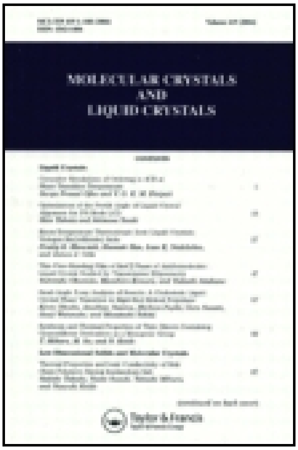

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Synthesis and Electro-Optical Properties of 9,10-Substituted Anthracene Derivatives for Flexible OLED Devices

Yoon Soo Han [a] , Seonju Jeong [a] , Sang Chul Ryu [b] , Eun Jung Park [b] , Eon Joo Lyu [c] , Giseop Kwak [c] & Lee Soon Park [c] [d]

[a] Division of Nano-Bio Technology, Daegu Gyeongbuk Institute of Science and Technology (DGIST), Dalseo-gu, Daegu, Korea

[b] LG Display Co., Ltd., Jinpyung-dong, Gumi-si Gyungsangbuk-do, Korea

[c] Department of Polymer Science, Kyungpook National University, Daegu, Korea

[d] Advanced Display Manufacturing Research Center, Kyungpook National University, Daegu, Korea

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Synthesis and Electro-Optical Properties of 9,10-Substituted Anthracene Derivatives for Flexible OLED Devices

Yoon Soo Han[1], Seonju Jeong[1], Sang Chul Ryu[2], Eun Jung Park[2], Eon Joo Lyu[3], Giseop Kwak[3], and Lee Soon Park[3,4]
[1]Division of Nano-Bio Technology, Daegu Gyeongbuk Institute of Science and Technology (DGIST), Dalseo-gu, Daegu, Korea
[2]LG Display Co., Ltd., Jinpyung-dong, Gumi-si Gyungsangbuk-do, Korea
[3]Department of Polymer Science, Kyungpook National University, Daegu, Korea
[4]Advanced Display Manufacturing Research Center, Kyungpook National University, Daegu, Korea

*A silylated anthracene derivative, bis[2-(p-trimethylsilyl)phenylethynyl]anthracene (**1**), was synthesized by using Sonogashira reaction. Compound **1** showed two absorption maxia($\lambda_{max,abs}$) at 444 and 447 nm, and fluorescence maximum ($\lambda_{max,em}$) at 478 nm. Compound **1** had higher melting temperature of 274°C compared to anthracene 210°C presumably due to confinement of π-conjugated system by trimethylsilyl end-capping, which will be beneficial in the high temperature operation condition of OLED devices. As compared to the commercially available, blue-light emitting, material **4** and tert-butylated compound **2**, the compound **1** showed small red-shift of 9 nm in the OLED emission from the by PL λ_{max} due to the increased electron density in the anthracene ring. The compound **1** was also found to be easily fabricated in the flexible OLED devices and showed a low threshold voltage and high current efficiency compared with other anthracene derivatives.*

Keywords: anthracene derivateives; flexible OLED; fluorescence; OLED

INTRODUCTION

Organic light-emitting diodes (OLEDs) based on organic molecules are currently the subject of intense research efforts due to their promise as

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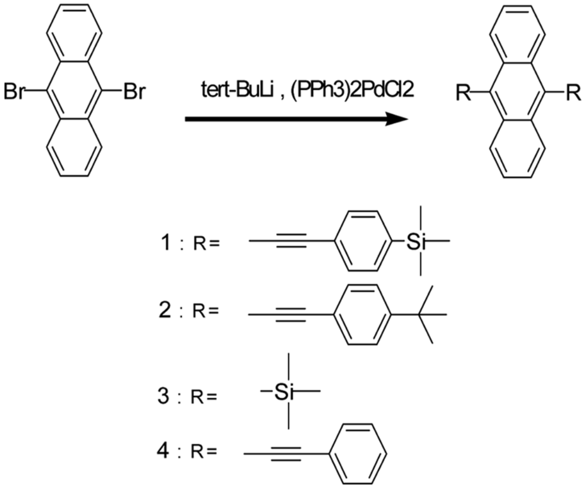
Address correspondence to
Prof. Lee Soon Park, Department of Polymer Science, Kyungpook National University, Sangyuk-dong, Buk-gu, Daegu 702-701, Korea (ROK). E-mail: lspark@knu.ac.kr

devices for full-color display applications [1–3]. Numerous conjugated molecules have been synthesized and reported to exhibit electroluminescence (EL) from blue to red range [4,5]. Although considerable research efforts have been directed to the practical use of OLEDs, problems still remain for example good blue emitting materials with high purity and quantum yield. Blue emitting materials reported include anthracene [5–7], carbazole [7–9], fluorene [10–13], stylene [14], oxidaiazole [15] and triazole [16] derivatives.

Anthracene is one of the simplest aromatic hydrocarbons studied for blue-light emission in OLEDs. To enhance the fluorescence quantum efficiency substitution in anthracene fused ring was tried to give dense electronic structure and efficient exciton confinement. For example, 9,10-phenylethynylated and 9,10-silylated anthracene derivatives exhibited significant bathochromic shift of fluorescence and higher quantum yield compared to other anthracene deivatives. Usually an incorporation of silylene moiety into π-conjugate system induces a relatively weak σ-π conjugation through the vacant d orbital of silicon atom. Thus end-capping of π-conjugate molecule by silyl group may be an effective method to increase the fluorescence quantum yield while maintaining the original conjugation length.

