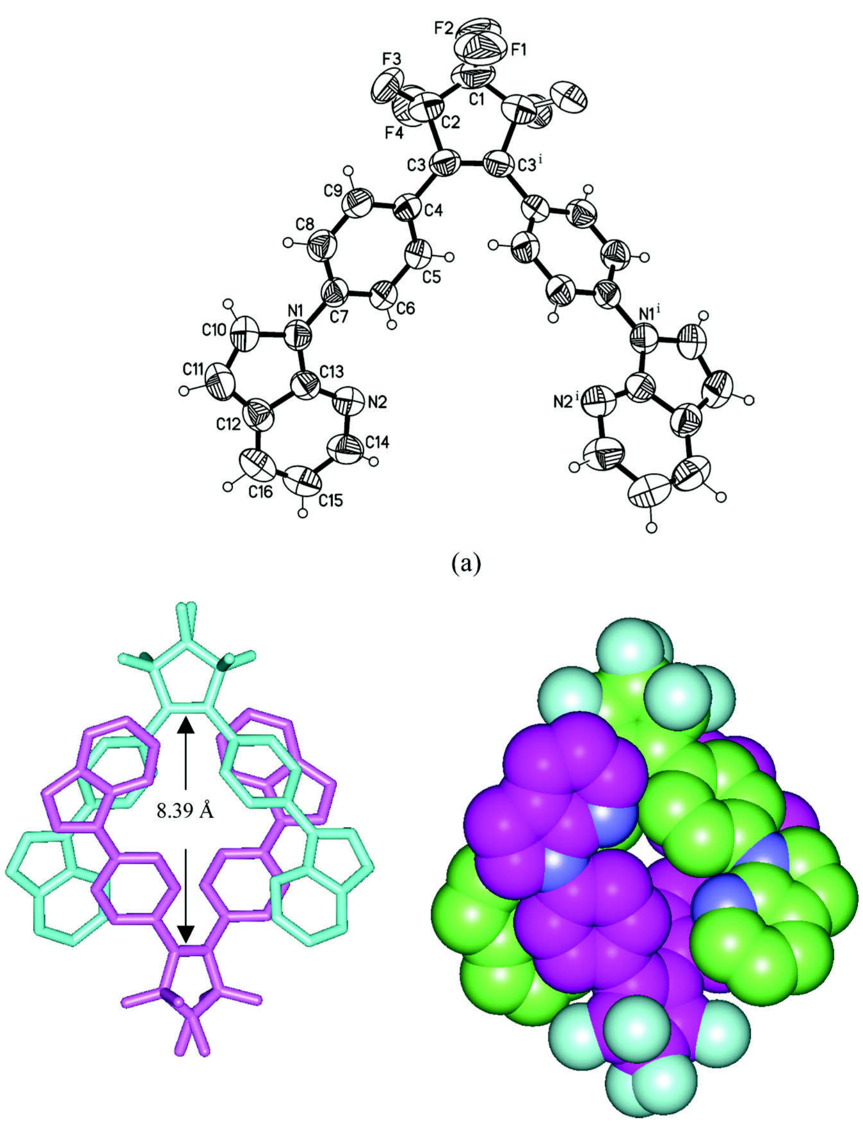
Fabrications of Electroluminescent Devices

The glass substrate pre-coated with indium-tin-oxide (ITO) was cleaned by an ultrasonic bath of acetone, followed by 2-propanol. Surface treatment was carried out by exposing ITO to UV-ozone plasma. The electroluminescence (EL) device is fabricated as follows. The hole-transporting layer, a 50 nm thick film of TPD (1,4-bis(phenyl-*m*-tolylamino)biphenyl) was deposited on the ITO surface by high vacuum thermal evaporation. Compound **5** was thermally evaporated on the TPD layer and a 50 nm thick Alq_3 layer was then deposited as the electron transporting layer. Finally LiF (1 nm) and Al (100 nm) were deposited on top of the organic layers by thermal evaporation. The fabricated multilayer organic light emitting devices have the structure of ITO/TPD (50 nm)/compound **3** (50 nm)/Alq_3 (50 nm)/LiF:Al. The organic layers were deposited at a rate of 0.2 nm/s at 10^{-5} Torr. The current-voltage and luminance were measured with a Keithley 2400 Source and a Newport optical meter.

