

# Solution-Processible Blue-Light-Emitting Polymers Based on Alkoxy-Substituted Poly(spirobifluorene)

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Alkoxy-substituted poly(spirobifluorene)s and their copolymers with a triphenylamine derivative have been synthesized by Ni(0)-mediated polymerization. The polymers were well soluble in common organic solvents. Pure blue-light emissions without the long wavelength emission of poly(fluorene)s have been observed in the fluorescence spectra of polymer thin films. The light emitting diodes with a device configuration of ITO/PEDT:PSS(30 nm)/polymer(60 nm)/LiF(1 nm)/Al(100 nm) have been fabricated. The electroluminescence spectra showed the blue emissions without the long wavelength emission as observed in the fluorescence spectra. The relatively poor electroluminescence quantum yield of the homopolymer (0.017% @ 20 mA/cm<sup>2</sup>) with color coordinates of (0.16, 0.07) has been improved by the introduction of triphenylamine moiety, and the copolymer with triphenylamine derivative exhibited an electroluminescence quantum yield of 0.15 % at 20 mA/cm<sup>2</sup> with color coordinates of (0.16, 0.08). Moreover, the introduction of polar side chains to the spirobifluorene moiety enhanced the device performance and led to the quantum yields of 0.6 to 0.7 % at 20 mA/cm<sup>2</sup>, although there was some expense of color purities.

**Keywords:** PLED, poly(fluorene), spirobifluorene, display.

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## I. Introduction

Organic electroluminescent materials have recently attracted much interest due to their physical properties and potential applications in light-emitting devices and flat panel displays [1]-[3]. The development of new materials displaying proper color with high efficiency and stability is essential for the development of a full color display. Polyfluorenes are promising new materials for blue-light-emitting diodes because of their high photoluminescence (PL) quantum efficiencies and thermal stability [4]-[6]. However, the troublesome long wavelength emission in the polymer films of polyfluorenes when heating during device formation or operation has been the crucial problem for practical applications. One of the possible origins for the long wavelength emission has been known as the formation of an excimer [7], [8]. There have been several approaches to suppress excimer emission including copolymerization or end-capping with anthracene, the attachment of bulky aryl groups at the 9-position of the fluorene, and so on [7]-[10].

The spiro structure in spirobifluorene is also believed to be an effective way to reduce the interchain interaction that leads to a broadening of the emission spectrum. It has been reported that very efficient and thermally stable devices were fabricated using some oligomers based on spirobifluorene [11], [12]. A 9,9'-spirobifluorene-based conjugated polymer, poly(9,9'-spirobifluorene-2,7-diyl) to suppress the excimer emission has been reported by Kreuder and others [13]. Yu and others have prepared the alternating copolymer of 9,9'-spirobifluorene and fluorene, which showed a blue emission with improved solubility in common organic solvents [14]. Recently, Vak and others reported on a novel spiro-functionalized polyfluorene

derivative, which has showed a stable blue emission [15].

Another origin of the long wavelength emission of polyalkylfluorene derivatives has been claimed by List and coworkers. They claimed that the long wavelength emission of the polyalkylfluorene could be caused by a keto-defect on the polymer chain [16]. The keto effect has also been studied at the end of polyfluorene, and the long wavelength emission has been observed in polyfluorene end-capped with 9-fluorenone [17]. The certain origin of the long wavelength emission of polyalkylfluorene is not yet clear. Both the excimer and the keto-defect may be attributed to the long wavelength emission. Regardless of the origin of the additional long wavelength emission, the importance of synthesizing stable blue-light-emitting polyfluorene with good processibility has increased for the realization of a polymer electroluminescence (EL) display.