The π-extension of aromatic molecules could be attained by various coupling reactions such as Sonogashira's, Stille's, and Suzuki's methods. Silylated aromatic molecules can be obtained by substitution reaction of silyl chlorides into lithiated aromatic molecules.



SCHEME 1 9,10-disubstituted anthracene derivatives used in this work.

In this work we synthsized new anthracene derivatives substitued by silyl group and tert-butyl group, bis[2-(p-trimethylsilyl)phenylethynyl] anthracene (**1** in Scheme 1) and bis[2-(4-(tert-butyl)phenyl)ethynyl] anthracene (**2** in Scheme 1), respectively. The electro-optical properties of there new anthracene compounds were investigated in comparison with the known anthracene compounds (**3**,**4** in Scheme 1). The silylated anthracene compound was found to have good thermal stability and thus studied for a potential blue-light emitting host material in the flexible OLED devices on the plastic film substarate.

EXPERIMENTAL

1. Materials

The reagents such as 9,10-dibromoanthracene, chlorotrimethylsilane, tert-butyllithium (1.7 M in pentane), 1,4dibromobenzene, butyllithium (1.6 M in hexane), 2-methyl-3-butyn-2-ol,triphenylphosphine,copper(Cu)iodide, bis(triphenylphospine)palladium (Pd) chloride, sodium hydride (60% dispersion in mineral oil), triphenylphospine and 4-*tert*-butylphenylacetylene were purchased from Aldrich Chemical Co. and used without further purification. Anthracene and 9,10-bis (2-phenylethynyl)anthracene (97%) (**4**) were also purchased from Aldrich Chemical and used as reference materials. Tetrahydrofuran, diethyl ether, trimethylamine and toluene used as solvents were distilled and dried before use.

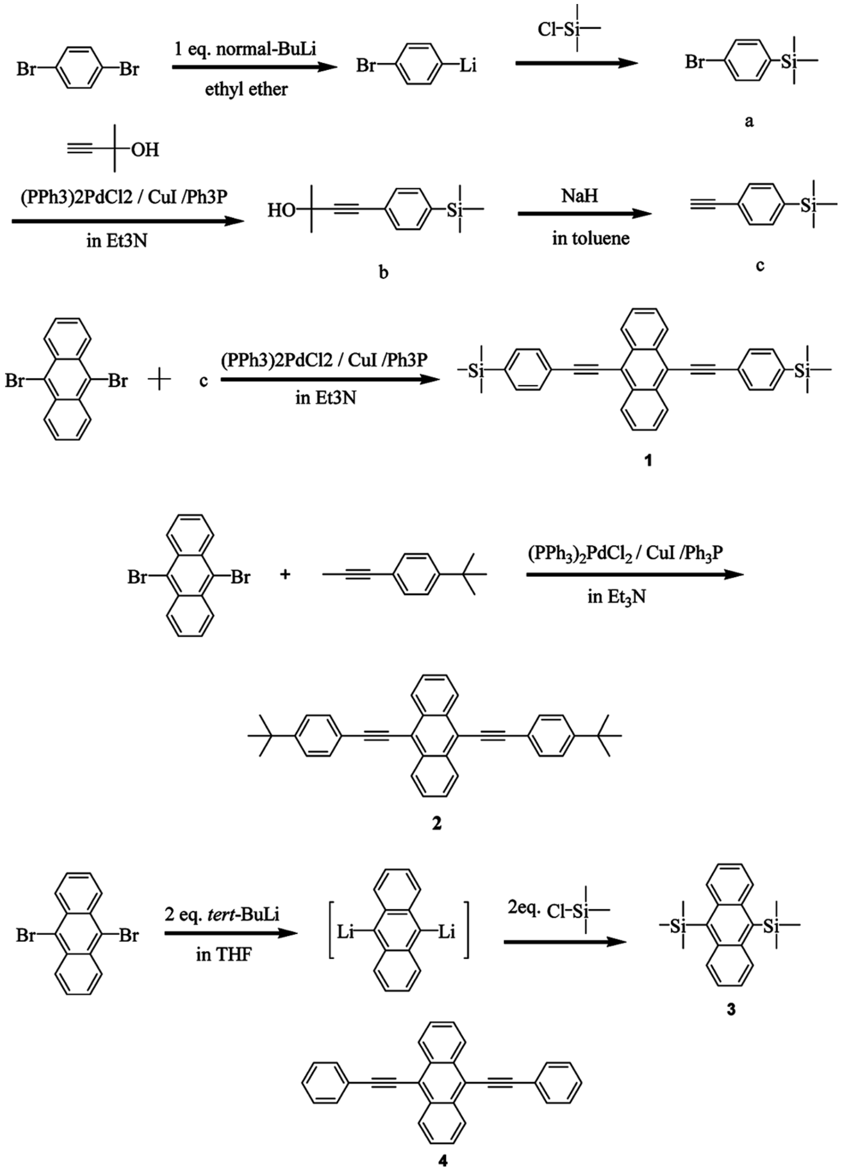
2. Synthesis of Emitting Materials

The synthesis of new anthracene derivatives for application in flexible OLED devices are shown in Scheme 2.

