Macromolecules **2002,** *35,* 2876−2881

Articles

Synthesis and Luminescence Properties of Poly[2-(9,9-dihexylfluorene-2-yl)-1,4-phenylenevinylene] and Its Copolymers Containing 2-(2-Ethylhexyloxy)-5-methoxy-1,4-phenylenevinylene Units

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Received September 17, 2001; Revised Manuscript Received January 9, 2002

ABSTRACT: A PPV derivative bearing the dihexylfluorenyl pendants, poly[2-(9,9-dihexylfluorene-2-yl)-1,4-phenylenevinylene] (DHF−PPV) and its copolymers containing the dialkoxy-substituted phenylenevinylene units, i.e., poly[(2-(9,9-dihexylfluorene-2-yl)-1,4-phenylenevinylene)-*co*-(2-(2-ethylhexyloxy)-5-methoxy-1,4-phenylenevinylene)s], were prepared, and luminescence properties of the light-emitting diodes (LEDs) fabricated with them were studied. The copolymers 92.5 DHF/7.5 MEH−PPV and 50.5 DHF/49.5 MEH−PPV contained 7.5 and 49.5 mol % of the dialkoxy comonomer units, respectively. The structure of LED devices was ITO/PEDOT (25 nm)/polymer (80 nm)/Ca (50 nm)/Al (50 nm). The wavelengths of maximum emitted light of the devices were 519 nm (green), 560 nm (yellow), and 585 nm (orange-red) for DHF−PPV and the two copolymers. Turn-on electric fields decreased in the order of DHF−PPV (0.54 MV/cm) > 92.5 DHF/7.5 MEH−PPV (0.43 MV/cm) > 50.5 DHF/49.5 MEH−PPV (0.29 MV/cm). Luminance efficiencies of the three devices were 2.3, 1.0, and 0.9 cd/A for 92.5 DHF/7.5 MEH−PPV, 50.5 DHF/49.5 MEH−PPV, and DHF−PPV, respectively. The maximum luminance for the device of DHF−PPV was 1.6×10^4 cd/m^2, and the value increased to 2.7×10^4 cd/m^2 for 97.5 DHF/7.5 MEH−PPV. The maximum luminance of the other copolymer was about 1.9×10^4 cd/m^2. The study of time-resolved PL of the present polymers strongly suggests that a correlation exists between their PL decay behavior and EL efficiency. The mobilities of the charge carriers, i.e., hole and electron, are better balanced ($\upsilon_h/\upsilon_e \sim 10$) in DHF−PPV than in MEH−PPV ($\upsilon_h/\upsilon_e \sim 10^2$).

Introduction

Since the first report by the Cambridge group[1] on the electroluminescence (EL) of poly(*p*-phenylenevinylene)-(PPV), EL properties of a number of PPV derivatives have been studied by us[2] and others.[3] In our recent papers, we discussed the EL properties of PPV derivatives bearing either the carbazole[2c] or phenyloxadiazole[2d] pendants. Dissymmetrical attachment of pendant substituents onto the PPV backbone alters not only the wavelength of emitted light but also the external efficiency of the light-emitting diodes (LEDs) based on the polymers.

As a part of our continuing effort to establish the structure−EL properties relationship of PPV derivatives, we have synthesized polymers bearing dihexylfluorenyl pendants (see Chart 1). The first (DHF−PPV) and the last (MEH−PPV) polymers are homopolymers, and the other two are random copolymers containing the 2-ethylhexyloxy-5-methoxy-1,4-phenylenevinylene (MEH−PV) comonomer units. It is well-known that homo- and copolymers composed of only dialkyl-substituted fluorene repeating units without the vinylene units in the backbone are capable of emitting blue light in LED applications.[4,5] And the comonomer unit of the above copolymers is the building block of MEH−PPV which has been thoroughly studied for EL properties by Heeger's group[6] and others.[7]

This article describes the synthesis of the new polymers and EL properties of LEDs fabricated from them.

Experimental Section

The synthetic routes to the monomer and polymers are shown in Schemes 1 and 2. The synthetic details of monomer **7** can be found in the Supporting Information.