1,2-Bis{4-(N-7-azaindolyl)phenyl}-3,3,4,4,5,5-hexafluorocyclopentene (1)

To a stirred THF solution (25 mL) of *p*-bromo(*N*-7-azaindolyl)benzene (0.67 g, 1.2 mmol) was added a solution of *n*-BuLi (0.9 mL, 1.6 M



(a)

(b)

FIGURE 1 (a) Molecular structure of **1** along with atom labeling schemes and 50% thermal ellipsoids. One part of the disordered fluorine atoms is omitted for clarity. (b) Diagrams showing the interlocked pair of **1**. Selected bond lengths and angles; N1-C7: 1.424(3) Å, C3-C3^{i}: 1.353(5) Å, C3-C4: 1.475(3) Å, C13-N1-C7: 129.0(2)°, C4-C3-C3^{i}: 129.28(12)°. Symmetry transformation used to generate equivalent atoms: (i) 1-*x*, *y*, 0.5-*z*.

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in hexane, 1.44 mmol) at −78°C and stirred for 30 min. To this was added octafluorocyclopentene (0.25 mL, 0.6 mmol) at that temperature. The reaction mixture was warmed to ambient temperature slowly and stirred for 8 h. The solvent was removed under reduced pressure and extracted with CH_2Cl_2. After the usual work-up, the pure product **1** was isolated by chromatographic workup (eluent: ethyl acetate/hexane (1/5), $R_f = 0.3$) in 69% yield. Mp: 214°C. ^1H NMR ($CDCl_3$): δ 8.38 (dd, 2H, $J = 4.8$ Hz, $J = 1.5$ Hz), 7.95 (dd, 2H, $J = 7.8$ Hz, $J = 1.5$ Hz), 7.91 (d, 4H, $J = 8.7$ Hz), 7.57 (d, 4H, $J = 8.7$ Hz), 7.53 (d, 2H, $J = 3.9$ Hz), 7.15 (dd, 2H, $J = 7.8$ Hz, $J = 4.8$ Hz), 6.65 (d, 2H, $J = 3.9$ Hz). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 149.7, 143.0, 141.2, 137.6, 136.2, 132.4, 131.5, 128.9, 128.0, 127.3, 126.3, 123.6, 121.8, 116.9. MS: m/z 560 [M^+]. Anal. Calcd for $C_{31}H_{18}F_6N_4$: C, 66.43; H, 3.24. Found: C, 66.35; H, 3.18.

1,2-Bis{4-[(phenyl)(m-tolyl)amino]phenyl}-3,3,4,4,5,5-hexafluorocyclopentene (2)

Compound **2** was prepared using the same procedure as described for **1** using 4-bromo-4′-(phenyl-*m*-tolylamino)benzene instead of *p*-bromo (*N*-7-azaindolyl)benzene in 82% yield. (Eluent: CH_2Cl_2/hexane (1/5), $R_f = 0.4$.) Mp: 146–148°C. ^1H NMR ($CDCl_3$): δ 7.21–6.82 (m, 26H, *Ph*), 2.19 (s, 6H, Ph-CH_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 149.5, 146.7, 139.6, 130.7, 129.9, 129.3, 129.1, 127.2, 126.3, 125.9, 125.1, 124.6, 123.6, 122.4, 121.5, 120.3, 120.1, 117.0, 21.4. MS: m/z 690 [M^+]. Anal. Calcd for $C_{43}H_{32}F_6N_2$: C, 74.77; H, 4.67. Found: C, 74.51; H, 4.59.

