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The influence of tetrakis-ethylhexyloxy groups substituted in PPV derivative for PLEDs

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Abstract

New electroluminescent polymer with tetrakis-alkoxy group, poly[2,3,5,6-tetrakis(2-ethylhexyloxy)-1,4-phenylenevinylene] (**TEH-PPV**), has been synthesized by the Gilch polymerization. In solid film state, the **TEH-PPV** exhibits absorption spectra with maximum peaks at 451 nm, and PL spectrum at 505–545 nm. As compared to MEH-PPV, **TEH-PPV** with tetrakis-ethylhexyloxy groups in phenylene unit can get shorter conjugation length, and had more blue shifted absorption and emission peaks due to steric hindrance, in spite of increasing the number of alkoxy substituents which may increase the effective conjugation length caused by the electron-donating effect. The polymer LEDs (ITO/PEDOT/polymer/Al) of **TEH-PPV** showed emission with maximum peaks at around 505–590 nm. Tetrakis-ethylhexyloxy groups induced very typical vibronically structured band in solid film state, since the conjugated backbone is twisted by steric hindrance. And they can enhance the internal efficiency of the conjugated polymer as emissive layer in PLED because of the restraint of inter-chain interaction by the avoidance of close packing to give decent device performance.

Keywords: PPV; Tetrakis-alkoxy group; Polymer light-emitting diodes

1. Introduction

Conjugated polymers have been incorporated as active materials into several kinds of electronic devices such as transistors, photovoltaic cells (PVCs) and organic light-emitting diodes (OLEDs) including flexible displays [1–4]. Many investigations have been focused on poly(p-phenylenevinylene)s (PPVs) since the discovery of electroluminescence (EL) in 1990 by the Cambridge group [5], and abundant derivatives of PPV have been reported with various substituents such as alkoxyl [6,7], silyl [8], phenyl [9], fluorenyl [10], halide [11–14], or cyano groups [15,16]. By the introduction of flexible side chains on the polymer backbone, one could achieve solubility and processability of aromatic conjugated polymers [17–19]. Flexible side chains also give rise to steric hindrance along the polymer backbone for the control of effective conjugation length and emission color of the polymers [20,21]. A decrease in the efficiencies of photoluminescence (PL) and electroluminescence (EL) is often caused by the intermolecular interactions of such linear polymers [22,23]. Conjugated polymers with sterically hindered side chains appended to the polymer backbone have exhibited excellent PL efficiencies in the solid state, and a series of polymers combining PPV segments and hyperbranched structures can lower the intermolecular interactions [24–26]. However, only few sterically hindered polymers have been reported due to the difficulty of their synthesis.

In this communication, in order to apply sterically hindered side chain with easy synthetic route, tetrakis-ethylhexyloxy groups were utilized. The present investigation deals with

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the synthesis, characterization, photophysics, and EL properties of new conducting polymer, poly[2,3,5,6-tetrakis(2-ethylhexyloxy)-1,4-phenylenevinylene] (**TEH-PPV**), which was synthesized by Gilch reaction [27,28]. As compared to MEH-PPV [29–31], **TEH-PPV** with tetrakis-ethylhexyloxy groups in phenylene unit can get shorter conjugation length, and had more blue shifted absorption and emission peaks due to steric hindrance, in spite of increasing a number of alkoxy substituents which may increase the effective conjugation length caused by the electron-donating effect. The new conjugated polymer shows highly promising features for the development of stable polymeric green light emitting diodes.