Here, we report on the synthesis and characterization of alkoxy-substituted poly(spirobifluorene), poly(4',7'-di-*n*-octoxy-9,9'-spirobifluorene-2,7-diyl) (SF8) as a blue-light-emitting polymer with good solubility and its copolymer with triphenylamine moiety, and on poly(4',7'-di-*n*-octoxy-9,9'-spirobifluorene-2,7-diyl-*co*-4-*n*-butylphenyl-diphenylamine-4',4''-diyl) (SF8TPA) to achieve a charge balance through improving the hole injection in a polymer light-emitting diode. Moreover, we have also introduced a spirobifluorene unit containing a polar side chain of the 3,5-dioxaheptyl group into the above polymer, which might be expected to increase device performance through the improved interface adhesion with the electrodes.

## II. Experimental Section

### 1. Instrumentation

Nuclear magnetic resonance spectra were recorded on a Bruker AM 300 spectrometer with tetramethylsilane as an internal reference. Using a Dupont 9900 analyzer, a thermo gravimetric analysis and differential scanning calorimetry of polymers were performed under a nitrogen atmosphere at a heating rate of 10 °C/min. The number and weight average molecular weights of polymers were determined by gel permeation chromatography analysis on a Waters GPC-150C instrument, using tetrahydrofuran (THF) as an eluent and polystyrene as a standard. UV-visible spectra were measured using a Hitachi UV3501, and photoluminescence spectra were measured using a Spex Fluorolog-3 Spectrofluorometer at room temperature. Cyclic voltammetry measurement was performed on an AUTOLAB/PGSTAT12.

### 2. Monomer Preparation

The monomer, 2,7-dibromo-3',6'-bis-*n*-octyloxy-9,9'-

spirobifluorene, was synthesized following the method in our previous report [18]. We synthesized 2,7-dibromo-3',6'-bis(3,5-dioxaheptyl)-9,9'-spirobifluorene following the similar preparation method for 2,7-dibromo-3',6'-bis-*n*-octoxy-9,9'-spirobifluorene. We prepared 4-*n*-Butylphenyl-bis(4'-bromophenyl)amine according to the previous paper [19]. We purchased 9,10-dibromoanthracene and 9-bromoanthracene from Aldrich Chemical Co., which we used with purification following the literature methods [20].

### 3. Polymerization

A polymer material, poly(3',6'-di-*n*-octoxy-9,9'-spirobifluorene-2,7-diyl) (SF8), was prepared through Ni(0) mediated polymerization. A Schlenk tube containing 6 mL of toluene, 6 mL of dimethylformamide, bis(1,5-cyclooctadienyl) nickel (0), 2,2'-bipyridyl and 1,5-cyclooctadiene (the latter three in molar ratios, 1:1:1) was heated under nitrogen to 80 °C for 0.5 hr. The monomer (1.35 mmol) dissolved in 6 mL of degassed toluene (molar ratio of monomers and end capper/nickel complex = 0.57) were added to the above solution and the polymerization was maintained at 80 °C for 24 hours. An excess of 9-bromoanthracene was added and the solution was maintained at 80 °C for 12 hours. After a reaction, the polymers were precipitated from an equivolume mixture of concentrated hydrochloric acid, methanol, and acetone. The crude product was collected, dissolved in CHCl<sub>3</sub>, and then reprecipitated from methanol. Residual impurities were removed by extraction with acetone in a Soxhlet apparatus, and finally the polymers were dried under a vacuum. The other homopolymer of poly(3',6'-bis(3,5-dioxaheptyl)-9,9'-spirobifluorene-2,7-diyl) (SF7O2) and the copolymers, SF8TPA and poly(3',6'-bis(3,5-dioxaheptyl)-9,9'-spirobifluorene-2,7-diyl-*co*-4-*n*-butylphenyl-bisphenylamine-4',4'-diyl) (SF7O2TPA) were also prepared following the above polymerization condition with 2,7-dibromo-3',6'-di-*n*-octoxy-9,9'-spirobifluorene, 2,7-dibromo-3',6'-bis(3,5-dioxaheptyl)-9,9'-spirobifluorene and 4,4'-dibromophenyl-4'-*n*-butylphenylamine (yields > 90%). A 10 mol.% of 4,4'-dibromophenyl-4'-*n*-butylphenylamine was used for the copolymers.