1,2-Bis{4-[(phenyl)(m-tolyl)amino]biphenyl}-3,3,4,4,5,5-hexafluorocyclopentene (3)

Compound **3** was prepared using the same procedure as described for **1** using 4-bromo-4′-(phenyl-*m*-tolylamino) biphenyl instead of *p*-bromo (*N*-7-azaindolyl)benzene in 75% yield. (Eluent: CH_2Cl_2/hexane (1/3), $R_f = 0.5$.) Mp: 129–131 °C. ^1H NMR ($CDCl_3$): δ 7.57–6.87 (m, 34H, *Ph*), 2.28 (s, 6H, Ph-CH_3). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 148.2, 147.6, 147.5, 142.5, 139.4, 132.9, 129.6, 129.2, 129.1, 127.6, 126.1, 125.9, 125.3, 124.6, 124.3, 123.7, 123.1, 122.0, 21.4. MS: m/z 842 [M^+]. Anal. Calcd for $C_{55}H_{40}F_6N_2$: C, 78.37; H, 4.78. Found: C, 78.23; H, 4.66.

1,2-Bis{4-[(1-naphthyl)(phenyl)amino]phenyl}-3,3,4,4,5,5-hexafluorocyclopentene (4)

Compound **4** was prepared using the same procedure as described for **1** using 4-bromo-4′-(1-naphthylphenylamino)benzene instead of *p*-bromo(*N*-7-azaindolyl)benzene in 79% yield. (Eluent: CH_2Cl_2/hexane hexane (1/5), $R_f = 0.3$.) Mp: 118–120°C. ^1H NMR ($CDCl_3$): δ 7.91–7.82

(m, 6H, *Ph*), 7.52–7.03 (m, 22H, *Ph*), 6.82 (d, $J = 8.8\,\text{Hz}$, 4H, *Ph*). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 150.0, 146.9, 142.5, 135.2, 131.0, 130.2, 129.4, 128.7, 128.4, 128.1, 127.4, 127.1, 126.6, 126.3, 124.4, 123.9, 123.5, 122.9, 119.7, 119.2, 118.4. MS: m/z 762 [M^+]. Anal. Calcd for $C_{49}H_{32}F_6N_2$: C, 77.15; H, 4.23. Found: C, 76.97; H, 4.06.

1,2-Bis{4-[(1-naphthyl)(phenyl)amino]biphenyl}-3,3,4,4,5,5-hexafluorocyclopentene (5)

Compound **5** was prepared using the same procedure as described for **1** using 4-bromo-4′-(1-naphthylphenylamino)biphenyl instead of *p*-bromo(*N*-7-azaindolyl)benzene in 72% yield. (Eluent: CH_2Cl_2/hexane (1/3), $R_f = 0.45$.) Mp: 226–227°C. ^1H NMR ($CDCl_3$): δ 7.92 (d, 2H, $J = 8.1\,\text{Hz}$), 7.90 (d, 2H, $J = 8.1\,\text{Hz}$), 7.79 (d, 2H, $J = 8.3\,\text{Hz}$), 7.52–6.96 (m, 34H). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ 148.6, 147.9, 143.2, 142.4, 135.3, 132.1, 131.2, 129.8, 129.2, 128.4, 127.6, 127.3, 126.7, 126.6, 126.5, 126.3, 126.2, 126.0, 124.1, 123.3, 122.6, 122.4, 121.2, 120.6, 116.7. MS: m/z 914 [M^+]. Anal. Calcd for $C_{61}H_{40}F_6N_2$: C, 80.07; H, 4.41. Found: C, 80.22; H, 4.55.

3. RESULT AND DISCUSSION

In order to develop amorphous molecules with high quantum efficiency, we have designed and synthesized a novel class of photochromic compounds based on diarylethene. The synthetic routes of **1**, **2**, **3**, **4**, and **5** are given in Scheme 1. These compounds were prepared by the addition of perfluorocyclopentene to the corresponding lithiated compound, prepared from the reaction of n-BuLi and 1-bromoarylamine at −78°C, give a yellow suspension. The standard work up and crystallization from CH_2Cl_2/hexane gives titled compounds as a pure pale yellow solid in moderate yield (69–82%). All compounds are stable in air and soluble in common organic solvent except hexane and pentane and have been characterized by ^1H-NMR, ^{13}C-NMR and elemental analyses, including X-ray diffraction analysis of **1**.

