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Efficient and Bright Blue Electroluminescence of Poly[4,4′-biphenylene-α-(9″,9″-dihexyl-3-fluorenyl)vinylene]

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ABSTRACT: A blue electroluminescent polymer, poly(biphenylenevinylene) derivative containing a bulky fluorenyl group, was prepared by the nickel-catalyzed coupling reaction. The structure and properties of the polymer, PBPHFV, were analyzed by various spectroscopic methods. The obtained polymer had good solubility and thermal stability. The polymer film showed maximum absorption and emission peaks at 370 and 485 nm, respectively. A blue electroluminescence ($\lambda_{max} = 465$ nm) was obtained with intensities of 4116 cd/m^2 when the light-emitting diodes of ITO/PEDOT/PBPHFV/LiF/Al were fabricated. The maximum efficiency of the devices was 0.22 lm/W with a turn-on voltage of 4.3 V. With the optimum ratio of PBPHFV to PVK as 1:5, the luminance and efficiency of the devices reached up to 9342 cd/m^2 and 1.66 lm/W, respectively.

Introduction

Since light-emitting devices (LED) using conjugated polymers were first realized in the early 1990s,[1,2] a great number of different conjugate polymers suitable for LEDs have been investigated and developed. These conjugate polymers are especially attractive for use as the emitting layers in LEDs.

Poly(*p*-phenylenevinylene) (PPV),[1−6] which was the first reported electroluminescent polymer, and its derivatives have several advantages as an emitting material, including high thermal stability, good film quality, and suitable color tunability. However, these polymers also have some disadvantages, such as low oxidative stability and difficulties in processability and emitting blue color.

Poly(*p*-phenylene) (PPP) and its derivatives[7−12] have also been extensively investigated for use as light-emitting materials because they are thermally and oxidatively stable polymers. PPP, however, exhibits low solubility and high turn-on voltage. Furthermore, the low work function metals are required for efficient carrier injection in PPP derivatives. Because the ring torsion angles within the biphenyl segments of PPP are about 23° and those of PPP derivatives are above 23°, PPP and its derivatives are intrinsically violet-blue emitter to have some difficulties in color tunability.[13]

Solubility, oxidative stability, low turn-on voltage, and color tunability (especially blue emission) are desirable properties for conjugated polymers used in LEDs. Moreover, good thermal stability is needed because heat is generated when current passes through the device.

Poly(4,4′-diphenyldiphenylenevinylene) (PDPV) having the structural characteristics of PPP and PPV is an attractive polymer for electroluminescence because it has a very high photoluminescence efficiency in solid state along with good solubility in common organic solvents. However, PDPV having diphenyl substituents in the 1,2 position of C═C showed the green emission.[14,15]

Thus, we tried to optimize molecular structures for blue emission with high efficiency. On the basis of the biphenylenevinylene segments composed of alternating PPP and PPV units,[16] the large fluorene substituent was introduced into the vinyl bridge. Since poly(9,9-dihexylfluorene), the green-blue emitter,[17−20] has superior photo- and electroluminescent properties and good solubility, we introduced the 9,9-dihexylfluorene moiety as the pendant group at the vinyl bridge. The introduced bulky fluorene moieties will give steric interactions to the adjacent phenyl rings. These steric interactions may cause twisting of the polymer chains which reduce the effective conjugation length as well as the formation of excimers owing to interchain interactions and π-staking between the conjugated segments.

In this article, we describe the synthesis of a new soluble blue-light-emitting polymer, poly[4,4′-biphenylene-α-(9″,9″-dihexyl-3-fluorenyl)vinylene] (PBPHFV), and its application to LEDs. A large 9,9-dihexylfluorene pendant group in the vinyl bridge of poly(biphenylenevinylene) could lead to enhance solubility of the resulting polymer and shorten the effective conjugation length that results in bright blue EL emission.

Experimental Section

Materials. All reagent and solvents were purchased from Aldrich Chemical Co. and Fluka. Only analytical grade quality chemicals were used. PEDOT was purchased from Bayer. Spectroscopic grade $CHCl_3$ (Aldrich) was used for all absorption and emission experiments. All other compounds were used as received.