2. Experimental section

2.1. Materials and instruments

All reagents were purchased from Aldrich or TCI, and used without further purification. Solvents were purified by normal procedure and handled under moisture-free atmosphere. ^{1}H and ^{13}C NMR spectra were recorded with a Varian Gemini-200 (200 MHz), Unityplus-300 (300 MHz) and Inova-500 (500 MHz) spectrometers and chemical shifts were recorded in ppm units with TMS as the internal standard. Flash column chromatography was performed with Merck silica gel 60 (particle size 230–400 mesh ASTM) with ethyl acetate/hexane or methanol/methylene chloride gradients unless otherwise indicated. Analytical thin layer chromatography (TLC) was conducted using Merck 0.25 mm silica gel 60F pre-coated aluminum plates with fluorescent indicator UV254. UV spectra were recorded with a Varian 5E UV/vis spectrophotometer. The PL and EL spectra of the device were measured using an Oriel InstaSpec IV CCD detection system in combination with a monochromatic light source. For PL spectrum measurements, xenon lamp was used as the excitation source, and incident beam took the maximum absorption peak of the polymers. Molecular weights and polydispersities of the polymers were determined by gel permeation chromatography (GPC) analysis with a polystyrene standard calibration. To examine electrochemical properties of the resulting polymer, the polymer film was cast from THF solution onto a platinum plate as a working electrode. Cyclic voltammetric waves were produced by using a EG&G Parc model 273 potentiostat/galvanostat at a constant scan rate of 100 mV/s. Elemental analysis (Korea Basic Science Institute, Daegu) was observed toward identification of the structure of polymers. FAB mass spectra were taken at Korea Basic Science Institute Seoul Branch and Korea Basic Science Institute Daegu Branch.

2.2. EL device fabrication and measurements

For the EL experiment, poly(3,4-ethylenedioxythiophene) (PEDOT) doped with poly(styrenesulfonate) (PSS), as the hole-injection-transport layer, was introduced between emissive layer and ITO glass substrate cleaned by successive ultrasonic treatments. A solution of PEDOT/PSS in isopropanol was spin-coated on the surface-treated ITO substrate. On top of the PEDOT/PSS layer, the emissive polymer film was obtained by spin casting ODCB (*ortho*-dichlorobenzene) solution of the polymer. The emissive polymer thin film prepared had a uniform surface with a thickness of around 110 nm. The emissive film was dried in vacuum, and aluminum electrodes were deposited on the top of the polymer films through a mask by vacuum evaporation at pressures below 10^{-7} Torr, yielding active areas of 4 mm^2. For the determination of device characteristics, current density–voltage (J–V) characteristics were measured using a Keithley 236 source measure unit. All processing steps and measurements mentioned above were carried out under air and at room temperature.

2.3. Synthesis of monomer and polymer

2.3.1. 2,5-Bis[(2-ethylhexyl)oxy]benzo-1,4-quinone (3)

A two-necked flask equipped with a reflux condenser was charged with 2,5-dihydroxy-1,4-benzoquinone (10 g, 71.4 mmol), the appropriate 2-ethylhexyl alcohol (111 ml, 714 mmol) and boron trifluoride etherate (19.9 ml, 157 mmol). The distilled THF (100 ml) had to be added to dissolve the 2-ethylhexyl alcohol completely. The homogeneous reaction mixture was heated at 80 °C for 2 h. Crude compound was obtained from the reaction mixture by crystallization at −20 °C and purified by recrystallization (twice) from THF at −20 °C. Then, it was dried under reduced pressure for 12 h to give coupling compound (20.6 g, 79.0%): R_f 0.30 (SiO_2, 5% EtOAc in hexane). ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 0.84–0.93 (m, 12H), 1.25–1.50 (m, 16H), 1.75–1.84 (m, 2H), 3.78 (d, 4H, J = 5.50 Hz), 5.81 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 11.10, 14.29, 23.18, 23.82, 29.08, 30.40, 38.72, 72.55, 106.01, 159.28, 182.19. ($C_{22}H_{36}O_4$) (364.52): Calcd. C 72.49, H 9.95; Found C 72.31, H 10.11.

2.3.2. 2,5-Bis[(2-ethylhexyl)oxy]benzen-1,4-diol (4)

To a solution of compound **3** (6 g, 16.46 mmol) in ethyl acetate (50 ml) or ethanol was added 10% Pd–carbon (600 mg). The atmosphere of the reaction was exchanged for hydrogen gas (1 atm) and the reaction mixture stirred at room temperature for 24 h. The dispersion was filtered over Celite and washed with ethyl acetate several times. The resulting solution was concentrated *in vacuo* and purified by silica gel chromatography to provide the desired product (5.2 g, 86.2%) as a white solid: R_f 0.40 (SiO_2, 5% EtOAc in hexane). ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 0.86–0.95 (m, 12H), 1.21–1.52 (m, 16H), 1.66–1.74 (m, 2H), 3.78 (d, 4H, $J = 6.23$ Hz), 6.56 (s, 2H). ($C_{22}H_{38}O_4$) (366.53): Calcd. C 72.09, H 10.45; Found C 72.16, H 10.68.