2.1. *9,10 bis(2-(4-(trimethylsilyl)phenyl)ethynyl)anthracene (1)*

(a) 4-bromotrimethylsilylbenzene. A 500 ml three-necked flask equipped with a three-way stopcock was flushed with nitrogen, and 1.4 dibromobenzene (30 g, 127 mmol) and dry diethyl ether were placed into the flask. To the solution, n-butyllithium (80 ml) was added dropwise at 0°C and stirred for 2 ~ 3 hr. Then chlorodimethylsilane (19.5 ml, 152 mmol) was added drop-wise at 0°C in the mixture and stirred for 3 hr. The reaction was quenched with saturated ammonium chloride aqueous solution. The mixture was washed with brine and the organic layer was dried with anhydrous magnesium sulfate. After removal of solvent the product was purified by vacuum distillation (90°C, 10 mmhg). The Product 4-bromo trimethylsilylbenzene was

obtained as colorless oil in 54% yield. ^{1}H-NMR (CDCl$_3$), δ: 0.26 (s, 9H, si(CH$_3$)$_3$), 7.40 (b, 4H, Ar), FT-IR (KBr): 1576, 1482, 1254, 1108, 810 cm^{-1}.



SCHEME 2 Synthesis of 9,10-disubstituted anthracene derivatives **1–3**.

(b) 4-(3-hydroxy-3-methyl-butynyl) trimethylsilyl-benzene. A 250 ml, round-bottom flask was equipped with a reflux condenser and was then flushed with dry nitrogen. Triethylamine (50 ml), triphenylphosphine (0.240 g, 0.92 mmol), copper(I)iodide (0.105 g, 0.55 mmol), bis(triphenylphospine)palladium(II)chloride (129 g, 0.18 mmol) and 2-methyl-3-butyn-2-ol (9.15 ml, 91 mmol) were placed in the flask, and the mixture was stirred for 1 hr. Then a solution of 4-bromotrimethylsilylbenzene (14 g, 61 mmol) in triethylamine (50 ml) was added and stirring was continued for 24 hr at refluxed temperature. After the triethylamine in the reaction mixture was evaporated, diethyl ether (ca. 300 ml) was added, and the insoluble salt was removed by filteration. The solution was washed with 1N hydrochloric acid and then with water. The organic phase was dried over anhydrous magnesium sulfate overnight, and diethyl ether was evaporated. The crude product was purified by column chromatography using silica gel and hexane-ethyl acetate (9:1 v/v) as eluent, then dried over vacuum completely to give the desired product (7 g), as a white solid in the 50% yield. ^{1}H-NMR ($CDCl_3$), δ: 0.25 (s, 9H, si(CH_3)$_3$), 1.55 (s, 6H, C(CH_3)$_2$), 2.86 (b, 1H, OH), 7.33 (b, 4H, Ar), FT-IR (KBr): 3344, 2232, 1598, 1504, 1258, 1110, 810 cm^{-1}.

(c) 4-trimethylsilylphenylacetylene. After a 250 ml round-bottom flask was flushed with drynitrogen, a solution of 4-(3-hydroxy-3-methyl-1-butynyl) trimethylsilylbenzene (6.5 g, 279 mmol) in toluene (50 ml) was dissolved sufficiently, and sodium hydride (0.17 g, 60% dispersion in mineral oil) was added and then the mixture solution was heated to 100°C and stirred for 5 hr. After completion of reaction, the reaction mixture was filtered with cellite to remove insoluble salt and impurities. The toluene solvent was evaporated as completely as possible. The crude product was purified by silica gel column chromatography using hexane-ethyl acetate (9:1 v/v) as eluent to give the desired product (3 g) as a orange color liquid in 61% yield. ^{1}H-NMR ($CDCl_3$), δ: 0.27 s, 9H, si(CH_3)$_3$), 2.94 (s, 1H, CH$\equiv$), 7.44 (b, 4H, Ar), FT-IR (KBr): 3308 (CH$\equiv$), 2112 (C$\equiv$C), 1598, 1492, 1254, 1104, 822 cm^{-1}.

(d) 9,10-bis(2-(4-(trimethylsilyl)phenyl)ethynyl)anthracene. This compound was prepared by Sonogashira reaction. A Schlenk tube was equipped with a reflux condenser and was then flushed with dry nitrogen. Triethylamine (40 ml), triphenylphosphine (0.060 g, 0.23 mmol), copper(I)iodide (0.066 g, 0.34 mmol), bis(triphenylphospine) palladium (II)chloride (0.040 g, 0.06 mmol) and 4-trimethylsilylphenylacetylene (3 g, 17 mmol) were placed in the tube and the mixture was stirred for 1 hr. Then a solution of 9,10 dibromoanthracene

(1.9 g, 5.7 mmol) in triethylamine (40 ml) was added and stirring was continued for 24 hr at refluxed temperature. The mixture is filtered and the triethylamine was evaporated. For the extraction of the reaction product diethyl ether (ca. 200 ml) was added and then the solution was washed with 1N hydrochloric acid and water. The organic phase was dried over anhydrous magnesium sulfate overnight. The solvent was evaporated, and the residue was passed through a short column of silica gel (hexane: ethyl acetate = 9:1). Recrystallization of eluted materials with hexane gave 9,10-bis(2-(4-(trimethylsilyl)phenyl) ethynyl)anthracene (0.3 g) as orange needle crystals in 10% yield. ^{1}H-NMR ($CDCl_3$), δ: 0.31 (s,9H, si$(CH_3)_3$), 7.74 (b, 4H, Ar), 7.62, 8.68 (b, 8H, An), FT-IR (KBr): 2952, 1589, 1247, 1105, 846, 765 cm^{-1}.

