Synthesis and Characterization of Fluorene-Based Polymers Having Azine Unit for Blue Light Emission
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\textbf{Abstract.} Two types of donor-acceptor copolymers were designed and synthesized by combination of an electron donor unit of fluorene sequences and an electron acceptor azine unit such as 1,2,4,5-tetrazine and 1,3,5-triazine. They were well soluble in common organic solvents with the number average molecular weight ($M_n$) of 7.0 and 14.5 kg mol\textsuperscript{-1}, respectively, and have good thermal stability showing about at 360 °C with 5 wt\% loss in TGA. Two copolymers exhibited intense blue photoluminescence with emission peak maxima at 437 and 421 nm in CHCl\textsubscript{3}, and 451 and 422 nm in the film state, respectively. These polymers exhibited good fluorescence quantum efficiencies in CHCl\textsubscript{3} ($\phi_l = 0.63, 0.97$). Energy levels of the highest occupied molecular orbital and lowest unoccupied molecular orbital energy levels estimated by cyclic voltammetry were to be $-5.83, -6.0$ eV and $-2.85, -2.88$ eV, respectively.

1. Introduction

Conjugated polymers have gained large interest through various organic electronic applications such as organic light emitting diodes (OLEDs) \cite{1-3}, light emitting electrochemical cells (LECs) \cite{4-6}, organic solar cells \cite{7,8} and organic thin-film transistors \cite{9}. Blue light-emitting materials are of great significance for their unique applications in flat-panel displays and high-density information storage. Especially in full-color displays, the short-wavelength emission can serve as an excitation source for emission over the whole visible range \cite{10}. Therefore, development of high-efficiency blue emitting materials is important. However, to develop stable pure blue polymeric emitters with their color coordinates in the Commission Internationale de L’Eclairage (CIE) chromaticity diagram within the standard blue (CIE: $x < 0.15; y < 0.1$) are still rare since the large energy bandgap ($E_g$) of these materials makes the achievement of high efficiency and a good charge balance difficult \cite{11}. In this framework, the rational design of p-type conjugated polymers by incorporating electron-withdrawing groups, such as azine unit, seems to be a straightforward strategy to tune carrier injection and transporting properties of known materials \cite{12}. A new donor-acceptor (D-A) system has been designed, which is expected to lower both the energy level of the highest occupied molecular orbital ($E_{HOMO}$) and energy level of the lowest unoccupied molecular orbital ($E_{LUMO}$), giving rise to a series of intriguing properties such as improved resistance to oxidation, facilitated electron injection, and ambipolar characteristics, which could simplify device fabrication and operation.

In this paper, the synthesis, characterization and photoluminescence properties of fluorene-based polymers, PF-tetrazine or PF-triazine, shown in Fig.1 were investigated. Herein, the
fluorene sequences were selected as D for the reason of good thermal and electrochemical stability, high fluorescence yield and facile chemical functionalization [13,14], while 1,2,4,5-tetrazine (tetraZ) and 1,3,5-triazine (triaZ) were chosen as A for their electron deficiency [12].

![Chemical structures of PF-tetraZ and PF-triaZ](image)

**Fig. 1.** Chemical structures of PF-tetraZ and PF-triaZ

![Synthetic routes of PF-tetraZ and PF-triaZ](image)

**Scheme 1.** Synthetic routes of PF-tetraZ and PF-triaZ.

2. Experimental

2.1 Materials

Two polymers (PF-tetraZ and PF-triaZ) were synthesized according to the Scheme 1. 7-Bromo-9,9-bis(2-ethylhexyl)-9H-fluorene-2-carbonitrile 1, 2,4-dichloro-6-(octyloxy)-1,3,5-triazine 4 and 2,2'-((9,9-bis(2-ethylhexyl)-9H-fluorene-2,7-diyl)bis-1,3,2-dioxaborolane were synthesized according to the procedures reported previously [15-17]. Reagents and solvents were purchased from Kanto Chemical, Tokyo Chemical Industry, Aldrich and Nacalai Tesque Inc. Dimethylformamide (DMF) distilled after drying with CaH₂ was stored under an argon atmosphere. Tetrahydrofuran (THF) distilled after drying with sodium was stored under an argon atmosphere. The other solvents and all commercially available reagents were used without further purification.