2.3.3. 1,2,4,5-Tetrakis[(2-ethylhexyl)oxy]benzene (5)

A mixture of hydroquinone compound **4** (5 g, 13.64 mmol), potassium hydroxide (1.08 g, 16.37 mmol), sodium iodide (0.15 g, 1.02 mmol) and 2-ethylhexyl bromide (5.58 ml, 30.11 mmol) in ethyl alcohol (50 ml) was refluxed for 3 days. Even though compound **3** was synthesized by coupling reaction with Lewis acid, compound **5** was synthesized by substitution reaction with base. The reaction mixture was cooled

Table 1
The results of polymerization

Polymer	M_n[a] ($\times 10^3$)	M_w[a] ($\times 10^3$)	PDI[a]	T_g[b] (°C)	T_d[c] (°C)
TEH-PPV	16	65	3.9	157	397

[a] Molecular weight (M_w) and polydispersity (PDI) of the polymers were determined by gel permeation chromatography (GPC) in THF using polystyrene standards.

[b] Glass transition temperature measured by DSC under N_2.

[c] Onset decomposition temperature (5% weight loss) measured by TGA under N_2.

and filtered. The mixture was concentrated *in vacuo*, diluted with ethyl acetate, and washed twice with saturated sodium chloride–water solution. The obtained crude product was recrystallized with MeOH to provide the desired product (7.37 g, 91.4%) as colorless oil: R_f 0.1 (SiO_2, hexane 100%). ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) 0.86–0.98 (m, 24H), 1.26–1.60 (m, 32H), 1.64–1.73 (m, 4H), 3.81 (d, 8H, $J = 6.23$ Hz), 6.55 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 11.58, 14.11, 23.09, 23.82, 29.15, 30.52, 39.76, 73.00, 105.23, 143.77. ($C_{38}H_{70}O_4$) (590.96): Calcd. C 77.23, H 11.94; Found C 77.42, H 12.24.

2.3.4. 1,4-Bis(bromomethyl)-2,3,5,6-tetrakis-[(2-ethylhexyl)oxy]benzene (6)

To a stirred solution of compound **5** (5 g, 8.46 mmol) in trifluoroacetic acid (15 ml) was added bromomethyl methyl ether (1.69 ml, 18.6 mmol). After stirring for 24 h at room temperature, to the reaction mixture was added NaOH to neutralize, and extracted with EtOAc/$NaHCO_3$. The organic layer was dried over $MgSO_4$, filtered and concentrated *in vacuo*. The oily layer was purified by flash column chromatography to give monomer (6.07 g, 92.3%): R_f 0.35 (SiO_2, hexane 100%). ^{1}H NMR (200 MHz, $CDCl_3$): δ (ppm) 0.89–1.00 (m, 24H), 1.26–1.70 (m, 32H), 1.78–1.87 (m, 4H), 3.94 (d, 8H, $J = 6.60$ Hz), 4.59 (s, 4H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 11.18, 11.20, 14.10, 22.86, 23.15, 23.60, 23.62, 29.11, 29.12, 30.26, 40.45, 76.55, 127.25, 147.21. ($C_{40}H_{72}Br_2O_4$) (776.81): Calcd. C 61.85, H 9.34; Found C 62.01, H 9.63.