Homopolymerization of Monomer 7. Monomer **7** (0.50 g, 0.84 mmol) was dissolved in 50 mL of THF containing 2.0 mg of *tert*-butylbenzyl bromide.[8] The solution was cooled to 0 °C, to which potassium *tert*-butoxide (2.5 mL of 1 M THF solution) was added. The solution was stirred for 3 h at 0 °C. The polymerization mixture was poured into 200 mL of

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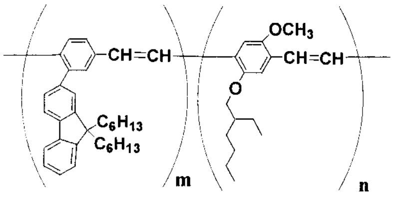
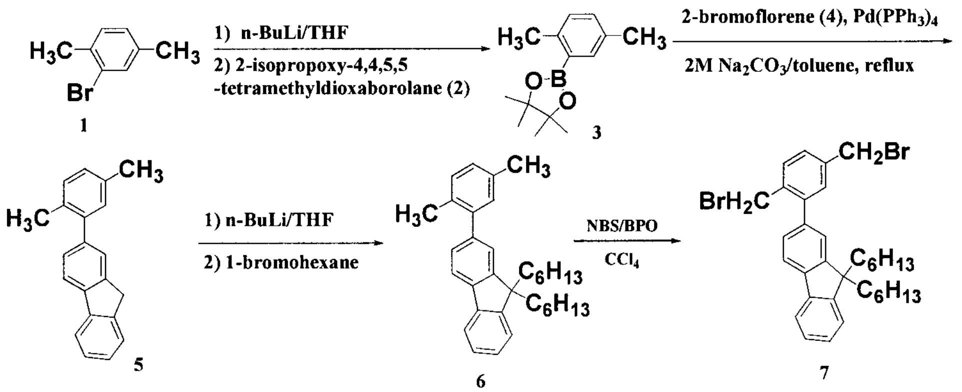
10.1021/ma011636t CCC: $22.00

Chart 1

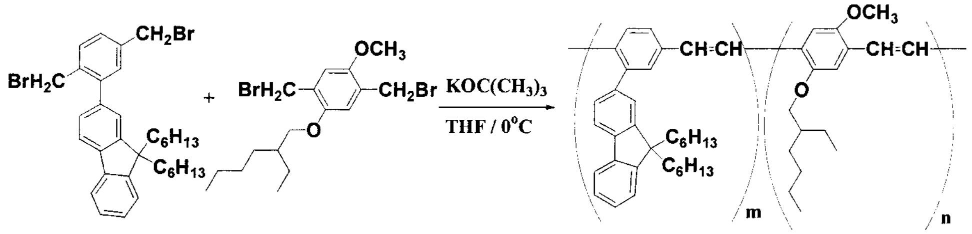


m = 1.0 ; n = 0 : DHF-PPV
(Poly[2-(9,9-dihexyl (fluorene-2-yl)-1,4-phenylenevinylene))
m = 0.925 ; n = 0.075 : 92.5 DHF / 7.5 MEH-PPV
m = 0.505 ; n = 0.495 : 50.5 DHF / 49.5 MEH-PPV
m = 0 ; n = 1 : MEH-PPV

Scheme 1. Synthetic Route to the 2-[1,4-Bis(bisbromomethyl)]phenyl-9,9-dihexylfluorene Monomer



Scheme 2. Synthesis of DHF−PPV, MEH−PPV, and Copolymers



m : n = 1 : 0 ; DHF-PPV
m : n = 0.925 : 0.075 ; 92.5 DHF / 7.5 MEH-PPV
m : n = 0.505 : 0.495 ; 50.5 DHF / 49.5 MEH-PPV
m : n = 0 : 1 ; MEH-PPV

methanol. The yellow precipitate was subjected to Soxhlet extraction for 24 h using methanol. The final polymer was dried at room temperature in a vacuum oven. The recovered polymer yield was 0.28 g (78%).