3,6-Bis(7-bromo-9,9-bis(2-ethylhexyl)-9H-fluorene-2-yl)-1,2-dihydro[1,2,4,5] tetrazine (2). To suspension of 1 (0.2 g, 0.41 mmol) in 1 mL ethanol was quickly added hydrazine (0.019 mL, 0.615 mmol) and sulfur (0.008 g, 0.25 mmol). The mixture was refluxed for 3h with stirring and then
placed in an ice bath for further solidification. The orange precipitate was collected by filtration and washed with cold ethanol (2 x 10 mL) to give a crude dihydrotetrazine, which was used in the next step without further purification.

3,6-Bis(7-bromo-9,9-bis(2-ethylhexyl)-9H-fluorene-2-yl)-[1,2,4,5]tetrazine (3). The orange solid of 2 was dissolved in acetic acid (1 mL) at room temperature with stirring. NaNO₂ (0.028 g, 0.41 mmol) was added to the solution at 0 °C. The purple precipitate signified the completion of the reaction was collected by filtration and washed with methanol (2 x 5 mL) to give a dark purple solid (0.176 g, 84.6%). ¹H NMR (400 MHz, CDCl₃) δ [ppm]: 8.08 (s, 2H), 8.01 (d, J= 8.25 Hz 2H), 7.78 (d, J= 8.25 Hz 2H), 7.62 (d, J= 8.25 Hz 2H), 7.56 (s, 2H), 7.50 (d, J= 8.25 Hz 2H), 2.12-2.03 (m, 8H), 1.28-1.25 (m, 4H), 0.91-0.77 (m, 32H), 0.56-0.50 (m, 24H). ¹³C NMR (100 MHz, CDCl₃) δ [ppm]: 177.3, 157.7, 153.3, 144.7, 138.2, 130.5, 130.4, 128.4, 127.6, 127.5, 122.0, 120.3, 55.5, 44.2, 34.7, 33.6, 28.0, 27.1, 22.6, 14.1, 10.3.

2-Octyloxy-4,6-bis(7-bromo-9,9-bis(2-ethylhexyl)-9H-fluorene-2-yl)-1,3,5-triazine(5).

9,9-Bis(2-ethylhexyl)-2,7-dibromofluorene (0.44 g, 0.80 mmol) was dissolved in dry THF (4 mL) and cooled to −78 °C under nitrogen atmosphere, in which 1.6 M n-BuLi/hexane solution (0.56 mL, 0.9 mmol) was added dropwise with stirring. The mixture was kept at −78 °C for 1 h and 4 (0.11 g, 0.4 mmol) was slowly added. The reaction mixture was stirred for another 15 min at −78 °C. Afterward, the solution was allowed to warm up to room temperature and stirred for 24 h, and quenched with addition of water. After extraction with CH₂Cl₂, drying over MgSO₄ and the solvent evaporation, the crude product was purified by column chromatography (silica gel, CH₂Cl₂/hexane, 1:2, as eluent) to afford a yellow oil (0.088 g, 51%). ¹H NMR (400 MHz, CDCl₃) δ [ppm]: 8.66 (s, 4H), 7.82 (d, J= 7.78 Hz 2H), 7.65 (d, J= 7.78 Hz 2H), 7.58 (d, J= 7.78 Hz 2H), 7.51 (d, J= 7.79 Hz 2H), 4.66 (m, 2H), 2.13-1.90 (m, 10H), 1.32-0.82 (m, 42H), 0.58-0.52 (m, 27H). ¹³C NMR (100 MHz, CDCl₃) δ [ppm]: 173.5, 170.3, 153.9, 150.4, 144..5, 139.4, 134.5, 130.1, 128.4, 127.5, 124.6, 121.7, 121.6, 119.6, 68.1, 55.3, 44.3, 34.7, 33.5, 31.8, 29.4, 29.1, 28.8, 28.1, 27.1, 26.0, 22.7, 22.6, 14.1, 14.0, 10.4.