Preparation of 4-Bromophenyl-9″,9″-dihexyl-3-fluorenyl Ketone. In a mixture of dihexylfluorene (**1**) (5 g, 22.8 mmol), CS_2 (70 mL), and $AlCl_3$ (3.95 g, 29.64 mmol) equipped with an ice bath, the 4-bromobenzoyl chloride was added. After 2 h stirring, the mixture was worked up with 2 N HCl aqueous solution, and then the mixture was extracted with diethyl

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ether. The extract was washed with 2 N KOH aqueous solution and then was washed several times with water. The washed ether solution was dried over $MgSO_4$ and filtered. The solvent was evaporated, and the crude product was recrystallized in hexane. Yield: 8.1 g, 69%; mp: 84 °C.

^1H NMR (500 MHz, CD_2Cl_2) (ppm): aromatic (C−H), δ 7.85−7.83 (m, 4H), 7.74−7.69 (m, 4H), 7.45−7.42 (m, 3H), aliphatic (C−H) δ 2.07−2.04 (t, 4H), 1.17−1.14 (m, 4H), 1.11−1.07 (m, 8H), 0.82−0.81 (t, 6H), 0.68−0.65 (m, 4H). ^{13}C NMR (CD_2Cl_2) (ppm): 196 (C═O), aromatic (C) 152.9, 151.7, 146.7, 140.7, 138.1, 136.5, 132.3, 130.4, 129.3, 127.9, 127.7, 125.4, 124.1, 121.5, 120.2 aliphatic (C) 56.2, 40.9, 32.4, 30.5, 24.7, 23.4, 14.6. FT-IR (KBr) (cm^{-1}): 3047 (aromatic C−H), 2846 (aliphatic CH_2), 1643 (C═O), 1064 (aromatic C−Br).

Preparation of 1,2-Bis(4′-bromophenyl)-1-(9″,9″-dihexyl-3-fluorenyl)ethene (BPHFE). In a two-necked flask equipped with a reflux condenser, a mixture of (4-bromobenzyl)triphenylphosphonium bromide (BTPB) (4) (7.42 g, 14.5 mmol), sodium hydride (1.16 g, 48 mmol), and toluene (70 mL) was refluxed for 8 h. After cooling, **3** (5 g, 9.66 mmol) was added and refluxed for 48 h. The mixture was worked up with ice water and then was extracted with ether. The extract was dried over $MgSO_4$ and filtered. The solvent was removed by evaporation. The residue was dissolved in *n*-hexane and then purified by flash column chromatography (silica gel, 10% ethyl acetate in hexane) to remove byproducts. After removal of *n*-hexane, recrystallization from ethanol afforded pure BPHFE. The product was collected by filtration and dried under vacuum. Yield: 3.4 g, 51%; mp: 87 °C.

^1H NMR (500 MHz, CD_2Cl_2) (ppm): aromatic and vinyl (C−H) 7.69−7.27 (m, 2H), 7.5 (m, 2H), 7.4−7.3 (m, 7H), 7.15−7.14 (m, 2H), 7.03−6.99 (m, 3H), aliphatic (C−H) 2.0−1.9 (t, 4H), 1.17−1.14 (m, 4H), 1.09−1.06 (m, 8H), 0.84−0.81 (m, 6H), 0.64−0.63 (m, 4H). ^{13}C NMR (CD_2Cl_2) (ppm): aromatic (C) 149.9, 149.6, 141.6 aliphatic (C) 53.9, 38.9, 30.3, 28.3, 22.5, 21.2, 22.5, 21.2. FT-IR (KBr) (cm^{-1}): 3008.4 (aromatic C−H), 2917.0 (aliphatic CH_2 symmetric), 2846.4 (aliphatic CH_2 asymmetric), 1064.5 (aromatic C−Br), 817.6 (C−H bending). Anal. Calcd for $C_{39}H_{42}Br_2$: C, 69.85; H, 6.27; Br, 23.88. Found: C, 69.81; H, 6.24; Br, 23.85.