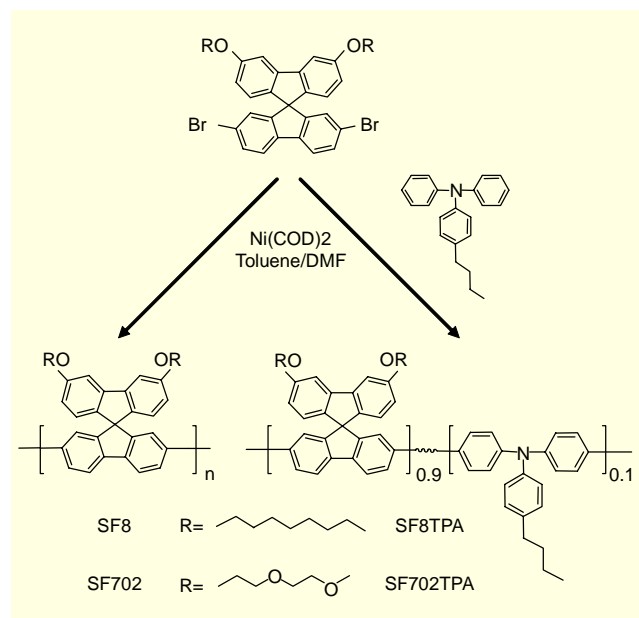
### 4. Device Fabrication and Characterization

A polymer device was fabricated using the spin coating method and a vacuum deposition of the metal electrode. The device structure was ITO/PEDT:PSS(30 nm)/polymer(60 nm)/LiF(1 nm)/Al(100 nm). The PEDT:PSS (Baytron AI 4083) was purchased from Bayer and used as a hole injection layer. Electroluminescence spectra were obtained with a Minolta CS-1000. The current/voltage and luminescence/voltage characteristics were taken with a current/voltage source/measure

unit (Keithley 238) and a Minolta LS-100.

### III. Results and Discussion

The molecular structures of the new polyfluorenes are shown in Scheme 1. We have already reported on the synthesis of the monomer, 2,7-dibromo-4',7'-di-*n*-octoxy-9,9'-spirobifluorene, which was used as an intermediate material of a blue-light-emitting small molecule, 2,7-bis(4-biphenyl)-4',7'-di-*n*-octoxy-9,9'-spirobifluorene [18]. Ni(0) mediated polymerization was also carried out using the above monomer and the comonomer of 4-*n*-butylphenyl-bis(4'-bromophenyl)amine. Moreover, we have introduced polar side chains of 3,5-dioxaheptyl into the above spirobifluorene monomer instead of normal octyl. The incorporation of fluorene monomer containing a polar side chain



Scheme 1. Synthetic scheme and structures of the polymers.

such as 3,5-dioxaheptyl has improved device performance, which might be explained by the increased interface adhesion with the polar electrodes [21], [22]. Therefore, we have expected to improve device performance using spirobifluorene containing polar side chains. We used 9-bromoanthracene as the end-capping reagent [7], [8].

We expected that the alkoxy group would help give much better solubility, and in fact, the alkoxy substituted spirobifluorene compounds synthesized in this work were readily soluble in common organic solvents such as THF, chloroform, toluene, etc. The molecular weight was measured by gel permeation chromatography with a THF eluent. The molecular weights of the homopolymers and the copolymers ranged from Mn 15,000 to 20,000 with PDI 2.1 to 2.5.

The glass transition temperatures ( $T_g$ ) of the polymers were 300, 150, 144, and 175°C for SF8, SF8TPA, SF702, and SF702TPA, respectively, determined by differential scanning calorimetry at 10°C/min under a nitrogen atmosphere. Considering that the  $T_g$  of poly(9,9-di-*n*-octylfluorene-2,7-diyl) is about 75°C, the glass transition temperatures of the polymers are extraordinarily high, which might be attributed to the spirobifluorene moiety. The previous paper about spiro-functionalized polyfluorene also revealed that the incorporation of spirobifluorene contributed to an increase of the glass transition temperature [14]. The thermal stabilities of the polymers were excellent with decomposition temperatures (5% weight loss measured by TGA analysis, at 10°C/min) under a nitrogen atmosphere in excess of 400°C. Table 1 shows the polymer properties of the materials used in this study.