Poly[9,9,9',9'',9''-hexaxis(2-ethylhexyl)][2,2':7',2''-ter-9H-fluorene]-7,7''-diyl]-(1,2,4,5)tetrazine-3,6-diyl]/ (PF-tetrAz). A mixture of 2,2'-((9,9-bis(2-ethylhexyl)-9H-fluorene-2,7-diyl)bis-1,3,2-dioxaborolane (0.037 g, 0.07 mmol), tetrakis(triphenylphosphine) palladium (Pd(PPh₃)₄, 4.1 mg), aqueous K₂CO₃ (2M, 0.7 mL) and 3 (0.071 g, 0.07 mmol) in toluene (1.0 mL) was refluxed with vigorous stirring for three days under argon atmosphere. After the reaction solution was cooled to room temperature, the resultant polymer was precipitated from methanol/HCl aq and reprecipitated from methanol/NH₃ aq and from methanol, respectively. The precipitate was successively extracted with acetone, hexane and CHCl₃ by Soxhlet extraction. The CHCl₃ extract was again precipitated from methanol. PF-tetrAz was obtained as an orange solid (0.077 g, 80%). ¹H NMR (400 MHz, CDCl₃) δ [ppm]: 8.12 (br, 4H), 7.82 (br, 4H), 7.70-7.59 (m, 8H), 7.49 (br, 1H), 7.36 (br, 1H), 2.17-2.0 (m, 12H), 1.11-0.80 (m, 54H), 0.63-0.57 (m, 36H). C₉₈H₁₄₂N₄ (1246.89): Calcd. C 85.66, H 9.85, N 4.49; Found. C 83.48, H 9.33, N 4.38.

Poly[9,9,9',9''-tetrakis(2-ethylhexyl)-9H-fluorene-7,7''-diyl]-92-octyloxy-1,3,5-triazine-4,6-diyl] (PF-triAz). Under an argon atmosphere, a solution of bis(1,5-cycloocta -diene)nickel(0) (Ni(cod)₂) (0.110 g, 0.40 mmol), 2,2'-bipyridine (bpy) (0.070 g, 0.448 mmol) and 1,5-cyclooctadiene (cod) (0.10 g, 0.92 mmol) in DMF (1 mL) was heated for 30 min at 80 °C. To the DMF solution was
added 5 (0.083 g, 0.184 mmol) dissolved in THF (1 mL) under argon. The reaction solution was heated for 3 days at 80 °C. After the reaction solution was cooled to room temperature, the resultant polymer was precipitated from methanol/HCl aq, and recrystallized from methanol/NH3 aq and from methanol, respectively. The precipitate was successively extracted with acetone, hexane and CHCl3 by Soxhlet extraction. The CHCl3 extract was again precipitated from methanol. PF-triAz was obtained as a white solid (0.060g, 83%). 1H NMR (400 MHz, CDCl3) δ [ppm]: 8.73 (s, 4H), 7.90 (s, 4H), 7.70 (s, 4H), 4.69 (s, 2H), 2.17 (s, 8H), 1.97 (m, 2H), 0.93-0.87 (m, 42H), 0.68-0.60 (m, 27H). C71H100N3O (1013.70): Calcd. C 84.05, H 10.23, N 4.14; Found. C 82.23, H 9.66, N 3.84.

2.2 General Method and Instrumentation

All synthetic manipulations were performed by a standard technique using a Schlenk tube under an argon atmosphere. Column chromatography was performed using a silica gel (Kanto Chem., 60 N, 63–120 mm). Nuclear magnetic resonance (NMR) spectra were recorded on a JEOL JNM-ECS 400 spectrometer. 1H and 13C chemical shifts are given in units of δ (ppm) relative to δ (TMS) = 0.00 and δ(CDCl3) = 77.0 ppm, respectively. Photoabsorption in the range of ultraviolet-visible (UV-vis) and photoluminescence (PL) measurements of the polymer samples in CHCl3 and in a form of a thin film coating on a quartz glass were performed using a Shimadzu UV-1800 spectrophotometer and an F-4500 fluorescence spectrophotometer (Hitachi). The fluorescence quantum yield in CHCl3 were relative to 9,10-diphenylanthracene in cyclohexane (φ0 = 0.90) as a standard. The number-average molecular weight (Mn) and the weight-average molecular weight (Mw) of the polymers were estimated by gel permeation chromatography (GPC) system (Shimadzu, LCsolution) using polystyrene standards with CHCl3 as an eluent. Cyclic voltammetry (CV) of polymers in thin film on a Pt disk was performed at a scan rate of 50 mV/s in acetonitrile containing 0.1 M Et4NBF4 at room temperature under Ar using a saturated calomel electrode (SCE) as the reference and a platinum wire as the counter electrode. The electrochemical data (vs SCE) obtained by cyclic voltammetry were made a correction with the redox potential (4.8 eV) of ferrocene/ferricinium [18, 19]. Elemental analyses were carried out with a Perkin-Elmer type 2400 apparatus. Thermal gravimetric analysis (TGA) and differential thermal analysis (DTA) were carried out by an Extra 6000 TG/DTA (Seiko) analyzer at a heating rate of 10 °C min⁻¹ in an argon atmosphere.