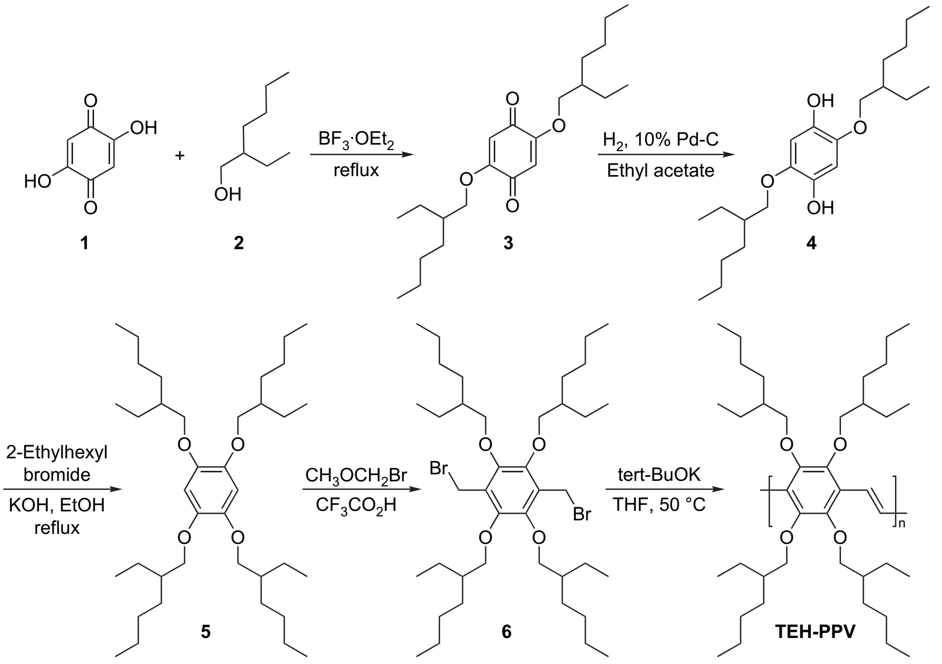
2.3.5. TEH-PPV (7)

To a stirred solution of monomer **6** (1 g, 1.29 mmol) in dry THF (50 ml), potassium *tert*-butoxide (7.72 ml, 1.0 M THF solution, 7.72 mmol) was added drop-wise for 2 h by a syringe pump at 50 °C. During this addition, the reaction mixture had color change from colorless to green, and the viscosity increased significantly. After the addition was complete, the reaction mixture was stirred additionally for 12 h at 50 °C. The polymerization solution was poured into 500 ml of methanol with stirring. The precipitated polymer was filtered off, and the resulting polymer was dissolved again in THF. The dissolving polymer was slowly poured into 500 ml of intensively stirred methanol. The precipitated polymer was filtered off, washed with water, and dried under reduced pressure at room temperature. A 350 mg sample of polymer was obtained as green fiber (Table 1).

3. Results and discussion

3.1. Synthesis and characterization

Scheme 1 displays the general synthetic routes toward monomer and poly(2,3,5,6-tetrakis(2-ethylhexyloxy)-1,4-phenylenevinylene) (**TEH-PPV**), which contains tetrakis-ethylhexyloxy-substituted phenylenevinylene unit. **TEH-PPV** can be incorporated with tetrakis-alkyl groups, which can control the polymer solubility and the inter-chain interactions in thin films. For the preparation of **TEH-PPV**, in the first step, the commercially available 2,5-dihydroxy-1,4-benzoquinone (**1**) was



Scheme 1. Synthetic route for the monomer and the polymer.

Table 2
Optical properties of **TEH-PPV**

Conditions	Abs λ_{max} (nm)	PL λ_{max} [a] (nm)	fwhm [b] (nm)
Solution	419	475 (505)	57
Film	451	505, 545 (589, 639)	63

[a] The data in the parentheses are the wavelengths of shoulders and subpeaks.

[b] Full width at half-maximum of the film PL spectra.

chosen as the starting material and coupled with 2-ethyl-hexyl alcohol (**2**) using boron trifluoride etherate in THF [32]. The resulting 2,5-bis[(2-ethylhexyl)oxy]benzo-1,4-quinone (**3**) was treated with palladium catalyst and hydrogen gas in ethyl acetate to generate 2,5-bis[(2-ethylhexyl)oxy]benzen-1,4-diol (**4**), which was coupled with ethylhexyl bromide using potassium hydroxide in ethyl alcohol. This coupled 1,2,4,5-tetrakis[(2-ethylhexyl)oxy]benzene (**5**) was bromomethylated with bromomethyl methyl ether in trifluoroacetic acid to generate monomer, 1,4-bis(bromomethyl)-2,3,5,6-tetrakis[(2-ethylhexyl)oxy]benzene (**6**). The polymer of this monomer, **TEH-PPV** (**7**), was prepared by Gilch polymerization. In order to reduce by-products, potassium *tert*-butoxide was added drop-wise for 2 h by a syringe pump at 50 °C to a stirred solution of monomer in dry THF. Generally, Gilch polymerization is carried out in 0 °C, however, this polymer needed high reaction temperature due to possession of tetrakis-ethylhexyloxy group, which induced steric hindrance in this polymerization condition.