The pale-yellow single crystals of **1** suitable for X-ray analysis were obtained by the vapor diffusion of benzene/hexane(2:1) into solution of **1** in CH_2Cl_2. The crystal structure and the selected bond lengths and angles of **1** are depicted, as shown in Figure 1. In the crystal structure of **1**, a 2-fold symmetry axis passes along the vector linked C1 and the central position between C3 and $C3^i$. The asymmetric unit, therefore, consists of a half of **1**. The bond lengths C3-$C3^i$, C3-C4 and C7-N1 are 1.353(5) Å, 1.475(3) Å and 1.424(3) Å, respectively, which are similar to those of previously reported diarylethene compounds [15]. The dihedral angle between the phenyl ring (C4-C9) and *N*-7-azaindole

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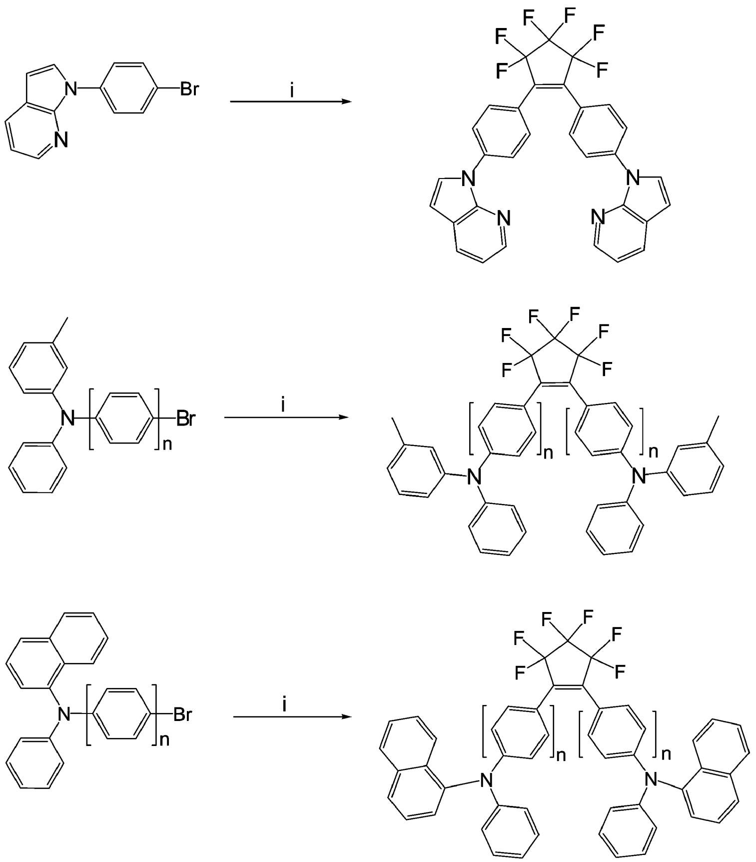

(1)

n=1(**2**)

n=2 (**3**)

n=1 (**4**)

n=2 (**5**)

SCHEME 1 Synthetic routes of **1**, **2**, **3**, **4**, and **5**. (i) n-BuLi/THF at –78°C, perfluorocyclopentene.

ring (N1, N2, C10-C14) is approximately 8.48(6)°, indicating an effective π conjugation of the *N*-7-azaindolyl functional group. Compound **1** has an interesting spatial arrangement in the crystal lattice. The crystal lattice molecules of **1** form an interlocked pair, resulting in a cage-like arrangement structure with an approximately S_2 symmetry. The separation distance between two paired molecules is about 8.39 Å. Notably, there are significant intermolecular interactions, such as hydrogen bonding and π-π stacking, in the crystal packing. The bond length of N2−H5^{ii} in two paired molecules [16] is 2.60 Å, which is sufficiently short to support intermolecular interactions between two adjacent molecules (hydrogen bonding). In addition, two 7-azaindole rings of the adjacent molecules are almost parallel with a dihedral