2.2. *9,10-bis(2-(4-(tert-butyl)phenyl)ethynyl)anthracene (2)*

Triethylamine (40 ml), triphenylphosphine (0.040 g, 0.15 mmol), copper(I)iodide (0.043 g, 0.23 mmol) and bis(triphenylphospine) paliadium (II)chloride (0.027 g, 0.04 mmol) were placed in the round-bottom flask flushed with drynitrogen, and the mixture was stirred for 30 min. After 4-tert-butylphenylacetylene was added and stirring was continued for 1 hr and then a solution of 9,10 dibromoanthracene (1.82 g, 3.8 mmol) in triethylamine (40 ml) was added and stirring was continued for 48 hr at refluxed temperature.

The mixture was filtered and the triethylamine was evaporated. For the extraction of the reaction product, diethyl ether (ca. 200 ml) was added and then the solution was washed with 1N hydrochloric acid and water. The organic phase was dried over anhydrous magnesium sulfate overnight. The solvent was evaporated and the residue was passed through a short column of silica gel (hexane: Ethyl acetate = 20:1). Recrystallization of eluted materials with hexane gave 9,10-bis(2-(4-(tert-butyl)phenyl)ethynyl)anthracene (0.5 g) as gold crystals in 27% yield. ^{1}H-NMR ($CDCl_3$), δ: 1.38 (s, 18H, si$(CH_3)_3$), 7.47, 7.72 (b, 8H, Ar), 7.62, 8.68 (b, 8H, An), FT-IR (KBr): 3058, 2958, 2190, 1508, 1261, 1106, 825 cm^{-1}.

2.3. *9,10 bis(trimethylsilyl)anthracene (3)*

This compound was synthesized according to the reported process [17]. The silylated anthracene derivative (1.5 g) was obtained as yellow crystal in 35% yield.

Mp: 115 ~ 117°C, ^{1}H-NMR ($CDCl_3$, 400 MHz), δ:0.67 (s, 18H, si$(CH_3)_3$), 7.39 (dd, 4H, Ar), 8.37 (dd, 4H). FT-IR (KBr pellets): 3074, 2948, 2894, 1512, 1438, 1406, 1250, 1162, 956, 840, 772, 682 cm^{-1}.

2.4. *Measurement*

Infrared spectra were obtained with a JASCO FT-IR 620 spectrometer. ^{1}H-NMR spectra were recorded on a Bruker 400 NMR spectrometer with chloroform-d ($CDCl_3$) as a solvent and tetramethylsilane (TMS; $\delta = 0$) as an internal reference. The UV/vis spectra and fluorescence spectra of synthesized products were measured on a JASCO V-650 spectrophotometer and JASCO FP-6500 fluorescence spectrometers, respectively. Thermal stability of the compounds was evaluated with a Seiko SSC5200H Disk Station (DSC220C) under dry nitrogen at a heating rate of 20°C/min.

2.5. *Device Fabrication and Electro-Optical Properties*

OLED devices were fabricated with the synthesized products as the emitting layer on the ITO coated glasses and PES (polyethersulphone) films as substrates. The organic compounds were deposited on the ITO substrate through a mask by vacuum evaporation at pressure below 10^{-8} Torr. The rate of deposition of the emissive layer was controlled to 0.2 Å/s, and emission area of the devices was 4 mm^2. The OLED devices with configuration of ITO/2-TNATA/□-NPD/compound 1,2 or 4/BCP/Alq_3/LiF/Al were fabricated with layer thickness of 1500/100/150/300/150/100/10/1500 Å thickness, respectively. The EL spectra and luminance/current density/voltage characteristics of the OLED devices were measured by using a Spectra Scan PR 650 spectrophotometer and a computer controlled voltage-current source (Keithley 236) at room temperature and the ambient condition.

RESULTS AND DISCUSSION

1. Synthesis and Characterization

The anthracene compounds used in the OLED devices are outlined in Scheme 1. The specific synthetic routes of the anthrancene delivatives (**1**–**4**) are shown in Scheme 2. Compound **1** was obtained through a slightly modified process from the reported one up to 3rd step [18–20] and Sonogashira reaction was used in the 4th step with $(PPh_3)_2PdCl_2$ as a catalyst. The final product was difficult to get by recrystallization in hexane because needle like crystal was very small in size, thus resulting in 10% low yield. Compounds **2** and **3** were obtained by 1step reaction as shown in Scheme 2 giving gold and yellow crystals in moderate yield. The compound **4** was obtained commercially.