Preparation of Poly[4,4′-biphenylene-α-(9″,9″-dihexyl-3-fluorenyl)vinylene] (PBPHFV). Into a 10 mL three-necked flask equipped with a nitrogen inlet, BPHFE (0.5 g, 0.75 mmol), nickel chloride (9.5 mg, 0.75 mmol), zinc (0.2256 g, 3.45 mmol), triphenylphosphine (0.295 g, 1.125 mmol), bipyridine (11.7 mg, 0.075 mmol), and *N*,*N*-dimethylformamide (DMF) (2.0 mL) were added under nitrogen. The reaction mixture was stirred at 90 °C for 12 h. After stirring, excess bromobenzene was added in the reaction mixture for end-capping. After adding bromobenzene, the reaction mixture was stirred at 90 °C for 6 h. The products were precipitated into a large excess of 20% hydrochloric acid/methanol solution. The white-green solids that remained were washed with methanol, water, and methanol sequentially. The polymer was collected and dried under vacuum. The yield of the polymer after complete work-up was 0.29 g (76%). ^1H NMR (500 MHz, CD_2Cl_2) (ppm): aromatic and vinylic (C−H) 7.68−7.04 (br, 16H), aliphatic (C−H) 1.90 (br, 4H), 1.1−0.9 (br, 12H), 0.79−0.68 (br, 10H). ^{13}C NMR (CD_2Cl_2) (ppm): aromatic (C) 151.5, 141.1, 130.4, 128.3, 127.4, 126.8, 123.1, 120.1 aliphtic (C) 55.4, 40.6, 31.8, 30.0, 24.1, 22.9, 14.4. FT-IR (KBr) (cm^{-1}): 3015.7 (aromatic C−H), 2925.7(aliphatic CH_2 symmetric) Anal. Calcd for $C_{39}H_{42}$: C, 91.76; H, 8.24. Found: C, 91.68; H, 8.15.

Fabrication of the LED. Poly(styrenesulfonate)-doped poly(3,4-ethylene dioxythiophene) (PEDOT) for a conducting polymer hole-injection layer was coated on an indium tin oxide coated glass substrate which had been washed with water, acetone, and isopropyl alcohol sequentially. A thin polymer film (800−1100 Å) was spin-coated (2200 rpm, 50 s) from a filtered (0.2 μm filter) 2.0 wt % PBPHFV solution in chlorobenzene on a PEDOT layer. LiF was vacuum-deposited as an electron injection layer. An aluminum/lithium alloy (Al:Li = 99.95:0.05 wt %) electrode (1300 Å) was deposited on top of the device at a high vacuum (below 1×10^{-5} Torr). Wires were attached to the respective electrodes with a conductive epoxy adhesive. All fabrication steps were performed in clean room conditions. Measurements were done at room temperature in air.