The resulting **TEH-PPV**, green polymer, was soluble in organic solvents such as chloroform, tetrahydrofuran, dichloromethane, and chlorobenzene. The number-average molecular weight (M_n) and the weight-average molecular weight (M_w) of the **TEH-PPV** were 16,000 and 65,000 with polydispersity of 3.9 as determined by GPC using THF as the eluant and polystyrene as the standard. The thermal properties of the polymer were determined by DSC and thermal gravimetric analysis under a nitrogen atmosphere at a heating rate of 10 °C/min. **TEH-PPV** shows glass transition at 157 °C and loses less than 5% of their weights on heating to 397 °C. These T_g and T_d are higher than those of other conjugated polymers. The high thermal stability of the resulting polymer prevents the deformation of the polymer morphology and degradation of the polymer light-emitting device by applied electric field of the LED (Table 2).

3.2. Absorption and photoluminescence properties of polymer

The linear UV/vis absorption and photoluminescence (PL) emission spectra of **TEH-PPV** as solution and thin film are shown in Fig. 1. The solution was prepared using THF as solvent and the thin film was prepared by spin-coating on quartz plates from the polymer solutions in *ortho*-dichlorobenzene (ODCB). The maximum absorption peaks appeared at round 419 nm in the solution of THF. The maximum absorption peak of **TEH-PPV** in solid thin film is red-shifted to around 451 nm. The large red-shifted absorption peak of **TEH-PPV** in solid film indicates that the inter-chain interaction, which can be attributed to the π–π* transitions of conjugated polymer backbone, assisted the planarity of the polymer chain and increased the conjugation length in the solid state.

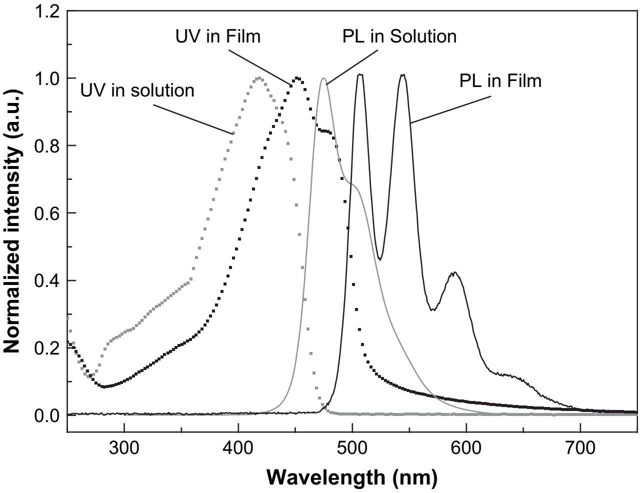


Fig. 1. UV-vis absorption and PL emission spectra of **TEH-PPV** in solution and solid thin film.

The PL emission spectra of **TEH-PPV** in THF solution show a maximum peak at 475 nm and a shoulder peak at 506 nm, and in case of solid thin film, they are also more red-shifted over 30 nm than solution conditions. The PL spectra of the solid film consist of a typical vibronically structured band comprising two maxima at 507 and 543 nm, a shoulder at 590 nm, and a tail. Since substituted alkoxy group in PPV can increase conjugation length, MEH-PPV was red-shifted [29–31]. By the way, **TEH-PPV** had more blue shifted absorption and emission peaks than MEH-PPV with di-alkoxy groups, which can be attributed to the steric effect of tetrakis-ethylhexyloxy groups. This steric bulkiness induces typical vibronically structured band caused by the reduction of the rigid property of the polymer in solid film state.