The FT-IR spectra of synthesized anthracene derivatives **1**, **2** and **3** are shown in Figure 1. The IR spectra of compound **1** and **3** exhibited strongly peak at 1250 and 835 cm^{-1}, confirming the presence of trimethylsilyl group connected through phenylethynyl group to the

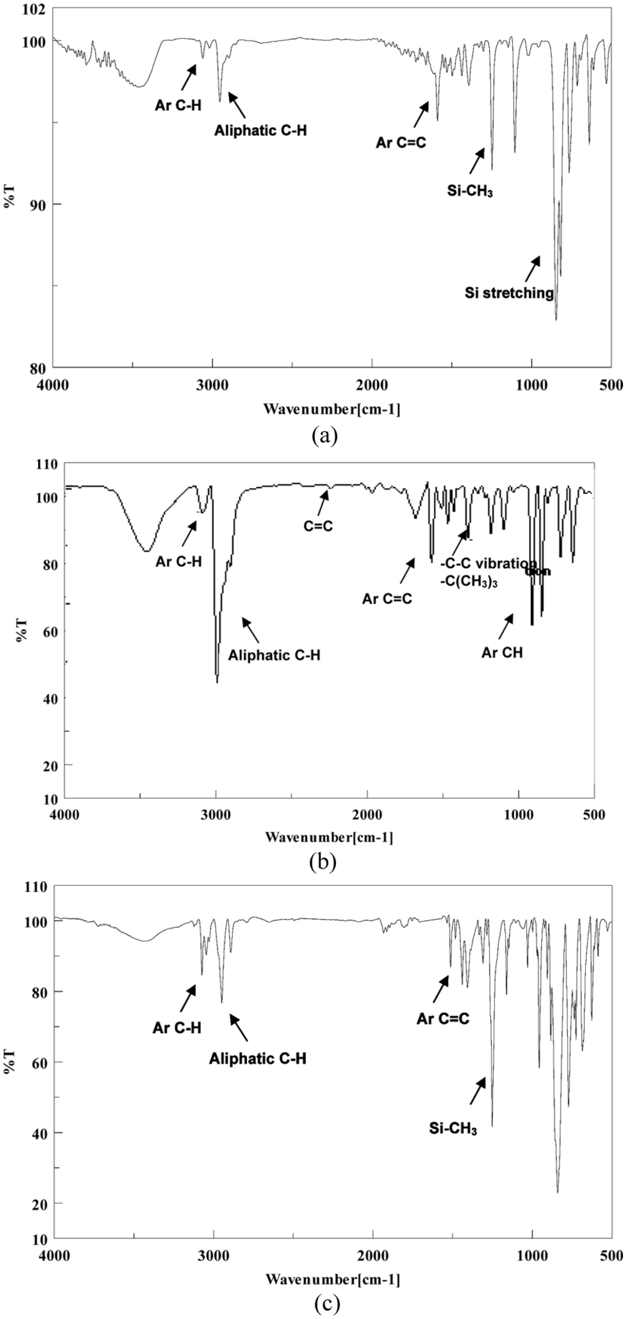


FIGURE 1 FT-IR spectra of anthracene derivatives **1–3**.

anthracene core. In the case of compound **2**, weak (C≡C) triple bond was exhibited at 2190 cm^{-1}, as well as strong aliphatic C–H peaks at 2958 cm^{-1} and C–C skeletal vibration of tert-butyl group at 1261 cm^{-1}.

The synthesis of compound **1–3** was also checked by ^{1}H-NMR as shown in Figure 2. The compound **1** and **2** exhibited 18 protons in trimethylsilyl 0.36 ppm and ter-butyl 1.38 ppm group, respectively. In addition, they exhibited characteristic H peaks of phenyl at 7.44, 7.47 and 7.72 ppm and anthracene 7.32, 7.67 ppm and 7.62, 8.68 ppm at units. In case of compound **3** trimethylsilyl protons appeared at 0.70 ppm as well as protons of anthracene at the 7.44, 8.40 ppm.

2. Electro-Optical Properties of Compound 1–4

The UV-visible absorption spectra of anthracene and anthracene derivatives **1–4** are shown in Figure 3. The measurement was conducted in dilute THF solution in which UV absorbance was controlled under 1.0. The UV absorption maxima ($\lambda_{max,abs}$) of anthracene derivatives (**1**, **2** and **4**) with similar skeletal structures exhibited two absorption maxima at (442, 467), (442, 464) and (437, 461) nm, respectively, which were shifted to longer wavelengths compared with that of anthracene (358 nm) due to the introduction of electron donating group such as phenyl acetylene- and trimethylsilyl group to the 9, 10 position of anthracene. The front absorption may be caused by π-π^* transitions in the anthracene core and the rear peaks by π-π^* transition of the side chains.

The cut-off wavelengths of anthracene and compounds **1–4** were 392 and 485, 483, 425, 479 nm, respectively. When those compounds were excited at the $\lambda_{max,abs}$, they showed the fluorescence peaks ($\lambda_{max,em}$) in the blue or greenish blue region of 401 and 478, 481, 438, 475 nm, respectively.