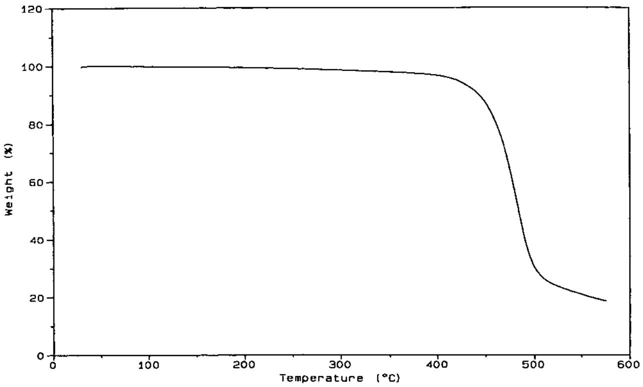


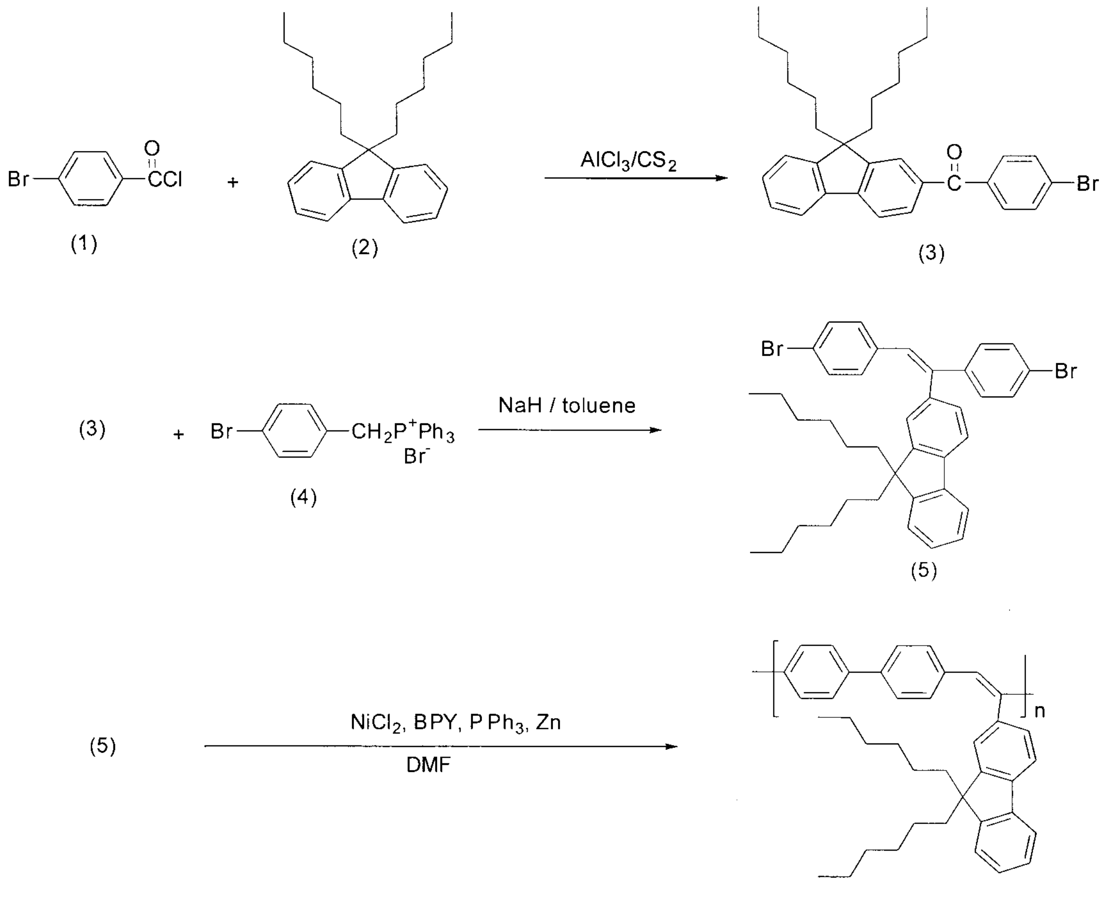
Figure 1. TGA thermogram of PBPHFV.

Instrument. Melting points were determined using an Electrothermal Mode 1307 digital analyzer. ^1H NMR and ^{13}C NMR spectral data were expressed in ppm relative to the internal standard and were obtained on a DRX 500 MHz NMR spectrometer. FT-IR spectra were obtained with a Bomem Michelson series FT-IR spectrometer, and the UV−vis absorption spectra were obtained in chloroform on a Shimadzu UV-3100 spectrophotometer. Molecular weight and polydispersity of the polymer were determined by gel permeation chromatography (GPC) analysis with polystyrene standard calibration (Waters high-pressure GPC assembly model M590 pump, μ-styrngel columns of 105, 104, 103, 500, and 100 Å, refractive index detectors, solvent THF). Elemental analyses were performed by Leco Co. CHNS-932. TGA measurements were performed on a Perkin-Elmer series 7 analysis system under N_2 at a heating rate of 10 °C/min. The photoluminescence spectra were recorded on a Perkin-Elmer LS-50 fluorometer utilizing a lock-in amplifier system with a chopping frequency of 150 Hz. For the measurements of device characteristics, current−voltage (*I*−*V*) changes were measured using a current/voltage source (Keithley 238) and an optical power meter (Newport 818-SL). Thickness of films was determined with a Sloan Dektak.