Copolymerization. A mixture of compound **7** and 1,4-bis(bromomethyl)-2-(2-ethylhexyloxy)-5-methoxybenzene was polymerized in THF using potassium *tert*-butoxide initiator under the same conditions as described in homopolymerization. The two copolymers prepared were purified by Soxhlet extraction with methanol. The two copolymers (refer to Scheme 2) were prepared using the monomer mixture of $m{:}n = 91{:}9.0$ and 50:50 in mole ratio. The actual compositions of the copolymers estimated from their H NMR are $m{:}n = 92.5{:}7.5$ and 49.5:50.5, respectively. The recovered polymer yields were 73 and 68%, respectively.

Characterization of Polymers. ^1H NMR spectra were collected on a Bruker Advance 300 spectrometer. UV−vis absorption spectra were obtained on a JASCO V-560 spectrophotometer. Electroluminescence (EL) and photoluminescence (PL) spectra were recorded with IS PC photon counting spectrofluorometer. Molecular weights of polymers were determined by gel permeation chromatography (GPC; Waters 410) using THF as eluent and polystyrene as standard. The thermal stability of polymers was studied by thermogravimetry, which was performed in air on an IBM 2050 instrument at a heating rate of 20 °C/min. The redox properties of polymers were examined by cyclic voltametry.[9] The polymer thin films were spin-coated on the ITO-coated glasses. Chlorobenzene was used as the solvent. The electrolyte solution employed was 0.1 M tetrabutylammonium perchlorate, Bu_4NClO_4, in *N*,*N*-dimethylformamide. The Ag/AgCl and Pt wire (600 μm in diameter) electrodes were utilized as reference and counter electrodes, respectively. Time-resolved PL spectra of the polymer films were obtained as described in one of our earlier papers.[10]

Fabrication of LED Devices. The devices have a structure of ITO/PEDOT (25 nm)/polymer (80 nm)/Ca (50 nm)/Al (50 nm). The ITO-coated glasses were cleaned as described earlier.[2a] The conducting PEDOT (polyethylenedioxythiophene doped with polystyrenesulfonate from Baeyer Co. with conductivity of $10\ S\ cm^{-1}$) layer (25 nm thick) was spin-coated onto the ITO-coated glasses. The emitting polymer layer (80 nm) then was spin-coated onto the PEDOT layer using the polymer solution in chlorobenzene. Finally, calcium (50 nm) and aluminum (50 nm) electrodes were sequentially deposited under a reduced pressure of 10^{-7} Torr on the polymer. The device efficiency and electroluminescence spectra were obtained as same method as described earlier.[2a]

Results and Discussion

General Properties of Polymers. All the polymers, DHF−PPV and the two copolymers, were prepared by the Gilch polymerization[11,12] of monomer **7** or monomer mixtures (refer to Scheme 1). They are soluble in common organic solvents such as chloroform, THF, dichloromethane, toluene, xylene, and *N*,*N*-dimethylformamide. The number-average molar mass ($\bar{M}_n$) of the polymers determined by GPC analysis was 420×10^3 for DHF−PPV, 290×10^3 for 92.5 DHF/7.5 MEH−PPV, and 280×10^3 for 50.5 DHF/49.5 MEH−PPV. Sepa-

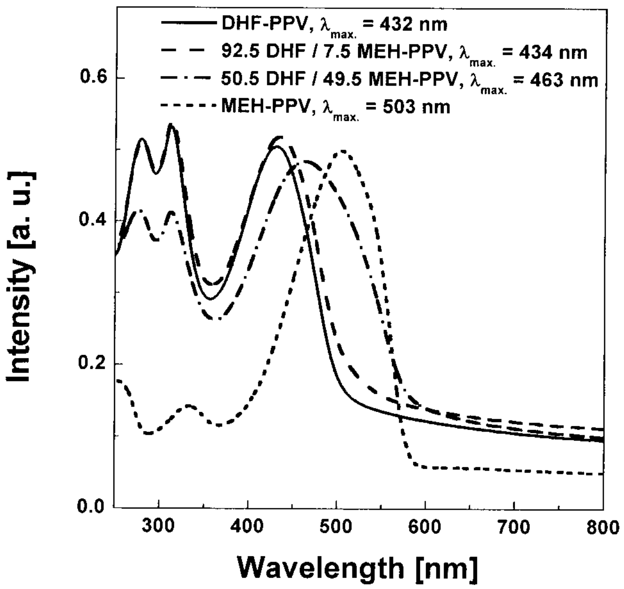


Figure 1. UV–vis absorption spectra of the polymers.

