Stabilized Blue Emission from Organic Light-Emitting Diodes Using Poly(2,6-(4,4-bis(2-ethylhexyl)-4*H*-cyclopenta[*def*]phenanthrene))

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ABSTRACT: A new electroluminescence (EL) polymer utilizing a new backbone, poly(2,6-(4,4-bis(2-ethylhexyl)-4*H*-cyclopenta-[*def*]phenanthrene)) (PCPP), with stabilized blue emission is reported. PCPP was synthesized from 4*H*-cyclopenta[*def*]phenanthrene over five steps. The photoluminescence (PL) spectra of PCPP did not show any peak in the long wavelength region even after annealing for 18 h at 150 °C in air. The OLED with the configuration of ITO/PEDOT:PSS/PCPP/Al generates EL emission with a maximum peak at 400 nm, low turn-on voltage (6.0 V), and excellent CIE coordinates ($x = 0.17$, $y = 0.12$) for the blue color without any filtering. The maximum brightness of the OLEDs using PCPP was 1500 cd/m^2 at 14 V. The maximum luminescence efficiency of the polymer LEDs with PCPP was 0.70 cd/A at 180 mA/cm^2. In addition to this, PCPP displays stabilized blue EL emission without any low-energy emission band in the region of 500−600 nm even after annealing for 18 h at 150 °C or operation of the device for 40 min in air.

Introduction

Conjugated polymers have been incorporated as active materials into several kinds of electronic devices such as transistors[1] and organic light-emitting diodes[2] (OLEDs), including flexible displays.[3] In the field of polymer-based electroluminescent devices, much interest has been paid to the main-chain conducting polymers such as poly(phenylenevinylene) (PPV),[2] poly(*p*-phenylene) (PPP),[4] poly(thiophene),[5] poly(fluorene),[6] their copolymers and soluble derivatives because of the potential application as large-area light-emitting diodes (LEDs).[7] Conjugated polymers with large band gaps, especially poly(fluorenes) (PFs),[8,9] have acquired much interest since they emit blue light and also enable full color via energy transfer to longer wavelength emitters.[10] PFs, including poly(2,7-(9,9-bis(2-ethylhexyl)fluorene) (PF2/6),[11] are a very attractive class of blue materials because they have a rigid biphenyl unit to obtain a large band gap for the efficient blue emission and the remote C-9 position to provide the desirable solubility for the processability of polymers without appreciably increasing the steric interactions in the polymer backbone.[8] The PFs show high photoluminescence (PL) efficiency and good charge transport. However, one of the problems of using polyfluorenes (PFs),[8,9,12] the most promising polymers for blue OLEDs, is their tendency to generate long wavelength emission around 550 nm either during annealing or passage of current, turning the desired blue emission color into the undesired blue-green emission and a drop of electroluminescence (EL) quantum efficiency.[13−15] To remove strong low-energy emission caused by the formation of keto defect sites[12] or aggregates/excimers,[9] several successful approaches have been reported, including the use of bulky end groups,[16] the use of cross-linkable groups,[17] the copolymerization with anthracene[18] or siprofluorene,[19] the control of the molecular weight distribution of PFs,[15] and the complete dialkylation at C-9 position.[20]

Here we report the synthesis and properties of a new EL polymer utilizing a new backbone, poly(2,6-(4,4-bis-(2-ethylhexyl)-4*H*-cyclopenta-[*def*]phenanthrene)) (PCPP), with stabilized blue emission. PL and EL spectra of PCPP do not show any peak in the long wavelength region which corresponds to keto defect sites or aggregates/excimers even after annealing for 18 h in air or operation of the device for 40 min.

Experimental Section

General. All reagents used, including 4*H*-cyclopenta[*def*]-phenanthrene, were purchased from Aldrich and used without further purification. Solvents were purified by normal procedures and handled under moisture-free atmosphere. ^{1}H and ^{13}C NMR spectra were recorded with a Varian Gemini-300 (300 MHz) or a Varian Gemini-500 (500 MHz) spectrometer, and chemical shifts were recorded in ppm units with TMS as the internal standard. UV spectra were recorded with a Varian CARY-5E UV/vis spectrophotometer. Molecular weight and polydispersities of the polymer were determined by gel permeation chromatography (GPC) analysis with a polystyrene standard calibration. An Oriel InstaSpec VI CCD detection systems of Busan Branch of Korea Basic Science Institute was used for photoluminescence spectroscopy. For the determination of device characteristics, current−voltage ($I-V$) 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. Cyclic voltammetric waves were produced by using a EG&G Parc model 273 potentiostat/galvanostat at a constant scan rate of 100 mV/s.