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TABLE 2 Photophysical Data for **2–5**[a]

Compound	Absorption (λ_{max}, nm) Solution	Absorption (λ_{max}, nm) Film	Emission (λ_{max}, nm) Solution	Emission (λ_{max}, nm) Film	Quantum Yield (Φ_{PL})[b]
2	301, 370	301, 373	512	538	1.33
3	317, 385	307, 382	530	543	1.57
4	263, 352	275, 365	565	565	1.22
5	271, 369	283, 378	580	580	1.45

[a] All data were collected in CH_2Cl_2 solution at room temperature.
[b] The quantum yield were obtained by comparing to Alq_3 (1.0) as a reference [18].

angle of 0.00(7)° and the shortest separation distance of 3.437(3)Å, indicating that there is π-π stacking interaction in the crystal packing (See the supporting information).

Table 2 summarizes the photophysical data of **2–5**. The UV/Vis spectra of all compounds exhibit intense absorption bands between 250 and 450 nm. The observed results indicate that the electronic transitions are mostly π-π^*, originated from diaryl groups [17]. At room temperature, the solution of **3** displays two absorption bands, a former one at $\lambda_{max} = 317$ nm and a latter one at $\lambda_{max} = 385$ nm, as shown in Figure 2. The same trends in the absorption of **2**, **4**

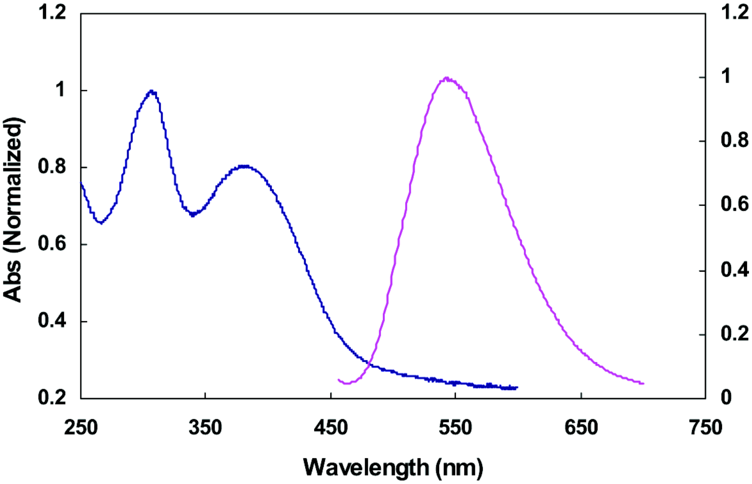

FIGURE 2 Absorption and emission spectra of **3** in thin films.

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and **5** are also observed. The red-shift in the absorption maximum of **3** and **5**, as compared to those of **2** and **4**, is due to an increase of π-conjugation from the biphenylene unit. These observed absorption spectra are comparable to those typical of diarylethene derivatives [16]. All compounds yield green emissions in both solution and thin film at room temperature. The emission $\lambda_{\max}$ of **2–5** in film are at 538 nm for **2**, 543 nm for **3**, 565 nm for **4**, 580 nm for **5**, respectively. The PL (photoluminescence) efficiencies of all compounds were noteworthy. It has been well known that Alq_3 is regarded as a standard green emitter with unity value of PL efficiency [18]. As compared to that of Alq_3, all compounds show high PL quantum efficiency. The extended conjugation in **3** and **5** may be responsible for the enhanced photoluminescent efficiencies, compared to those of **2** and **5**. Based on this observation, we expected that all compounds would be potential green emitters in OLEDs.