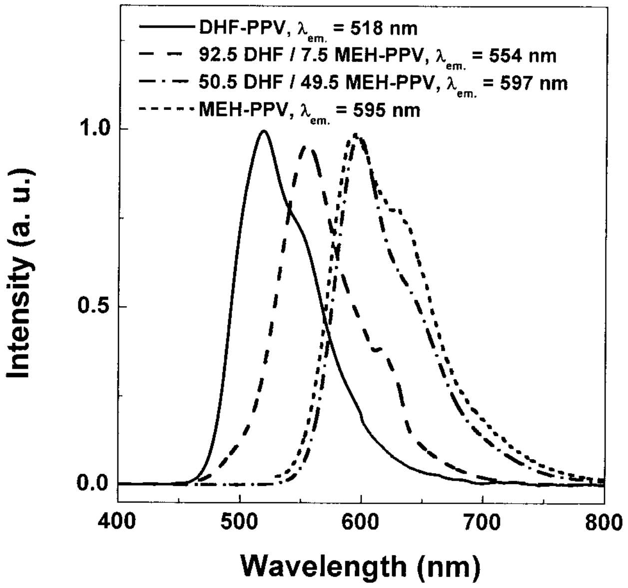


Figure 2. Photoluminescence spectra of the polymers obtained at the excitation wavelength of 370 nm.

rately, we prepared poly(2-(2-ethylhexyloxy)-5-methoxy-1,4-phenylenevinylene) (MEH–PPV), and its molar mass was found to be $M_n = 310 \times 10^3$. The polydispersity indices of the four polymers were 2.7, 2.8, 3.2, and 4.2. Thermogravimetric analysis performed in air show that thermal stability of the present polymers is fairly good. The temperature revealing 5 wt % loss decreased from 429 °C for DHF–PPV to 377 °C for MEH–PPV. The corresponding temperatures of the two copolymers lie in between. The presence of the alkoxy substituents in MEH–PPV and copolymers appears to lower the initial decomposition temperature. The glass transition temperature of the polymers could not be identified on their thermograms obtained by differential scanning calorimetry (DSC). This can be ascribed to the rigidity of their main chains.

UV–vis Absorption, Photoluminescence, and Electronic Structure. The UV–vis absorption and photoluminescence (PL) spectra of the four polymers are compared in Figures 1 and 2. According to the spectra shown in Figure 1, all the polymers exhibit two major absorptions: one below 350 nm and the other over a longer wavelength region. The first absorptions in the short wavelength region are originated from π electronic transitions of aromatic structures and the second from the π–π^* transitions of main-chain π-systems. The absorption maximum position (λ_{max}) of the second peak steadily increases from 432 nm for DHF–PPV to 435, 463, and 504 nm for the two copolymers and MEH–PPV, respectively. In other words, as the content of the MEH–PV comonomer unit increases, the λ_{max} value moves toward the longer wavelength. The bathochromic shift[13] by the alkoxy groups has been well established. The optical band gaps (E_g's) estimated from the UV–vis absorption edges (503 nm for DHF–PPV, 517 and 582 nm for two copolymers, and 582 nm for MEH–PPV) are 2.5 eV (DHF–PPV), 2.4 and 2.1 eV (two copolymers), and 2.1 eV (MEH–PPV). It is evident that inclusion of the MEH–PV unit lowers the band gap energy.