Results and Discussion

The method for preparing this polymer is outlined in Scheme 1. First, the Friedel−Crafts acylation of 9,9-dihexylfluorene was carried out with 4-bromobenzoyl chloride. The resulting 9′,9′-dihexyl-3-fluorenyl-4″-bromophenyl ketone, which was recovered after recrystallization in good yield, was converted to BPHFE by the Wittig reaction. The polymerization was carried out using a nickel-catalyzed coupling reaction with good yield. After the polymerization, the end-capping reaction of the bromine end group, which hampers thermal stability and efficiency of PL, was accomplished by bromobenzene. The polymer structure shown was consistent with the elemental analysis and the spectroscopic data from ^1H NMR and FT-IR. The obtained polymer was readily soluble in common organic solvents such as chloroform, methylene dichloride, and toluene. The weight-average molecular weight of the polymer, as determined by gel permeation chromatography using polystyrene standard, was $M_w = 15\,500$ (PDI = 1.5). The thermal property of the synthesized polymer was evaluated by the means of TGA under nitrogen atmosphere. TGA curve shows that the polymer exhibits good thermal stability (Figure 1). The weight loss of the polymer is less than 5% on heating to 420 °C. The DSC measurement showed glass transition at around 136 °C.

Scheme 1



PBPHFV

Figure 2 shows the optical absorption and photoluminescence spectra of a dilute solution of the polymer in chloroform. The absorption spectrum of the dilute solution has a maximum peak of 370 nm, with a shoulder at 450 nm. The absorption spectrum of the thin film also shows the maximum absorption and the absorption edge is about 370 and 450 nm, respectively.

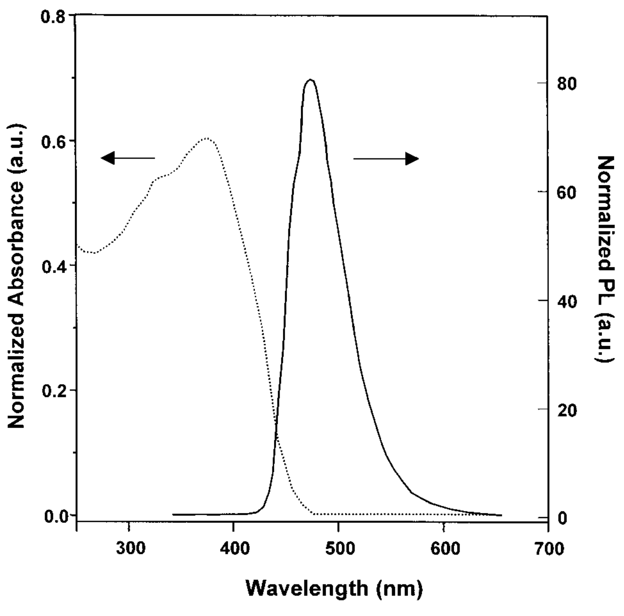


Figure 2. UV–vis absorption and photoluminescence spectra of PBPHFV in chloroform.

The maximum absorption peak of the polymer is 60–70 nm blue-shifted as compared with that of PPV[21] and 20–30 nm red-shifted as compared with that of PPP[22] (Figure 3). This shift may originate from the biphenylenevinylene units composed of alternating PPP and PPV units. In contrast to other soluble conjugated polymers, the absorption spectra of dilute solution and film of the

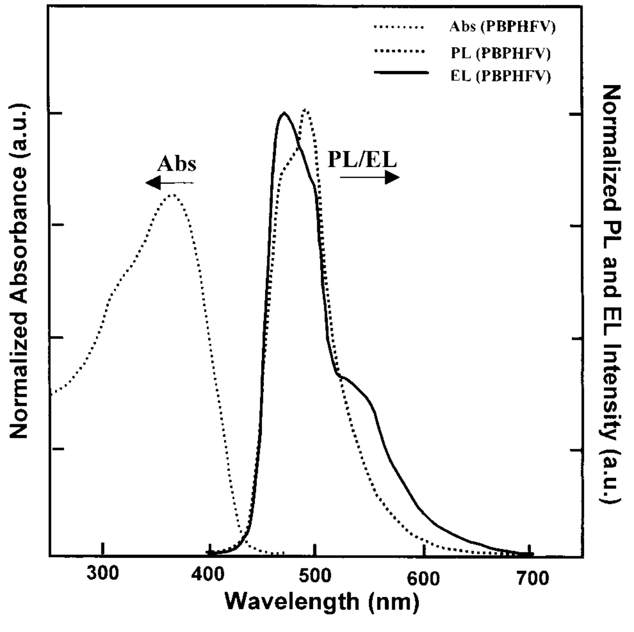


Figure 3. Solid-state absorption, photoluminescence, and electroluminescence spectra of PBPHFV.