The EL device **A** was fabricated using compound **3** as emitting material (structure **A**: ITO/TPD/compound **3**/Alq_3/Al:Li). Moreover, in order to evaluate electron transporting ability, an additional device **B** was also fabricated with **3** as electron transfer material. (structure **B**: ITO/TPD/compound **3**/Al:Li). Figures 3 and 4 show the I-V-L characteristics of two devices. Compound **3** in device **A** displays bright a green emission with $\lambda_{\max} = 534$ nm, which is well matched to that of the PL spectrum in thin film. There are no characteristic emission

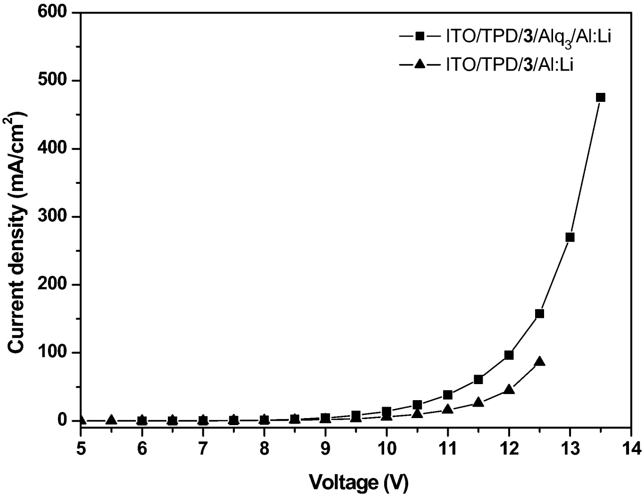

FIGURE 3 Voltage versus current-density characteristics of **3**.

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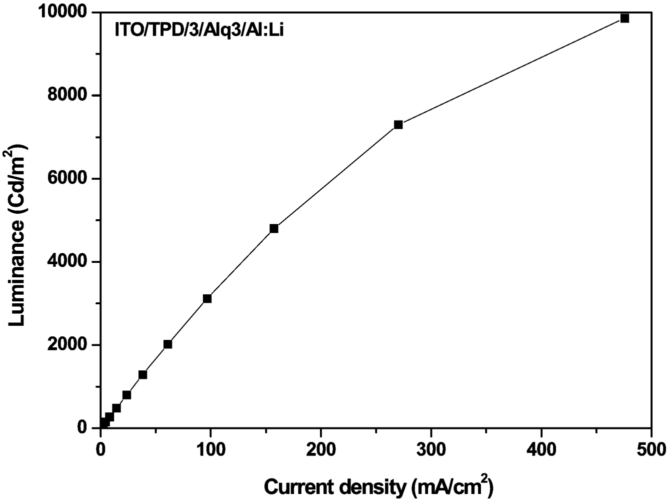

FIGURE 4 Luminance versus current-density characteristics of **3**.

peaks from TPD and Alq_3, indicating that the emission originates mostly from compound **3**. The device **B** failed to produce reasonable light output. The turn-on voltage and maximum current density are at 6.5 V and 100 mA/cm^2, respectively. This result suggests that the compound **3** falls in electron injection and transporting ability relative to those of Alq_3. In general, fluorine groups, which can act as electron-withdrawing groups, stabilize either HOMO (Highest Occupied Molecular Orbital) or LUMO (Lowest Unoccupied Molecular Orbital) in molecular materials [19]. The low-lying LUMO energy levels give rise to easy electron injection, leading to diminution of operating voltage in OLEDs. Based on this consideration, the fluorine groups in perfluorocyclopentene do not have a significant influence on the LUMO energy levels. However, device **A** produced a bright green light with about 5.0 lm/W at 10,000 cd/m^2 (turn-on voltage: 11 V; luminance: 7677 cd/m^2 at ~13 V). The observed high efficiency of device **A** is very likely from the contribution of perfluorocyclopentene group as well as the effective conjugation of aryl and diarylamine units.

In summary, we were able to incorporate a perfluorocyclopentene group into bis(diarylamino)aryl units. Compounds **2** and **3** exhibit intense green fluorescence emissions both in solution and in thin film. EL devices based on compounds **3** were fabricated. Device **A** (ITO/TPD/**3**/Alq_3/Al:Li) offers bright green emission with good luminous efficiency.

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