Fig. 2 shows the changing PL spectra of **TEH-PPV** in the mixture with MeOH and THF from 0:10 to 9:1 ratio. In case of usual PPV derivatives with alkoxy groups for substituents, the solubility in MeOH is not good. By the way, **TEH-PPV** can be dissolved in the mixture with MeOH and THF of 9:1 ratio if it dissolves in THF in the first time. As increasing the hydrophilic solvent (MeOH) content ratio, the change of spectra is not much, since all condition are solutions. However, the maximum peaks were slightly red-shifted from 475 to 477 nm, and the vibronically structured band of shoulder at around 506 nm became very clear by decreasing maximum peak and increasing shoulder peak. We can guess that this phenomenon can explain of maximum peaks of 505 and 545 nm in solid film state (Figs. 3 and 4).

3.3. Electrochemical properties of the polymers

The electrochemical properties of the polymer were determined from the band gaps which were estimated from the absorption onset wavelength, and the HOMO energy levels which were estimated from the cyclic voltammetry (CV). The absorption onset wavelength of **TEH-PPV** was observed

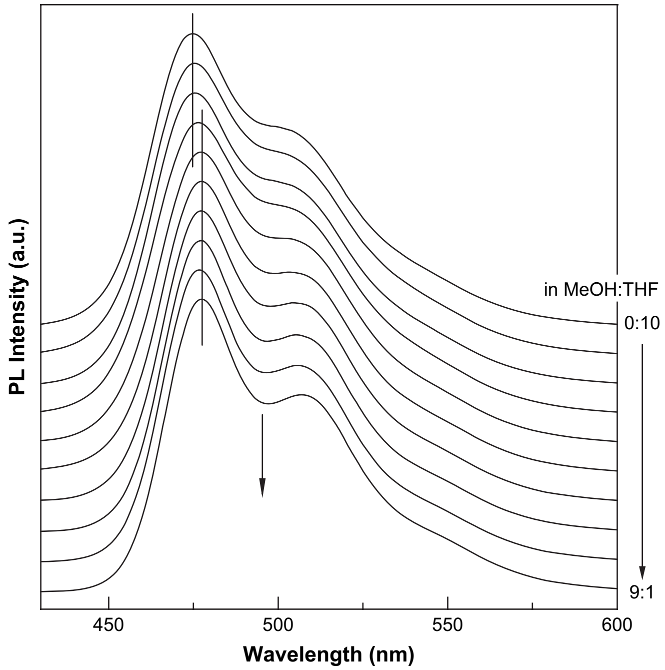


Fig. 2. (A) Fluorescence spectra of **TEH-PPV** ([**TEH-PPV**] = 1 × 10^{-6} M) in mixture with changing ratio of MeOH and THF (MeOH:THF = 0:10 to 9:1). All emission scans were collected by exciting at 420 nm.

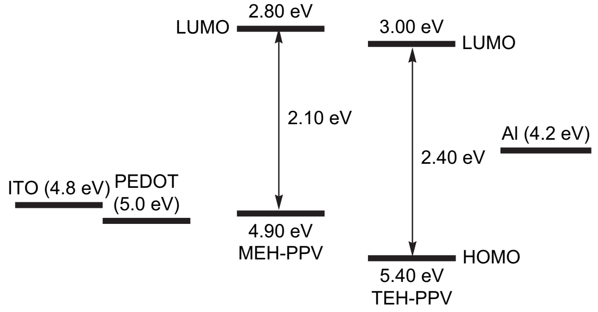


Fig. 4. Comparison of the energy levels of MEH-PPV and **TEH-PPV** with the work functions of the electrodes.

to be 516 nm in solid thin film, which corresponds to band gap of 2.40 eV. The CV was performed with a solution of tetrabutylammonium tetrafluoroborate (Bu_4NBF_4) (0.10 M) in acetonitrile at a scan rate of 100 mV/s at room temperature under the protection of argon. A platinum electrode (~0.05 cm^2) coated with a thin polymer film was used as the working electrode. Pt wire and $Ag/AgNO_3$ electrode were used as the counter electrode and reference electrode, respectively. The energy level of the $Ag/AgNO_3$ reference electrode was determined to be 4.65 eV, which was calibrated by the ferrocene/ferrocenium (FC/FC^+) redox system with 4.8 eV. The oxidation potentials is derived from the onset of electrochemical p-doping, and HOMO levels were calculated according to the empirical formula: ($E_{HOMO} = -([E_{onset}]^{ox} + 4.6)$ (eV). The polymer exhibits irreversible processes in an oxidation scan. The oxidation onset of **TEH-PPV** was estimated to be 0.8 V, which correspond to HOMO energy level of 5.40 eV. The LUMO energy levels of the polymer can be calculated with the HOMO and optical band gap. The LUMO energy level of **TEH-PPV** was thus determined to be 3.00 eV (Table 3).