Figure 2 shows PL spectra of the polymers obtained at the excitation wavelength 370 nm. The PL spectrum of DHF–PPV reveals an emission peak whose maximum is located at 518 nm. The peak has one shoulder at 548 nm. The position of emission peaks moves toward longer wavelength as the content of the MEH–PV unit in the polymers is increased. A significant shift is observed even when the copolymer contains only 7.5 mol % of the MEH–PV unit; the maximum emission position (λ_{em}) of the emission peak of DHF–PPV is 517 nm, whereas that of 92.5 DHF/7.5 MEH–PPV is 554 nm. Such a notable red shift of the emission peak by including a very low level of the MEH–PV unit strongly implies that excitons formed in higher energy structures migrate to lower energy structures containing the MEH–PV unit. The λ_{em} values increases to 597 and 595 nm respectively for 50.5 DHF/49.5 MEH–PPV and MEH–PPV. The degree of red shift is greatly diminished, when the content of the MEH–PPV unit becomes higher. All the emission peaks reveal a common feature that a shoulder appears in the longer wavelength side.

Table 1. PL Rise and Decay Time at Room Temperature

	rise (ps)	decay 1 (ps)	decay 2 (ps)
DHF–PPV	28	350 (82.7%)	1080 (17.3%)
92.5 DHF/7.5 MEH–PPV	32	430 (76.7%)	1000 (23.3%)
50.5 DHF/49.5 MEH–PPV	26	230 (85.0%)	1190 (15.0%)
MEH–PPV		210 (84.6%)	870 (15.4%)

Table 2. PL Efficiencies of Polymers in Solution and Film

polymer	Φ(solution)[a]	relative efficiency[b]
DHF–PPV	0.87	5.3
92.5 DHF/7.5 MEH	0.94	6.8
50.5 DHF/49.5 MEH	0.47	2.1
MEH–PPV	0.22	1

[a] We used THF as a solvent and Coumarin 307 as a reference.[14] The concentration was 5×10^{-6} mol/L of the repeating unit. [b] Relative value to the integrated PL intensity of MEH–PPV film.

Table 2 compares the PL efficiencies of different polymers in tetrahydrofuran (THF) solution (5.0×10^{-6} mol/L of the repeating unit) and for thin (100 nm thick) films. According to the data given in the table, surprisingly enough, 92.5 DHF/7.5 MEH–PPV reveals the highest PL quantum efficiency in solution as well as in bulk film.

From the cyclovoltametry,[15] we could determine the ionization potentials (IP), i.e., the HOMO levels, of the polymers. The oxidative onset potentials were taken as IP values. The LUMO levels were estimated from the

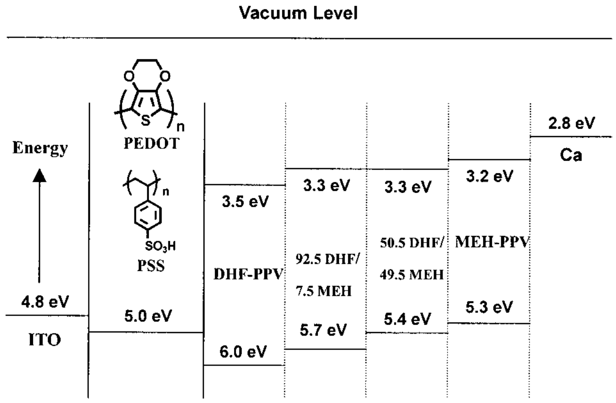


Figure 3. Electronic energy diagram of the polymers.

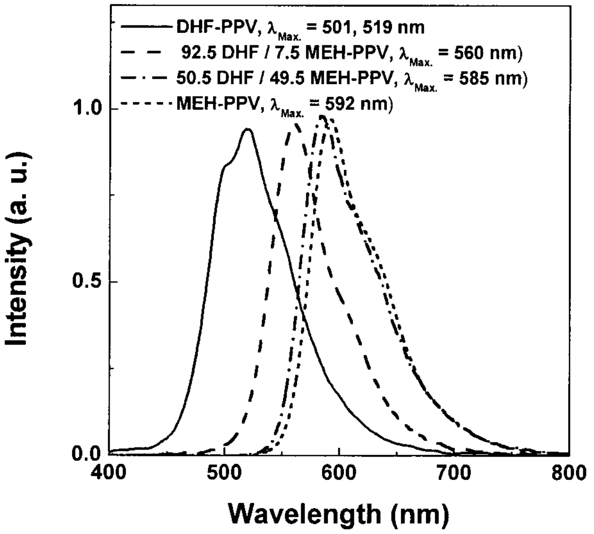


Figure 4. Electroluminescence spectra of LEDs.