Table 1. Characteristics of Electroluminescence Device[a]

	max luminescence	turn-on V	EL (λ_{max}), nm	max efficiency
PBPHFV	4116 cd/m^2 (8.6 V, 1.18 A/cm^2)	4.3 V (0.089 mA)	465	0.2157 lm/W (45 cd/m^2, 5.8 V, 0.011 A/cm^2)
PBPHFV:PVK (10:1)	4268 cd/m^2 (8.2 V, 0.85 A/cm^2)	4.1 V (0.229 mA)	466	0.8917 lm/W (410 cd/m^2, 5.8 V, 0.025 A/cm^2)
PBPHFV:PVK (5:1)	3981 cd/m^2 (8.2 V, 0.79 A/cm^2)	4 V (0.064 mA)	467	0.9217 lm/W (260 cd/m^2, 5.6 V, 0.016 A/cm^2)
PBPHFV:PVK (1:1)	9112 cd/m^2 (14.4 V, 0.43 A/cm^2)	4.8 V (0.009 mA)	470	1.3488 lm/W (44 cd/m^2, 6.6 V, 0.002A/cm^2)
PBPHFV:PVK (1:5)	9342 cd/m^2 (20.6 V, 0.16 A/cm^2)	7 V (0.014 mA)	475	1.6574 lm/W (40 cd/m^2, 11.8 V, 0.0007 A/cm^2)
PBPHFV:PVK (1:10)	7284 cd/m^2 (22.4 V, 0.19 A/cm^2)	6.8 V (0.028 mA)	480	0.7601 lm/W (41 cd/m^2, 14.8 V, 0.001 A/cm^2)

[a] Device structure was ITO/PEDOT/polymer/LiF/Al. The thickness of polymer film was 1000 Å.

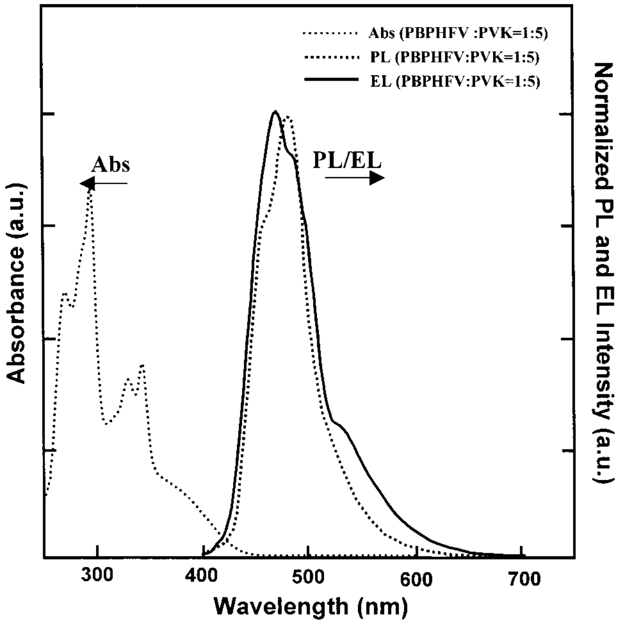


Figure 4. Solid-state absorption, photoluminescence, and electroluminescence spectra of (PBPHFV:PVK = 1:5).

polymer are similar, indicating that the conformation and intermolecular interactions of the polymer are similar in solution and solid state.[23]

The PL spectrum of the dilute solution of the polymer pumped by UV light (λ = 365 nm) has a maximum peak at 470 nm and a weak shoulder at around 460 nm. Although the PL spectrum of a solid film of PBPHFV shows a slight bathochromic shift from that of the solution, it is blue-shifted by 20−30 nm from that of poly(4,4′-diphenyldiphenylenevinylene) (PDPV) which has diphenyl substituents at the vinyl position. This result may be explained by the fact that the steric hindrance induced by the 9,9-dihexylfluorene substituent seems to cause a twist of the polymer backbone, leading to reduced effective conjugation lengths, even though the 9,9-dihexylfluorene substituent has slightly electron-donating effects.