Figure 4 shows the normalized fluorescence spectra of the anthracene and derivatives (**1–4**) in which excitations were conducted at various UV absorption peaks. These results indicated that the synthesized anthracene derivatives had monochromatic property, that is, same photoexcited states regardless of excitation wavelengths. In case of anthracene derivative **1**, the excitation spectrum started about 380 nm while absorption started from 350 nm. This may be the explained by the σ-π interaction in the silicon and phenylacetylene π-conjugated system.

The fluorescence quantum yields (Φ_s) of anthracene and derivatives **1–4** in dilute THF solution were measured and results are listed Table 1. The quantum yields was calculated by absorbance value and realative integrated fluorescence intensity. Quinine sulfate in 1N H_2SO_4 solution was used as the standard. The quantum yields anthracene derivatives THE solution was calculated by the following

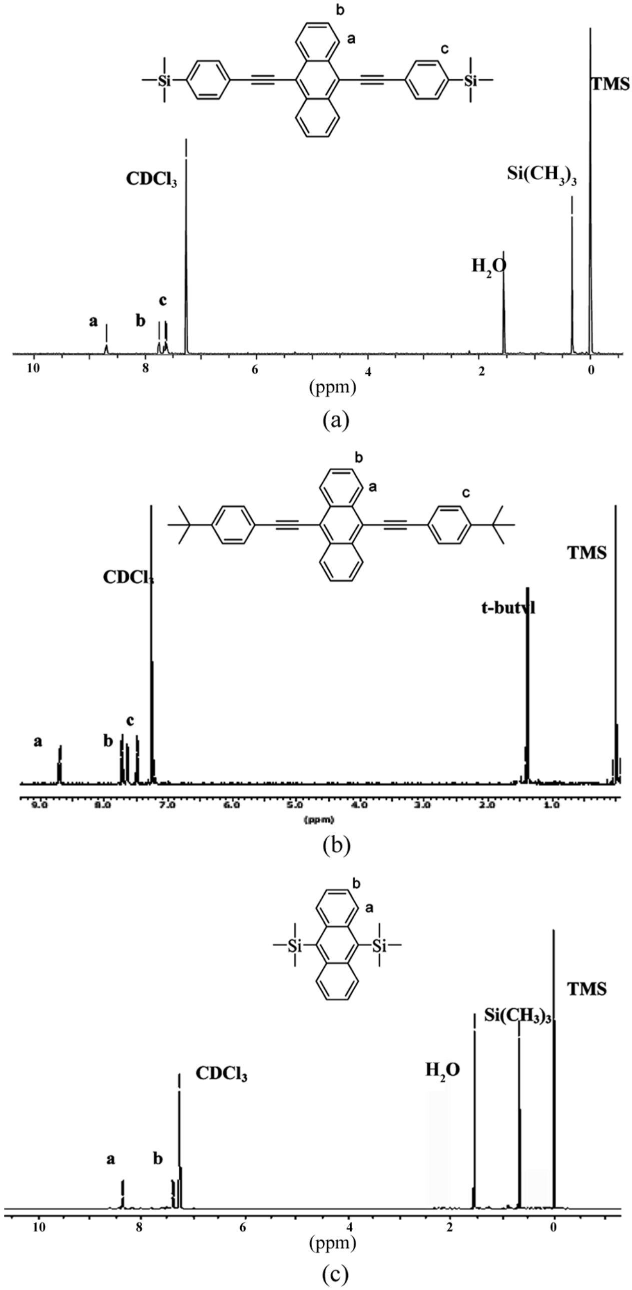


FIGURE 2 The ^{1}H-NMR spectra of anthracene derivatives **1–3**.

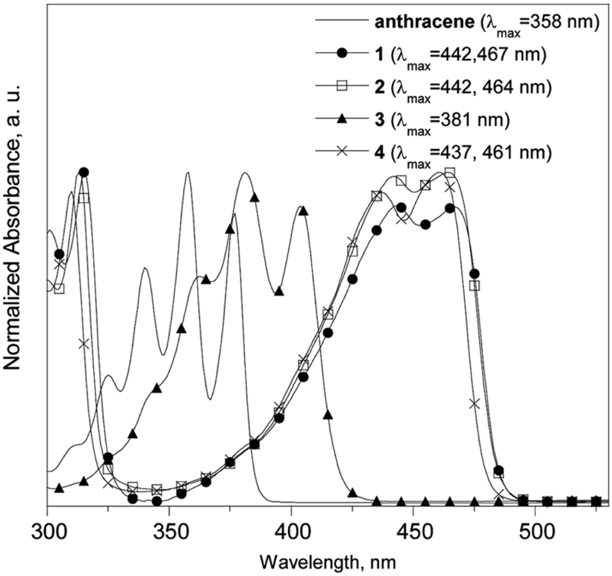


FIGURE 3 The UV-visible spectra of anthracence and anthracene derivatives **1–4**.