Along with the optical properties, electrochemical properties of the polymers were also investigated using cyclic voltammetry, from which the highest occupied molecular orbital (HOMO) of the polymers could be determined. HOMO levels are closely related to charge injection and transport properties of the conjugated polymers, so it is of importance to

Table 1. Polymer properties.

	SF8	SF8TPA	SF702	SF702TPA
Molecular weight (Mn/PDI) <sup>a</sup>	15,000/2.1	18,000/2.5	20,000/2.5	19,000/2.4
$\lambda_{max}$ absorption (nm) <sup>b</sup>	380	379	385	391
$\lambda_{max}$ emission (nm) <sup>b</sup>	430/450	440	425/450	447
Relative PL quantum efficiency <sup>c</sup>	0.58	0.32	0.39	0.32
HOMO level (eV)	5.75	5.37 / 5.70	5.57	5.27 / 5.62
Glass transition temperature, $T_g$ (°C)	300	150	144	175

a. measured by gel permeation chromatography (GPC) referenced to polystyrene standards

b. measured in thin films on quartz wafers

c. measured in CHCl<sub>3</sub> solution referenced to 9,9-diphenylanthracene

understand the correct electrochemical properties of the materials for electronic device applications. Electrochemical measurements were performed at room temperature with a three-electrode cell in a solution of  $\text{Bu}_4\text{NBF}_4$  (0.10 M) in acetonitrile at a scan rate of 50 mV/s. Polymer films were prepared by dipping platinum working electrodes into the polymer solutions and then air-drying them. A platinum wire was used as a counter electrode and an  $\text{Ag}/\text{Ag}^+$  electrode was used as a reference electrode. As shown in Table 1, the introductions of 4-*n*-butylphenyl-bis(4'-bromophenyl)amine into the main backbone and polar side group into the side chain reduced the HOMO level. Therefore, we could expect the enhanced hole injection between the anode and polymer layer in light-emitting diodes fabricated using these polymers.

Figure 1(a) shows the absorption and emission spectra of SF8 and SF8TPA films, and Fig. 1(b) shows those of SF7O2 and SF7O2TPA films. The absorption spectra of the films were similar to each other. The introduction of polar side chains to spirobifluorene side chains and the copolymerization with

triphenylamine comonomer have little effects on the band gap of the resulting polymers. The emission spectra of homopolymers, SF8 and SF7O2, are very similar to those of poly(alkylfluorene)s, which indicates that the introduction of spirobifluorene does not change the optical properties of poly(alkylfluorene)s. As for the copolymers, SF8TPA and SF7O2TPA, some change was observed and the emission maximum shifted to longer wavelength than in homopolymers. From these results, we believe that the origin of the emission in the copolymers is the triphenylamine containing unit rather than fluorene units.

A polymer light-emitting device was fabricated using the spin coating method and vacuum deposition of the metal electrode. The SF8 device showed a similar spectrum as the PL spectrum, which is also a typical EL spectrum of a blue-light-emitting polyfluorene; the EL maximum was 427 nm. Usually, the EL device of polyfluorene shows a broad additional band between 500 and 600 nm due to the long wavelength emission.