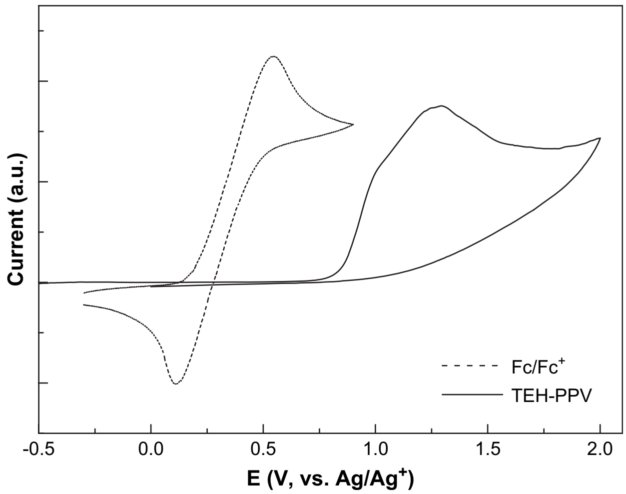


Fig. 3. Cyclic voltammograms of polymer and ferrocene used as a reference. Measurements were carried out in 0.1 M Bu_4NBF_4 in acetonitrile solution with a scanning rate of 100 mV/s.

3.4. EL property and current density–voltage–luminescence (J–V–L) characteristics

The electroluminescence (EL) spectrum of ITO/PEDOT/polymer/Al device is shown in Fig. 5. The EL spectra of **TEH-PPV** show two maxima peaks at 504 and 543 nm, and a shoulder at 588 nm. These features are similar to those observed in the PL spectrum of the corresponding polymer film. The results indicate that the EL and PL phenomena originated from the same excited state. As compared to the PL spectrum of thin film, the EL spectrum of the polymer showed decreased sub-peak. The effective conjugation length and the color of the polymer can be controlled by utilizing several types of substituents. Although alkoxy groups in PPV derivatives can increase conjugation length caused by the electron-donating effect, tetrakis-ethylhexyloxy groups of the new **TEH-PPV** can generate steric effect to induce shorter conjugation length and blue shifted fluorescence spectra than MEH-PPV with di-alkoxy groups. The emission color of the device with the configuration of ITO/PEDOT/**TEH-PPV**/Al was green with the CIE coordinates of $x = 0.33$, $y = 0.57$.

The current density–voltage–luminescence (*J–V–L*) characteristics and electroluminescence efficiency of the devices fabricated from **TEH-PPV** are shown in Figs. 6 and 7.

Table 3
Device performance characteristics

Polymer	EL λ_{max}[a] (nm)	Turn-on voltage[b] (V)	Voltage[c] (V)	Current density[c] (mA/cm^2)	Luminance[d] (cd/m^2)	LE_{max}[e] (cd/A)
TEH-PPV	505, 542 (590)	6	14	231	764	0.18

[a] The data in the parentheses are the wavelengths of shoulders and sub-peaks.

[b] Voltages required to achieve a brightness of 1 cd/m^2.

[c] Measured under the condition of maximum luminescence efficiency.

[d] Maximum brightness.

[e] Maximum luminescence efficiency.

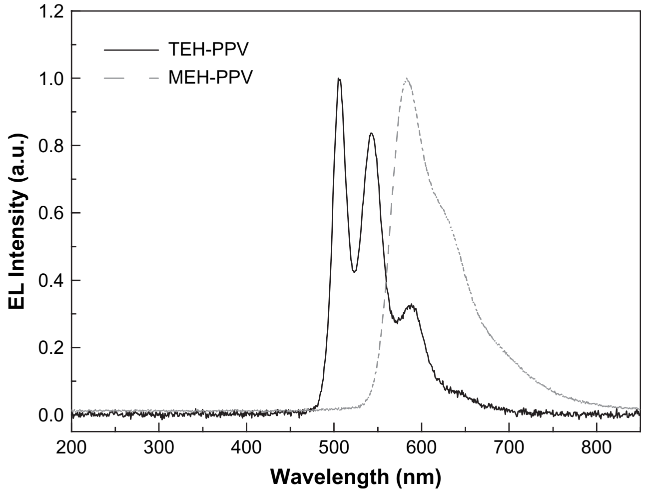


Fig. 5. Electroluminescence spectra of devices with the configuration of ITO/PEDOT/polymers/Al.