The polymer was blended with the poly(vinylcarbazole) [PVK] which serves several functions in the blend of emitting polymer materials.[24,25] Figure 4 shows the absorption, PL, and electroluminescence spectra of the blend polymer (PBPHFV:PVK = 1:5). The PL spectrum of the blend polymer is similar to that of the dilute solution of PBPHFV.

The electroluminescence from the ITO/PEDOT/PBPHFV/LiF/Al device using PEDOT as a hole injection layer[26] and LiF as an electron injection layer[27] shows the maximum at 465 nm and the shoulder at around 485 nm (Figure 3). The electroluminescence spectrum is similar to the PL spectrum of PBPHFV except for a small shoulder appearing around 550 nm. This small shoulder may have resulted from interchain excimer emission. The electroluminescence spectrum of the ITO/PEDOT/(PBPHFV:PVK = 1:5)/LiF/Al device shows a maximum at 470 nm and a more suppressed minor shoulder at 550 nm (Figure 4). This result suggests that the blended PVK decreases the interchain excimer by a dilution effect.

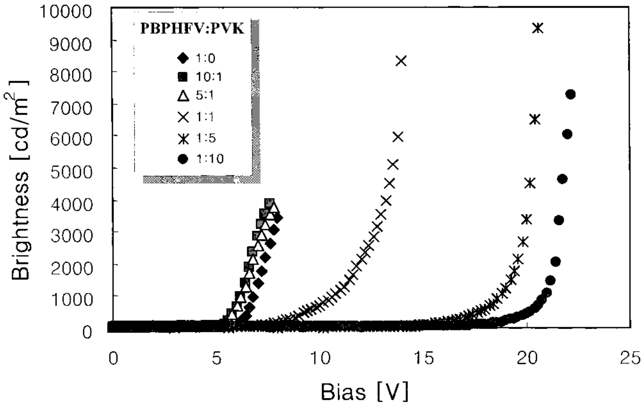


Figure 5. Luminescence−voltage characteristic of PBPHFV and blended polymer.

Table 1 shows the characteristics of the EL device using PBPHFV as an emitting material. Figure 5 shows the luminescence−voltage characteristic of PBPHFV and the blended polymer. The EL device prepared from the PBPHFV by using a PEDOT as hole injection layer, aluminum as a cathod, and LiF as an electron injection layer showed a turn-on voltage of 4.3 V (0.089 mA/cm^2), efficiency of 0.22 lm/W (45 cd/m^2, 5.8 V, 0.011 A/cm^2), and maximum brightness of 4116 cd/m^2 (8.6 V, 1.18 A/cm^2). As the ratio of PVK to PBPHFV is increased to (PBPHFV:PVK = 1:5), the maximum brightness and efficiency of the device increased though the turn-on voltage of device slightly increased. This suggests that the increase of maximum brightness and efficiency is caused by the dilution effect of PVK, which suppressed the interchain excimer, and/or by the hole transporting role of PVK. The EL device prepared from the blend with PVK (PBPHFV:PVK = 1:5) showed a turn-on voltage of 7 V(0.014 mA), efficiency of 1.66 lm/W (41 cd/m^2, 14.8 V, 0.01 A/cm^2), and maximum brightness of 9342 cd/m^2 (20.6 V, 0.16 A/cm^2).

The polymer showed a relatively low turn-on voltage, high quantum efficiency, and brightness. This high blue luminescence behavior of PBPHFV may be attributed to the biphenylenevinylene backbone, having the advantage of both PPV and PPP, and steric effects of 9,9-dihexylfluorene, which prevent the effective conjugation length, the formation of eximers owing to interchain interactions, and π-staking between the conjugated segments.

Conclusion

A new blue-light-emitting conjugated polymer, PBPHFV, having advantages of both PPP and PPV, has been designed and synthesized. The introduction of the large pendant group, 9,9′-dihexylfluorene, into the vinyl bridge led to steric hindrance which shortens the effective conjugation length and interrupts interchain interactions as well as enhances solubility. PBPHFV showed high thermal stability. The LEDs based on PBPHFV were shown to have low turn-on voltages, high brightness, and good efficiencies.

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