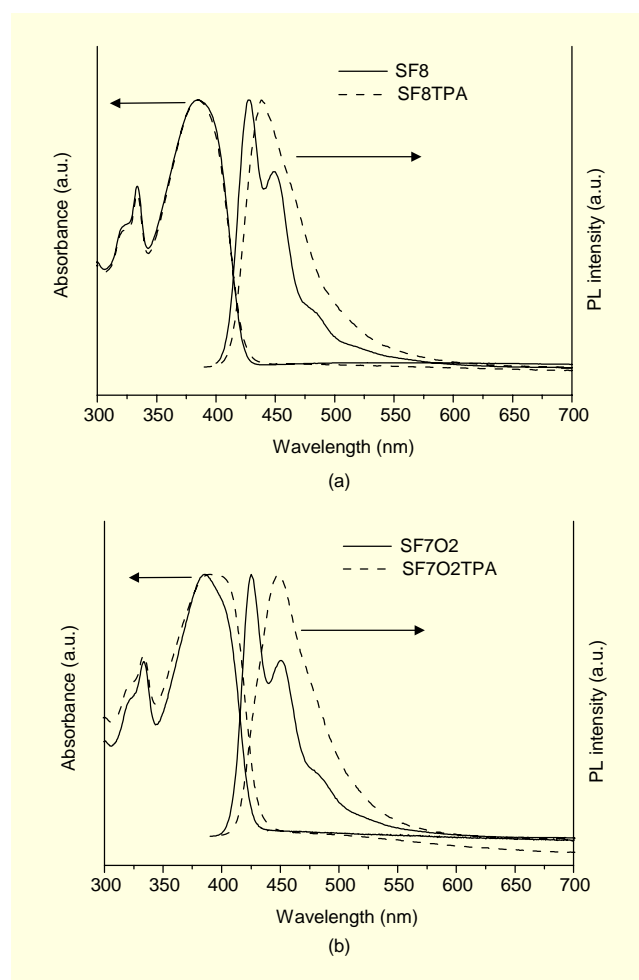


Fig. 1. Absorption and emission spectra of (a) SF8 and SF8TPA films and (b) SF7O2 and SF7O2TPA films.

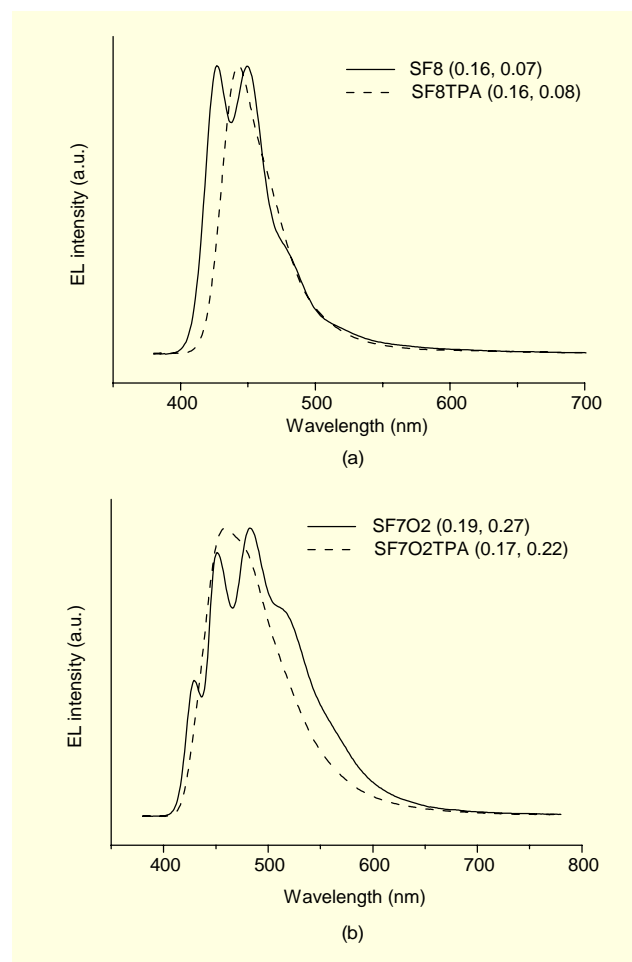


Fig. 2. Electroluminescence spectra of (a) SF8 and SF8TPA devices and (b) SF7O2 and SF7O2TPA devices. The device configuration was ITO/PEDT:PSS (30 nm)/polymer (60 nm)/LiF (1 nm)/Al (100 nm).