Synthesis of 4,4-Bis(2-ethylhexyl)-4*H*-cyclopenta[*def*]-phenanthrene (2). To a stirred solution of 1 g (5.3 mmol) of 4*H*-cyclopenta[*def*]phenanthrene (**1**) and catalytic amounts of triethylbenzylammonium chloride in 20 mL of DMSO under nitrogen was added 2.25 mL (12.61 mmol) of 2-ethylhexyl bromide. After 1 h at 60 °C, 5 mL of 50% aqueous NaOH solution was added. The reaction mixture was cooled to room temperature and stirred for 5 h. An excess amount of ethyl acetate was added to the reaction mixture to generate the precipitation of NaOH. After filtering off the precipitated

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NaOH, the organic layer was washed with 100 mL of 1.0 M aqueous HCl solution and 150 mL of distilled water. The organic phase was dried with $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash column chromatography (30 × 200 mm column, SiO_2, 100% of *n*-hexane) twice to give 2 g (91.0%) of extremely clean compound **2**, colorless oil: R_f 0.64 (SiO_2, hexane 100%). IR (CCl_4): ν 3049, 2958, 2926, 2872, 2557, 1922, 1506, 1458, 1442, 1419, 1379 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ 0.46−0.92 (m, 30H), 2.19 (d, 2H $J = 4.2$ Hz), 2.30 (d, 4H $J = 5.0$ Hz), 7.52−7.63 (m, 4H), 7.77−7.81 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 10.14, 13.82, 22.50, 27.09, 28.05, 33.89, 34.97, 44.02, 58.77, 120.34, 122.56, 125.12, 126.83, 127.60, 137.34, 149.06. HRMS (*m/z*, FAB^+) Cald for $C_{31}H_{42}$ 414.3287; found 414.3247.

4,4-Bis(2-ethylhexyl)-8,9-dihydro-4*H*-cyclopenta[*def*]-phenanthrene (3). The mixture of 4,4-bis(2-ethylhexyl)-4*H*-cyclopenta[*def*]phenanthrene (**2**) (2 g, 4.82 mmol), 10% palladium−charcoal (1 g), and ethanol (100 mL) was shaken for 20 h at room temperature under a hydrogen atmosphere (1 atm). After removal of the catalyst by filtration, the ethanol was evaporated under reduced pressure. The residue was purified by flash column chromatography (30 × 200 mm column, SiO_2, 100% of *n*-hexane) to give 1.8 g (90%) of compound **3**, colorless oil: R_f 0.64 (SiO_2, hexane 100%). IR (CCl_4): ν 3047, 3018, 2957, 2956, 2872, 2857, 1457, 1435, 1418, 1377 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.45−0.94 (m, 30H), 2.00 (d, 4H $J = 5.1$ Hz), 3.11 (s, 4H), 7.05−7.07 (m, 2H) 7.09−7.16 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 10.31, 13.97, 22.66, 26.15, 27.05, 28.27, 33.85, 34.88, 43.95, 58.09, 121.87, 124.52, 126.79, 129.98, 138.45, 147.69. HRMS (*m/z*, FAB^+) Calcd for $C_{31}H_{44}$ 416.3443; found 416.3461.