HOMO levels and optical band gaps (E_g's) determined from the absorption edges of the UV−vis absorption spectra. The work function of ITO was taken as 4.8 eV.[16] The HOMO and LUMO energy diagram thus obtained of the present polymers is given in Figure 3. According to the diagram, attachment of dihexylfluorene pendant on the PPV backbone increases the HOMO level more than the LUMO level; the HOMO and LUMO levels of PPV[17] are 5.5 and 3.2 eV, whereas they are 6.0 and 3.5 for DHF−PPV, respectively. In contrast, the alkoxy pendant lowers the HOMO level while they affect relatively little the LUMO level.

Electrical and Electroluminescence Properties of LED Devices. Electroluminescence of the LED devices constructed with the four polymers was studied in detail. The device configuration was ITO/PEDOT (25 nm)/polymer (80 nm)/Ca (50 nm)/Al (50 nm). Aluminum was deposited onto the calcium electrode (cathode) in order to protect the latter. The electroluminescence (EL) spectra of the devices are presented in Figure 4. In general, the shapes and positions of EL spectra are very similar to those of the corresponding PL spectra. The EL spectrum of the DHF−PPV device, however, reveals a rather strong shoulder around 498 nm, in addition to the major peak at 520 nm (green) and another shoulder around 550 nm. A closer examination of the PL spectrum (Figure 3) of DHF−PPV indicates that a fairly strong shoulder has been buried in the left-hand side of the major emission peak at 517 nm. The other devices show peak maxima at 561 nm (yellow), 585 nm (orange), and 592 nm (orange) with their shoulders locating at 588, 620, and 627 nm.

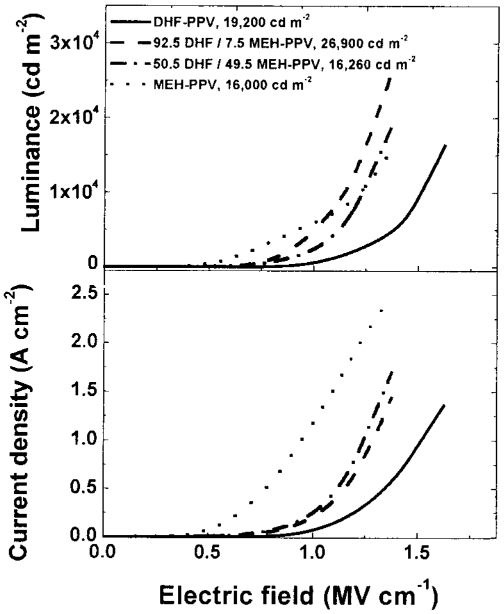


Figure 5. Dependence of current density and luminance on electrical field in the LED devices.

Figure 5 shows the $I-V$ characteristics of the four devices. The turn-on electric field increases from 0.28 MV/cm for the MEH−PPV device to 0.54 MV/cm for the DHF−PPV device. The other two copolymers exhibit the turn-on electric field of 0.43 and 0.29 MV/cm with the value being lower for the copolymer containing higher level of the MEH−PV unit. Comparison of the values of turn-on electric field of the devices with the HOMO levels (Figure 3) of the corresponding polymers leads to the conclusion that the smaller the difference between the work function of the ITO anode and the HOMO level of a polymer, the lower the turn-on electric field becomes. This, in turn, implies us that easier hole injection from the anode controls the turn-on electric field of the present devices, which should hold true only when the major carrier is hole. Moreover, it is reminded again that the LUMO levels (Figure 3) of the present polymers are not so much dependent on the compositions of the present polymers, and they all are lower than the work function of calcium metal (2.8 eV). Although the MEH−PPV device exhibits the lowest turn-on electric field, Figure 5 tells us that the devices based on DHF−PPV and copolymers are more efficient in emitting light per current density. The most surprising phenomenon is that the copolymer (92.5 DHF/7.5 MEH−PPV) containing the low level of the MEH−PV comonomer unit exhibits a remarkably higher light output per current density when compared with DHF−PPV, MEH−PPV, and another copolymer containing about 50 mol % of the MEH−PPV unit.