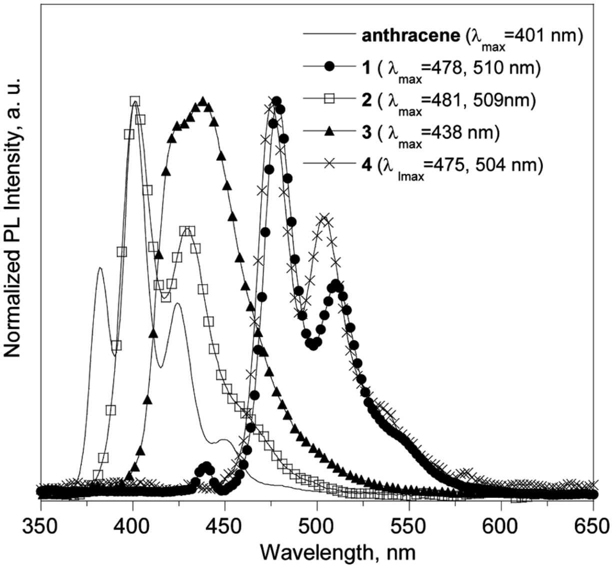


FIGURE 4 The Fluorescence spectra of anthracence and anthracene derivatives **1–4**.

TABLE 1 Physical and Optical Properties of Anthracene Derivatives

Compound	T_m (°C)	$\lambda_{(max,asb.)}$	$PL_{(max,em)}$	Φs
Anthracene	217	358	401	0.19
1	274	442, 467	478, 510	0.77
2	250	442, 464	481, 509	0.66
3	117	381	438	0.22
4	250	434, 461	475, 504	0.68

$^*\lambda_{max,asb}$: The wavelength of the maximum absorbance value.

$^*\lambda_{max,em}$: The wavelength of the maximum fluorescence.

$^*\Phi_s$: Quantum yield of samples in solution.

equation where the reference quantum yield Φ_r was 0.546 at the excitation wavelength 365 nm [22].

$$\Phi_s = \Phi_r(A_rF_s/A_sF_r)\ (n_s^2/n_r^2) \tag{1}$$

The A and F values are absorbance and integrated fluorescence intensity parameters of the sample and reference solution and n_s and n_r are the refractive index value of THF and 1N H_2SO_4 solution. The excitation wavelengths of anthracene and derivatives **1–4** were 358 and 467, 464, 430, 461 nm, respectively. As shown in Table 1, the end-capping of π-conjugated system with silyl group was found to increase the quantum yield, which may be due to the increase of electron density through the weak silicon σ-π conjugate system.

The anthracene derivatives also exhibited improved thermal stability as shown in Table 1. Compound **1** had higher melting temperature of 274°C compared to anthracene 210°C presumably due to confinement of π-conjugated system by trimethylsilyl end-capping, which will be beneficial in the high temperature operation condition of OLED devices. The energy band gaps and HOMO/LUMO levels of anthracene derivatives measured by AC-2 photoelectrometer and UV absorption edges are shown in Table 2 and Figure 5. It was also noted that compound **1** had slightly narrower band gap than compounds **2** and **4** due to σ-π conjugation mentioned above.

TABLE 2 Electro-Optical Properties of Anthracene Derivatives

Compound	HOMO (eV)	LUMO (eV)	$\lambda_{max,EL}$	Turn-on voltage	Luminance (max cd/m^2/volt)	CIE coordinate (x, y)
1	5.54	3.02	496	3	1,886	0.21, 0.32
2	5.41	2.85	492	4	1,143	0.22, 0.38
4	5.44	2.88	484, 524	4.75	4,754	0.26, 0.43

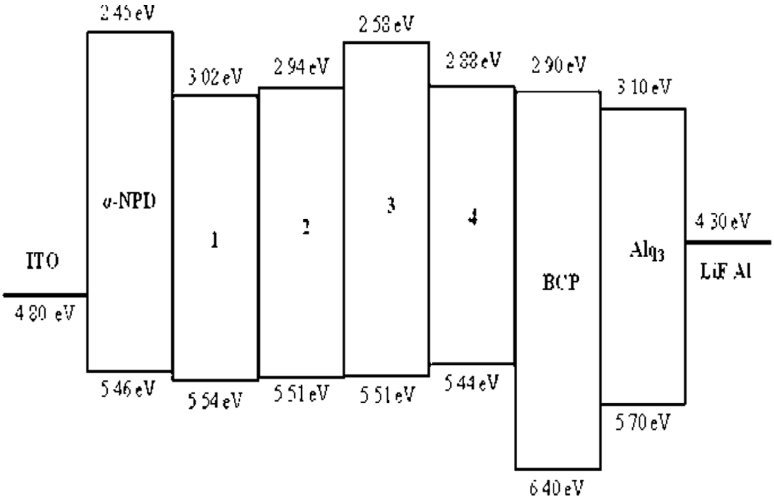


FIGURE 5 Energy band diagram of the compounds used in OLED devices.