Synthesis of 2,6-Dibromo-4,4-bis(2-ethylhexyl)-8,9-dihydro-4*H*-cyclopenta[*def*]phenanthrene (4). To prepare copper bromide adsorbed on alumina,[21] a solution of 10 g of copper bromide in 30 mL of distilled water was treated with neutral alumina (20 g, Merck aluminum oxide 90 active neutral) at room temperature. The reaction mixture was concentrated at 80 °C under reduced pressure. The resulting residue was dried under vacuum (4 Torr) at 100 °C for 15 h to generate the copper bromide adsorbed on alumina. To a stirred solution of 1.5 g (3.59 mmol) of 4,4-bis(2-ethylhexyl)-8,9-dihydro-4*H*-cyclopenta[*def*]phenanthrene (**3**) in 80 mL of carbon tetrachloride at room temperature was added 11.67 g of copper bromide adsorbed on alumina. After 5 h at 80 °C, the solid was filtered and washed with 2 × 30 mL of carbon tetrachloride. The combined organic phase was concentrated under reduced pressure and purified by flash column chromatography (30 × 200 mm column, SiO_2, 100% hexane) to give 1.88 g (91%) of compound **4,** pale yellow oil: R_f 0.58 (SiO_2, hexane 100%). IR (CCl_4): ν 3046, 2958, 2928, 2823, 2857, 1576, 1458, 1435, 1414, 1379, 1198, 1067 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 0.57−0.95 (m, 30H), 1.94 (d, 4H $J = 5.1$ Hz), 3.07 (s, 4H), 7.23 (s, 2H), 7.29 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 10.59, 14.25, 22.94, 25.88, 27.45, 28.45, 34.03, 35.29, 43.94, 58.97, 121.25, 125.64, 128.37, 131.66, 136.55, 149.48. HRMS (*m/z*, FAB^+) Calcd for $C_{31}H_{42}{}^{79}Br^{79}Br$ 572.1653, found 572.1640; calcd for $C_{31}H_{42}{}^{79}Br^{81}Br$ 574.1633, found 574.1625; calcd for $C_{31}H_{42}{}^{81}Br^{81}Br$ 576.1612, found 576.1566.

2,6-Dibromo-4,4-bis(2-ethylhexyl)-4*H*-cyclopenta[*def*]-phenanthrene (5). To a stirred solution of 1.5 g (2.61 mmol) of 2,6-dibromo-4,4-bis(2-ethylhexyl)-8,9-dihydro-4*H*-cyclopenta[*def*]phenanthrene (**4**) in 30 mL of carbon disulfide at room temperature was added dropwise 0.5 g (3.13 mmol) of Br_2 in carbon disulfide over 3 h. The reaction mixture was stirred for 1 h, concentrated under reduced pressure, and purified by flash column chromatography (30 × 200 mm column, SiO_2, 100% hexane) to give 1.2 g (80%) of compound **5**, white oil: R_f 0.44 (SiO_2, hexane 100%). IR (CCl_4): ν 3046, 2959, 2926, 2872, 2858, 2728, 1909, 1744, 1593, 1577, 1460, 1416, 1390, 1379, 1367, 1306, 1222, 1213, 1066 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 0.47−0.87 (m, 30H), 2.17 (d, 4H $J = 5.1$ Hz), 7.64 (s, 2H), 7.74 (s, 2H), 7.95 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 10.53, 14.20, 22.86, 27.55, 28.28, 34.06, 35.45, 44.03, 59.72, 121.84, 124.68, 125.63, 125.89, 128.64, 135.44, 150.97. Calcd for $C_{31}H_{40}{}^{79}Br^{79}Br$ 570.1497, found 570.1532; calcd for $C_{31}H_{40}{}^{79}Br^{81}Br$ 572.1476, found 572.1495; calcd for $C_{31}H_{40}{}^{81}Br^{81}Br$ 574.1456, found 574.1454.

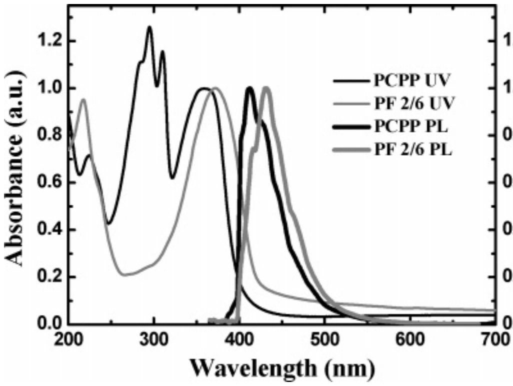
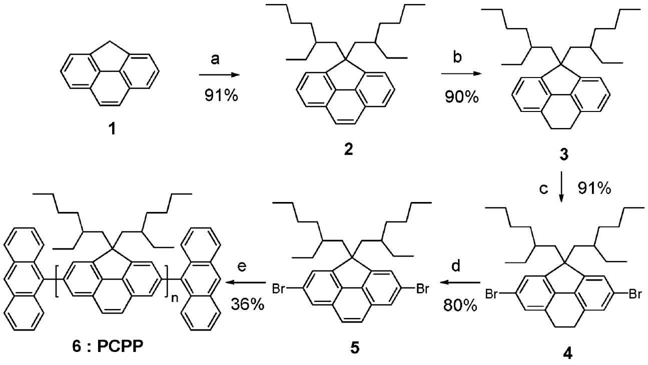


Figure 1. Normalized UV−vis absorption and photoluminescence spectra of PCPP and PF2/6 in the solid state.