However, SF8 did not show any long wavelength emission in the EL spectrum. We believe that the spiro structure in SF8 might effectively prevent interchain or intrachain-electron overlap, which leads to a blue emission without the long

wavelength emission. The color coordinates in CIE chromaticity is (0.16, 0.07), which is in a pure blue region. The EL spectrum of SF8TPA was also similar to the PL spectrum, and the long wavelength emission was not observed. The color coordinates, (0.16, 0.08) in CIE chromaticity is almost the same as that of the homopolymer.

Figure 2(b) shows the EL spectra of the SF7O2 and SF7O2TPA devices. Broader emissions than their PL have been observed and sky blue lights were obtained. As SF8 and SF8TPA devices showed deep blue emissions, the spectral broadening should be attributed to the polar side chains of the 3,5-dioxaheptyl group. We believe that some phase separation between non-polar main chains and polar side chains might have an affect on the spectral properties. This kind of spectral broadening has also been observed in a poly(alkylfluorene) derivative [23].

The maximum external quantum efficiency of SF8 was 0.017%. Previously, the poor quantum efficiency of polyfluorene has been improved by blending with hole transporting triarylmines due to the improved hole injection [24]. The end capping, or the copolymerization with the triarylmines or triarylamine-containing blocks, is another approach to improve device performance [25], [26]. Here, we adapted the previous approach of copolymerization with a triarylamine-containing block, which was 4-*n*-butylphenyl-bis(4'-bromophenyl)amine in our case. As shown in Fig. 3(a), the slope of the current density-luminance relationship has greatly increased, and the external quantum efficiency of the copolymer was improved by about 10 times at the same device configuration. The maximum external quantum efficiency of SF8TPA was 0.20 %.

The device efficiency has been further improved in the polymers containing polar side chains, SF7O2 and SF7O2TPA. As shown in Fig. 3(b), the external quantum efficiencies reached 0.7 %, which were more than 3 times higher than SF8TPA. Moreover, they exhibited a 10 times higher maximum luminance than SF8TPA. These enhanced device performances might be explained by interface adhesion improvements between light emitting polymer and electrodes.

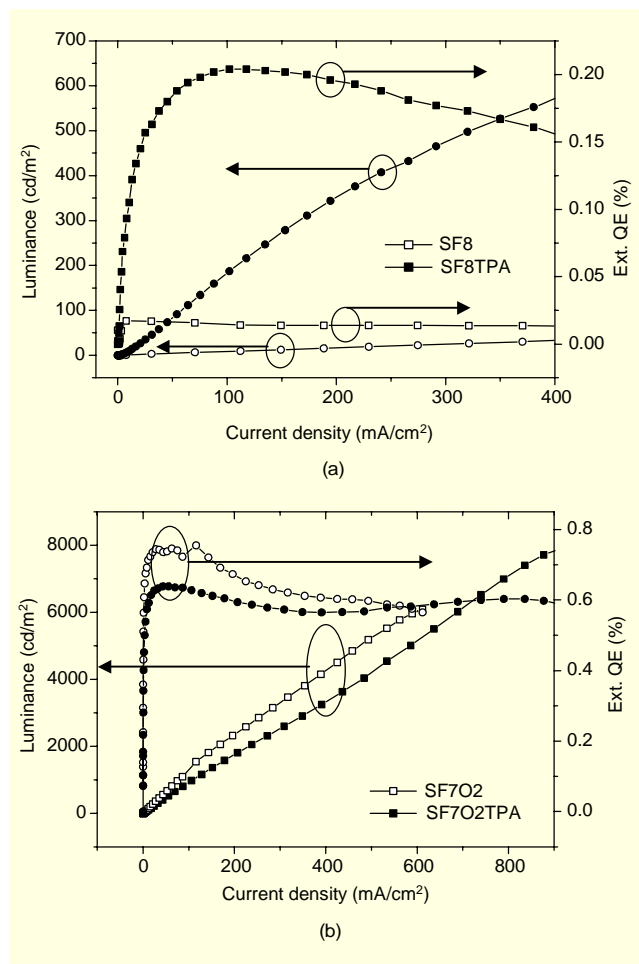


Fig. 3. Current density-luminance (J-L) and current density-external quantum efficiency characteristics of (a) SF8 and SF8TPA devices and (b) SF7O2 and SF7O2TPA devices. Squares indicate luminance and circles indicate external quantum efficiency. Open state is for the homopolymer devices and filled state for the copolymer devices.