3. Results and Discussion

3.1. Synthesis.

The general synthetic routes toward the monomer and polymer PF-tetrAz and PF-triAz are outlined in Scheme 1. Dihydrotetrazine intermediate 2, cyclized of 1 with anhydrous hydrazine by heating, was oxidized instantly into the fully aromatic tetrazine 3 for its instability. Tetrazine 3 was polymerized by Suzuki coupling reaction with 2,2',9,9'-bis(2-ethylhexyl)-9H-fluorene-2,7-diylbis-1,3,2-dioxaborolane to give PF-tetrAz. 2,7-Dibromo-9,9-bis(2-ethylhexyl)fluorene reacted with 4 to give 5. Homopolymerization of 5 by the Yamamoto reaction [20] afforded PF-triAz as a white solid. All the copolymers had good solubility in common organic solvents such as CHCl3, chlorobenzene, and o-dichlorobenzene. The polymers were identified by NMR and elemental analyses. The GPC results were summarized in Table 1. The Mn of PF-tetrAz and PF-triAz were 7.0 and 14.5 kg/mol, respectively. TGA results are shown in Fig. 2. Observed temperatures for 5wt % loss were 363 and 387 °C, respectively, which suggest that both have good thermal stability comparable to polyfluorenes [21].
Table 1. GPC and TGA results of the polymers.

| Polymer     | $M_n$ (kg·mol$^{-1}$) | $M_w$ (kg·mol$^{-1}$) | $M_w/M_n$ | $T_d$ (°C)$^a$ |
|-------------|------------------------|------------------------|-----------|----------------|
| PF-tetrAz   | 7.0                    | 7.6                    | 1.08      | 363            |
| PF-triAz    | 14.5                   | 21.9                   | 1.52      | 387            |

$^a$Temperature of 5% weight loss determined by TGA under an argon atmosphere.

Fig. 2. Thermal gravimetric analysis (TGA) curves of the polymers.

3.2. Optical properties.

The photophysical properties of dilute solution and thin films of all the polymers were investigated with UV-vis and PL, and the results are shown in Fig. 3. The UV-vis absorption and the emission spectra data for polymer were summarized in Table 2.

![Absorption and PL spectra](image-url)

Fig. 3. UV-vis absorption spectra and photoluminescence (PL) of the polymers in CHCl$_3$ (a) and film state (b).
Table 2. Optical properties, energy gaps and levels of the polymers.

| Polymer   | Abs. $\lambda_{\text{max}}$ (nm) | Em. $\lambda_{\text{max}}$ (nm) | $\phi_h$ | $E_g^{\text{opt}}$ (eV) | $E_{\text{HOMO}}$ (eV) | $E_{\text{LUMO}}$ (eV) | $E_g^{\text{ec}}$ (eV) |
|----------|-----------------|-----------------|---------|-----------------|-----------------|-----------------|-----------------|
| PF-tetraz | 390             | 393             | 437     | 451             | 0.63            | 2.80            | −5.83           | −2.85           | 2.96            |
| PF-triaz  | 394             | 395             | 421     | 422             | 0.97            | 2.98            | −6.0            | −2.88           | 3.12            |

Absorption and PL spectra of PF-tetraz and PF-triaz in CHCl$_3$ are shown in Fig. 3a. The absorption maxima in wavelength ($\lambda_{\text{max}}$) at 390 nm for PF-tetraz and 394 nm for PF-triaz are due to $\pi-\pi^*$ transition of the conjugated main chains. The absorption $\lambda_{\text{max}}$ of PF-triaz is in long wavelengths compared with that of PF-tetraz, which indicates that $\pi$-conjugation of PF-triaz is longer than that of PF-tetraz. The emission peaks in solution were observed at 437 nm for PF-tetraz and 421 nm for PF-triaz. Stokes shifts of PF-triaz in the solution was 27 nm, which was much smaller than that of PF-tetraz (47 nm). These small Stokes shifts also support PF-triaz having rigid and regular conformation, which enabled utilization of absorbed energy to PL efficiently.