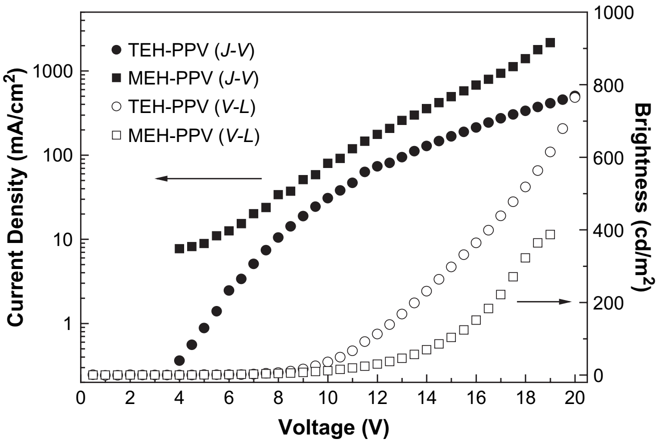


Fig. 6. Current density–voltage–luminescence (*J*–*V*–*L*) characteristics of OLEDs of **TEH-PPV** and MEH-PPV with the configuration of ITO/PEDOT/polymers/Al.

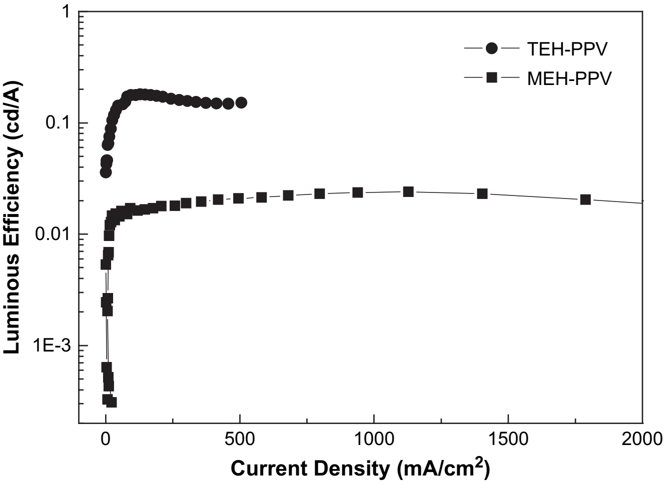


Fig. 7. Efficiencies of OLEDs of **TEH-PPV** and MEH-PPV with the configuration of ITO/PEDOT/polymers/Al.

In the forward bias, the turn-on voltage of **TEH-PPV** is 6.0 V, and the maximum brightness is 765 cd/m^2 at 20 V, which is better than 387 cd/m^2 of MEH-PPV. The maximum luminescence efficiency of **TEH-PPV** is 0.18 cd/A at 14 V, which is also higher than 0.024 cd/A of MEH-PPV. Therefore, we conclude that tetrakis-ethylhexyloxy groups, which prevent close packing in order to give fine device performance in solid state, can enhance the internal efficiency of the conjugated polymer as emissive layer in PLED due to restraint of inter-chain interaction.

4. Conclusion

In conclusion, we present here the synthesis of a novel conjugated polymer, **TEH-PPV**, which exhibits interesting properties for the development of PLED. **TEH-PPV** had more blue shifted absorption and emission peaks due to steric hindrance. Tetrakis-ethylhexyloxy groups introduced in the phenylene unit induced typical vibronically structured band in solid film state, since the conjugated backbone is twisted by steric hindrance. The sterically hindered tetrakis-ethylhexyloxy units may result in an amorphous solid state, which give decent device performance. This also suppresses ordered regions within the emissive layer of PLED and thus enhances the internal efficiency of the device.

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