Table 2. Device characteristics.

	@ 10 cd/m <sup>2</sup>				@ 100 cd/m <sup>2</sup>				@ 1000 cd/m <sup>2</sup>			
	Operation voltage (V)	Current density (mA/cm <sup>2</sup> )	Ext. QE (%)	Luminous efficiency (lm/W)	Operation voltage (V)	Current density (mA/cm <sup>2</sup> )	Ext. QE (%)	Luminous efficiency (lm/W)	Operation voltage (V)	Current density (mA/cm <sup>2</sup> )	Ext. QE (%)	Luminous efficiency (lm/W)
SF8	6.2	110	0.01	0.005	-	-	-	-	-	-	-	-
SF8TPA	6.4	10	0.1	0.05	8.0	50	0.2	0.07	-	-	-	-
SF7O2	4.8	1	0.6	0.7	5.6	8	0.7	0.8	7.4	75	0.7	0.6
SF7O2TPA	4.2	2	0.5	0.5	5.0	12	0.6	0.6	6.6	110	0.6	0.5



## IV. Conclusion

We have synthesized the polymer based on alkoxy-substituted spirobifluorene to suppress the long wavelength emission of polyfluorene with good solution processibility. A pure blue emission was obtained with the color coordinates of (0.16, 0.07) and (0.16, 0.08) for SF8 and SF8TPA, which did not show any spectral change upon EL device operation. The somewhat poor LED properties of SF8 were improved by the copolymerization with hole transporting comonomer, 4-*n*-butylphenyl-bis(4'-bromophenyl)amine. As the light emission was kept in a blue region, we have succeeded in improving external quantum efficiency by about 10 times compared with the homopolymer. Moreover, further efficiency improvements have been achieved by introducing the polar side chains of the 3,5-dioxaheptyl group into a spirobifluorene monomer, although some spectral broadenings have been observed. We believe that interface adhesion improvements between light emitting polymer and electrodes play an important role in device efficiency.

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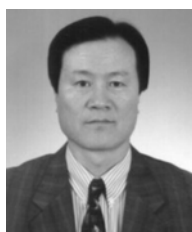
**Lee-Mi Do** received the BS, MS and PhD degrees in chemistry from Hannam University in 1982, 1984, and 1990. She also earned the PhD degree in surface chemistry from Tokyo Institute of Technology (TIT) in 1995. After graduation, she joined ETRI in 1996 and has worked on organic semiconductor devices, particularly OLED and OTFTs.



**Taehyoung Zyung** graduated from Seoul National University in 1977, and worked at KIST as a researcher for three and a half years from 1978. He received the PhD in physical chemistry at Texas Tech University in 1986. He performed a post-doctoral study as a research associate at the University of Illinois at Urbana-Champaign during 1986 to 1989. He joined ETRI in 1989 and has worked on organic semiconductor devices. He has been published in more than 100 SCI journals and has filed about 30 patents. He is now an Executive Director of Future Technology Research Division.



**Jaemin Lee** received the BS, MS, and PhD degrees in chemistry from Korea Advanced Institute of Science and Technology (KAIST) in 1999, 2001 and 2005. During his graduate period, he conducted research on conjugated polymers and related technologies. He now works at Corporate R&D Center in LG Chem/Research Park, Daejeon, Korea.



**Hong-Ku Shim** received the BS and MS degree in chemistry from Hannam University in 1969 and 1981, respectively. He earned the PhD degree in chemistry from Korea University in 1984. He has been a Professor of the Department of Chemistry at KAIST since 1984. His main research interest is in conjugated polymers and polymer light-emitting diodes (PLEDs). Lately, he has also been performing research in other organic electronics fields, such as in organic thin-film transistors and organic solar cells.