The absorption and PL spectra of PF-tetraz and PF-triaz in thin solid film state are shown in Fig. 3b. Compared with absorption spectra in solution, the absorption of PF-tetraz and PF-triaz is slightly broaden and red shift compared with their absorption in film. The broadening and red shift of the film absorption spectra indicated that there were some aggregations or interactions of the polymer chains in the solid state. The optical band gap ($E_g^{\text{opt}}$) estimated from the onset of the absorption spectrum in the film state was 2.8 eV for PF-tetraz, which was smaller than that of PF-triaz (2.98 eV). Compared with PL spectra of PF-tetraz and PF-triaz in solution, a red shift was observed in their film. The PL maximum of PF-triaz in the film state was shorter in wavelength than that of PF-tetraz. This can be attributed to the higher polarizability of the tetraz group and restrained intermolecular aggregation [22].

PF-tetraz and PF-triaz showed intense blue PL emission with CIE coordinates ($x, y = 0.16, 0.12; 0.16, 0.07$, respectively) in the film state and good relative $\phi_h$ of 0.63, 0.97 in CHCl$_3$, which suggest that PF-tetraz and PF-triaz are considered to be good polymeric blue emitters superior to a series of polyfluorene derivatives [23].

3.3. Electrochemical Properties.

Electrochemical analysis of the thin-film sample of the polymers was employed to estimate $E_{\text{HOMO}}$ and $E_{\text{LUMO}}$. The CV results of PF-tetraz and PF-triaz are shown in Fig. 4 and the estimated values are summarized in Table 2. During the anodic scan, both of the compounds showed irreversible oxidation peaks, which are assigned to the oxidation of the fluorene units. PF-tetraz had $E_{\text{HOMO}}$ of −5.83 eV, which was higher about 0.17 eV than that of PF-triaz (−6.0 eV). This could be caused by the delocalization of the HOMO because of good planarity between the fluorene and tetraz segments. Upon the cathodic sweep, PF-triaz exhibited quasireversible reduction waves, whereas PF-tetraz showed weak irreversible reduction waves. The $E_{\text{LUMO}}$ of PF-tetraz (−2.85 eV) is slightly lower than that of PF-triaz (−2.74 eV), on account of higher electron-deficiency of tetraz compared to triaz. The above results suggest that electronegativity of the azine unit is responsible for lowering of both $E_{\text{HOMO}}$ and $E_{\text{LUMO}}$ of the polymers.
Fig. 4. Cyclic voltammograms of PF-tetrAz and PF-triAz.

Estimated from the $E_{\text{HOMO}}$ and $E_{\text{LUMO}}$, the $E_g^{\text{ec}}$ values of 2.96 and 3.12 eV are obtained for PF-tetrAz and PF-triAz, respectively, which are similar to $E_g^{\text{opt}}$. These energy gaps with suitable $E_{\text{HOMO}}$ and $E_{\text{LUMO}}$ are good for lessening driving voltage and augmenting efficiency of OLEDs [24].

4. Conclusion

In summary, two efficient blue luminescence polymers PF-tetrAz and PF-triAz, have been developed by combination of the fluorene sequences as a electron-donor and the tetrAz or triAz core as an electron-acceptor. These polymers were obtained in high yields, and they had a good solubility in common organic solvents and a good thermal stability. In the CHCl$_3$ solution, they showed similar electronic properties of absorption and emission, the values of $\lambda_{\text{max}}$(abs) and $\lambda_{\text{max}}$(em) of each polymer were about 390 and 430 nm, respectively. In the state of the thin films, PF-triAz showed deep blue fluorescence with the values of $\lambda_{\text{max}}$(em) at 422 nm and emission indicatable by CIE coordinates (0.16, 0.07) in the film state. PF-tetrAz and PF-triAz showed acceptable $E_{\text{HOMO}}$ (−5.83, −6.0 eV) for hole transport and proper $E_g$ (2.96, 3.12 eV) for blue emission. According to these results, these D-A copolymers have a great potential to be applied in OLEDs and LECs as the blue-light emitting materials.

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