3. Electroluminescent Properties

Anthracene derivatives were tested as emitting layer in the OLED devices with configuration of ITO/α-NPD (150 Å)/compound **1**, **2** or **4** (300 Å)/BCP (150 Å)/Alq3 (100 Å)/LiF (10 Å)/Al. The electroluminescence

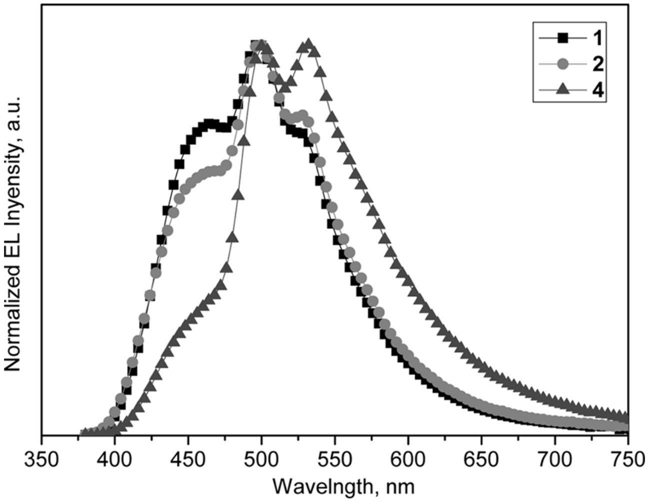
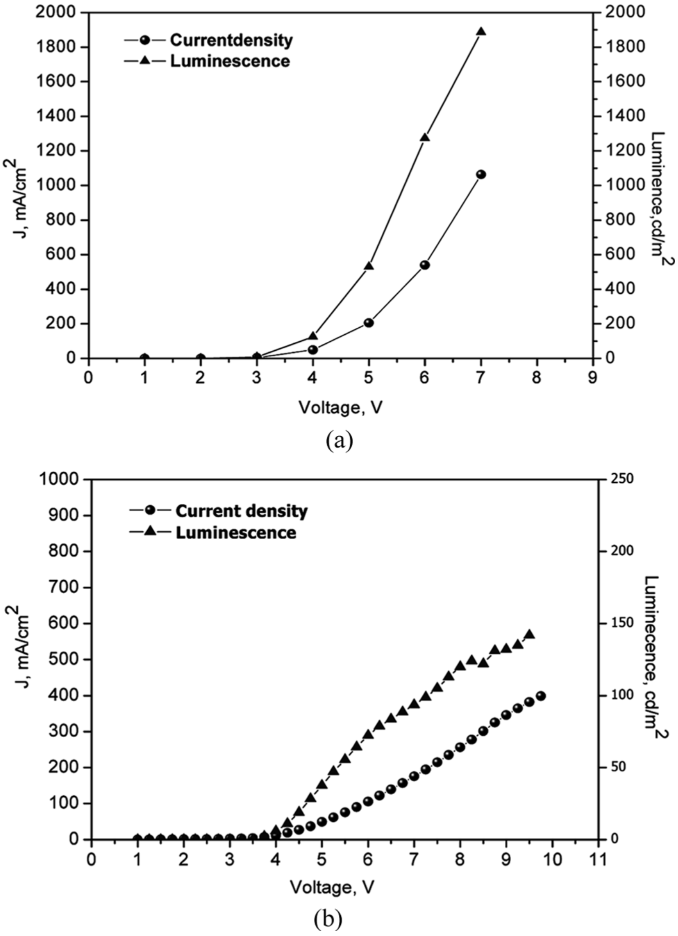


FIGURE 6 Electroluminescence spectra of the compound **1**, **2** and **4** in the OLED device.

(EL) spectra of anthracene derivatives (**1**, **2**, **4**) exhibited different emission peaks as shown in Figure 6. Derivatives **1** and **2** showed $\lambda_{\text{max,EL}}$ in the blue region at 496 nm and 492 nm which were shifted from the $\lambda_{\text{max,PL}}$ at 478 nm and 481 nm, respectively due to the aggregate effect. The derivative **4** showed $\lambda_{\text{max,EL}}$ at 532 nm which was shifted quite large from $\lambda_{\text{max,PL}}$ at 475 nm.

This suggests that derivative **4** can make aggregates easily compared to **1** and **2** which have bulky substituents at both ends of anthacene π-conjugate system. The relatively low luminance intensity



(a)

(b)

FIGURE 7 Efficiency-luminance-voltage characteristics of compound **1** in the OLED devices with (a) ITO glass and ITO-PES film as substrate.

of derivatives **1** and **2** compared to **4** suggested that the former had lower electron mobilities than derivative **4** as shown in Table 2.

The anthracene derivative **1** exhibited easy evaporation property in the OLED device fabrication. Therefore derivative **1** was used in the flexible OLED devices which usually require mild deposition condition. The J-V-L curves of OLED with derivative **1** as emitting layer on the ITO glass and ITO coated PES (polyether sulfone) film substrate are shown in Figure 7. The Flexible OLED made with derivative **1** on PES film showed lower luminance value compared to glass substrate. This was considered to be due to unoptimized ITO deposition condition on PES substrate and higher surface roughness of ITO-PES film compared to the ITO glass. The derivative **1**, however, showed potential as a good emitting material for the flexible OLED devices.

CONCLUSIONS

We have successfully synthesized a silylated anthracene derivative **1** as an emitting material for the flexible OLED devices. It was obtained by a Sonogashira reaction and possessed higher fluorescence quantum yields in solution. As compared to the commercially available blue-light emitting, material 4 and tert-butylated compound **2**, the compound **1** showed small red-shift of 9 nm in the OLED emission from the by PL $\lambda_{\max}$ due to the increased electron density in the antheacene ring.

The compound **1** was also found to be easily fabricated in the flexible OLED devices and showed a low threshold voltage and high current efficiency compared with other anthracene derivatives.

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