This is better compared in Figure 6, which shows the dependence of luminance efficiency on the current density for the four LED devices. One can see that the 92.5 DHF/7.5 MEH−PPV device's maximum luminance efficiency is about 2.4 cd/A, significantly higher than those (0.65−1.0 cd/A) of other devices. The efficiencies of the devices fabricated with DHF−PPV and 50.5 DHF/49.5 MEH−PPV are not so much different from each other, with the latter being only slightly better. One of the plausible conjectures to explain the enhancement of device efficiency by inclusion of a relatively low level

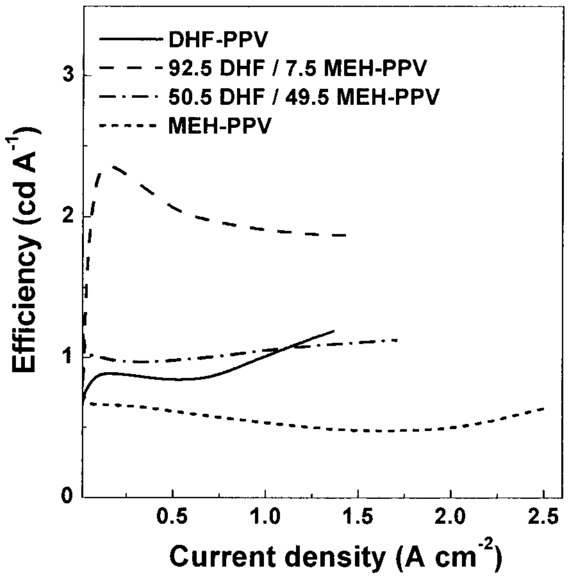


Figure 6. Dependence of luminance efficiency of LEDs on current density.

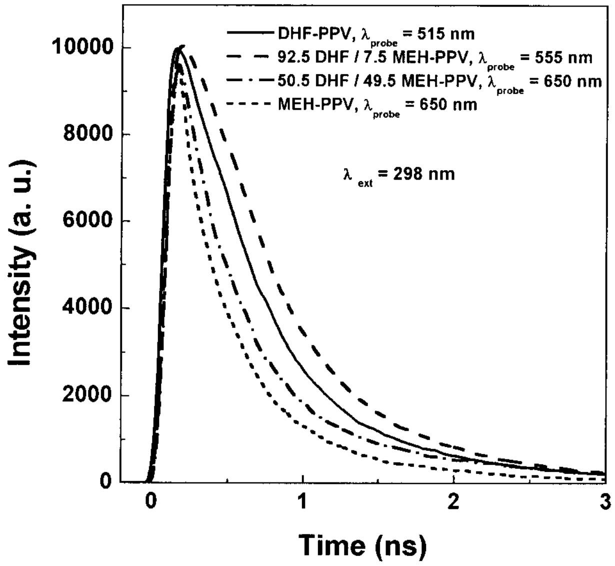


Figure 7. Time-resolved PL decay of the polymers.

of the MEH−PV unit is that inclusion of a small amount of the comonomer unit along the main chain of the polymers provides the sites where confinement of excitons are promoted. As the level of the MEH−PV unit in the backbone increases, confinement of excitons is expected to diminish.