Scheme 1. (a) $C_6H_{13}Br$, 50% Aqueous NaOH, DMSO; (b) Pd/C, H_2, EtOH; (c) $CuBr_2Al_2O_3$, CCl_4; (d) CS_2, Br_2; (e) $Ni(COD)_2$, 2,2′-Bispyridine, Cyclooctadiene, 9-Bromoanthrecene, DMF/Toluene



Poly(2,6-(4,4-bis(2-ethylhexyl)-4*H*-cyclopenta-[*def*]-phenanthrene)) (6). In a three-neck flask were placed bis-(1,5-cyclooctadiene)nickel(0) ($Ni(COD)_2$) (349 mg, 1.27 mmol), 2,2′-dipyridyl (198 mg, 1.27 mmol), cyclooctadiene (0.16 mL, 1.27 mmol), and DMF (5 mL). After three freezing−thaw cycles, the catalyst was heated to 80 °C for half an hour to form the purple complex.[23] To the reaction mixture, 300 mg (0.52 mmol) of 2,6-dibromo-4,4-bis(2-ethylhexyl)-4*H*-cyclopenta[*def*]phenanthrene (**5**) in toluene (5 mL) was added. After 3 days heating at 80 °C, 500 mg of 9-bromoanthracene, the end-capper, dissolved in 5 mL of anhydrous toluene was added to the reaction mixture. The reaction mixture was further heated at 80 °C for 24 h. After cooling to room temperature, the reaction mixture was poured into the solution of 100 mL of aqueous HCl solution, 100 mL of acetone, and 200 mL of methanol and stirred for 3 h. The solid was filtered and dissolved with a minimum amount of chloroform. A minimum amount of methanol was added to generate precipitation. The pale yellow solid was purified by Soxhlet extraction with methanol and dried in a vacuum for 48 h to generate 77 mg of PCPP, white powders. Anal. Calcd: C, 90.23; H, 9.77. Found: C, 89.89; H, 9.98.

Results and Discussion

Synthesis and Characterization. The general synthetic routes toward the monomers and polymer are outlined in Scheme 1. For the preparation of PCPP, in the first step, 4,4-bis(2-ethylhexyl)-4*H*-cyclopenta[*def*]-phenanthrene (**2**) was synthesized from commercially available 4*H*-cyclopenta[*def*]phenanthrene (**1**),[21] with 2-ethylhexyl bromide and a catalytic amount of

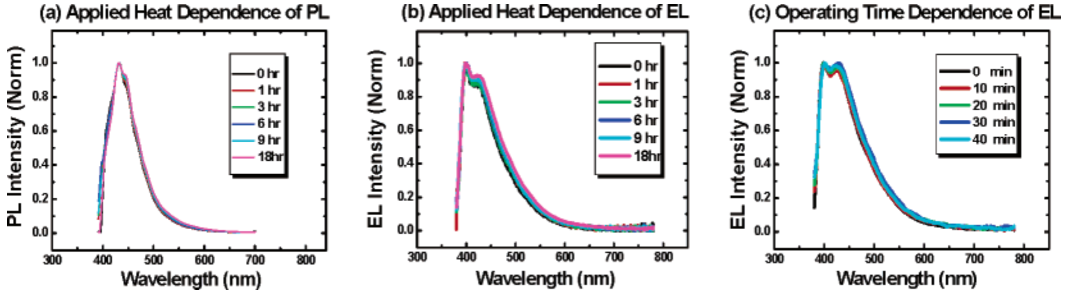


Figure 2. (a) PL spectra of PCPP after annealing the thin film at 150 °C in air. (b) EL spectra of PCPP after annealing the device (ITO/PEDOT:PSS/polymer/Al) at 150 °C in air. (c) EL spectra of PCPP after operation of the device for different time in air.

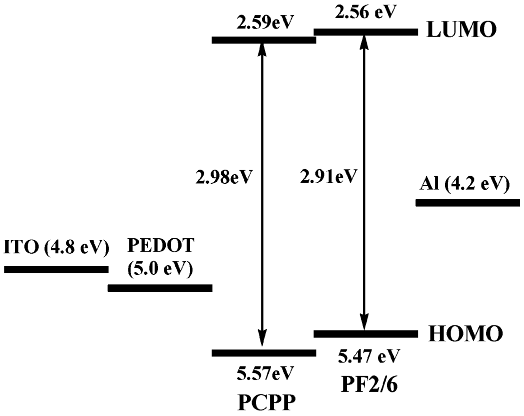


Figure 3. Energy band diagram of PCPP and PF.

triethylbenzylammonium chloride. After flash chromatography twice with hexane, compound **2** was extremely clean without any monoalkylated byproduct depending on the 500 MHz proton NMR.