This assumption is strongly supported by the fact that fluorescence lifetime is significantly lengthened when the MEH−PV comonomer unit is included in a low level into DHF−PPV as shown in the time-resolved photoluminescence spectra of Figure 7. Fluorescence decayed fastest for MEH−PPV followed by 50 DHF/50 MEH−PPV, DHF−PPV, and 92.5 DHF/7.5 MEH−PPV in that order. When the PL decay curves were fit to the biexponential function containing a slow and a fast decay time constants, we obtained the results as shown in Table 1. We clearly see that the fast decay time constant governs the majority part of the PL decay, which decreases in the order of 92.5 DHF/7.5 MEH−PPV (430 ps) > DHF−PPV (350 ps) > 50.5 DHF/49.5 MEH−PPV (230 ps) > MEH−PPV (210 ps). Moreover, we clearly observe the rise time for DHF−PPV and copolymers, indicating an efficient energy transfer from the fluorene pendants to the backbone.[10] In contrast, with the exception of MEH−PPV, the slow time constant did not vary as much with the content of the MEH−PV unit. Since it is generally accepted that the PL and EL mechanisms are basically the same, it is expected that the excitons confined more effectively are more likely to be involved in an emissive decay, resulting in a higher luminescence efficiency.[18]

In addition, balance in carriers mobilities is another important factor controlling the LED device efficiency. As is well-known, PPV transports the holes much faster than the electrons.[19,20] According to our own measurements,[18a] by the time-of-flight method[18b] PPV's hole mobility is about 200 times its electron mobility. Therefore, much of the holes injected from the anode into the emitting layer are wasted on the cathode. That is one of the reasons for the poor device performance based on PPV. Although MEH−PPV is known to perform better in LED,[3a] it is still far from satisfaction. The presence of the alkoxy substituents hinders the interchain contacts that is expected to reduce the chances for the formation of interchain exciton pairs. Formation of interchain exciton pairs was claimed to be another factor diminishing device efficiency.[7b] The balance in the mobility of the carriers, however, is not improved by the two alkoxy substituents. In MEH−PPV the mobility of holes is more than 100 times faster than the mobility of electrons.[21] In contrast, the imbalance in the mobility of the carriers is significantly reduced in DHF−PPV,[22] in which the hole mobility is only about 10 times faster than the electron mobility. We believe that the presence of 9,9-dihexylfluorenyl pendants stabilizes the positive hole, which results in the reduction in the mobility of the positive hole carrier. This, in turn, is expected to reduce the imbalance in the mobilities of the positive and negative carriers. As the content of the MEH−PV units in the copolymers is increased, the imbalance in the carrier mobility grows. In short, a combination of reduced turn-on electric field, augmented excitons confinement, and improved balance in the mobilites of carriers is judged to be the major reason for a higher device efficiency of 92.5 DHF/7.5 MEH−PPV among the polymers described in this report. It is added that one of the recent publications by Lee et al.[23] describes an interesting approach in achieving a balance in the mobilities of carriers. They attached the electron-transporting oxadiazole pendants to the PPV backbone through long dodecyloxy spacers so that the oxadiazole groups[2d] can be uniformly dispersed in the hole-transporting substituted PPV matrix. This is in contrast with our earlier work where the oxadiazole groups were attached directly to the PPV backbone through the *p*-phenylene bridge.

Conclusion

We have described the synthesis and luminescence properties of a series of new PPV derivatives bearing fluorenyl pendants. Attachment of the fluorenyl pendants onto the phenylene rings of PPV not only renders solubility but also improves the LED device efficiency. Because of the presence of the bulky pendant, interchain distance must have increased significantly as compared with unsubstituted PPV, which would reduce the possibility for the formation of interchain exciton pairs. This could be one of the major reasons for the improved

LED device performance. In addition, the presence of the pendants reduced the imbalance in the hole and electron mobilities most probably owing to the dissymmetric coulomb potentials along the main chain caused by dissymmetric substitution.[22]

Further improvement in the EL performance could be achieved by incorporating a relatively low level of the dialkoxyphenylenevinylene (MEH−PV) unit into the fluorenyl polymer. This comonomer unit appears to efficiently confine the excitons and also increases the lifetime of luminescence. This investigation clearly demonstrates that fine-tuning of chemical structure can lead to significant enhancement in LED performance of conjugated polymers.

Acknowledgment. This work was supported by the Korea Science and Engineering Foundation through CRM of Korea University. K. Kim is a recipient of the Brain Korea 21 fellowship. The support by the Samsung Institute of Advanced Science and Technology is acknowledged.

Supporting Information Available: The synthetic details of monomer **7**. This material is available free of charge via the Internet at http://pubs.acs.org.

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MA011636T