Compound **2** was hydrogenated using 10% palladium on carbon and then brominated by alumina-supported copper(II) bromide[22] to generate dibromide **4** with excellent selectivity. 2,6-Dibromo-4,4-bis(2-ethylhexyl)-4*H*-cyclopenta[*def*]phenanthrene (**5**) was synthesized by the dehydrogenation using bromine in carbon disulfide.[23] The polymerization of the monomer **5** was affected under Yamamoto conditions,[24] using bis(1,5-cyclooctadiene)nickel(0) (Ni(COD)) catalyst, 2,2′-dipyridyl, and cyclooctadiene. The end-capper, 9-bromoanthracene, was also used[25] to generate the desired polymer **6**, PCPP. PF2/6, with anthracene end-capping groups, was also synthesized using a method similar to that with PCPP.

The resulting PCPP, white polymer, was soluble in organic solvents such as chloroform, tetrahydrofuran, dichloromethane, and chlorobenzene. Molecular weights and polydispersities of the polymer were determined by gel permeation chromatography (GPC) analysis with a polystyrene standard calibration. The number-average molecular weight (M_n) and the weight-average molecular weight (M_w) of the PCPP were 15 000 and 50 000 with a polydispersity of 3.3 as determined by GPC using THF as the eluent and polystyrene as the standard. The thermal properties of the polymer was determined by DSC and thermal gravimetric analysis under a nitrogen atmosphere at a heating rate of 10 °C/min. PCPP shows a glass transition at 180 °C and loses less than 5% of their weights on heating to 400 °C.

Optical and Photoluminescence Properties. UV spectra were recorded with a Varian CARY-5E UV/vis spectrophotometer. The PL and EL spectra of the device were measured using an Oriel InstaSpec CCD detection systems. For PL spectrum measurements, xenon lamp was used as the excitation source, and incident beam took the maximum absorption peak of polymers. The UV−vis absorption spectrum and PL spectrum, which were obtained with the thin film by spin-coating on quartz plates from the polymer solutions in chlorobenzene, are shown in Figure 1. The absorption spectrum of PCPP and PF2/6 exhibited the maximum peaks at 359 and 372 nm, which were contributed from the $\pi-\pi^*$ transition of the conjugated main chains, respectively. The maximum absorption peak of PCPP was more blue-shifted as compared to that of PF2/6. The PCPP exhibited an additional absorption peak at 294 and 310 nm due to the $\pi-\pi^*$ transition of cyclopenta[*def*]-phenanthrene units. The absorption onset wavelengths of PCPP and PF2/6 were 416 and 426 nm, which correspond to band gaps of 2.98 and 2.91 eV, respectively.

The PL spectrum of PCPP consists of typical vibronically structured bands comprising a maximum at 410 nm and a shoulder. The PL spectrum of PCPP was blue-shifted about 20 nm relative to the corresponding features measured from PF2/6, as shown in Figure 1. The PL spectra of the thin films of PCPP after annealing the device for up to 18 h at 150 °C in air were also obtained. As shown Figure 2a, the PL spectra of PCPP did not show any peak in the long wavelength region which corresponds to keto defect sites or aggregates/excimers even after annealing for 18 h at 150 °C in air.

Electrochemical Properties of the Polymers. The energy band diagrams of the polymers were determined from the band gaps, which were estimated from the absorption edges, and the HOMO energy levels, which were estimated from cyclic voltammetry.[26] 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. A Pt wire and an Ag/AgNO$_3$ electrode were used as the counter electrode and reference electrode, respectively. After coating, the film adhering to the electrode was dried in a vacuum oven for 10 h; cyclic voltammetric waves were produced by using a EG&G Parc model 273 potentiostat/galvanostat. During the anodic scan, the oxidation onset potentials of PCPP were in the range of 1.0 eV and exhibited an irreversible p-doping process. From the onset potential for the oxidation, the HOMO energy level of the polymer was estimated regarding the energy level of the FOC reference.[27] The HOMO energy level of the present PCPP is about 5.57 eV. The LUMO energy of PCPP, calculated

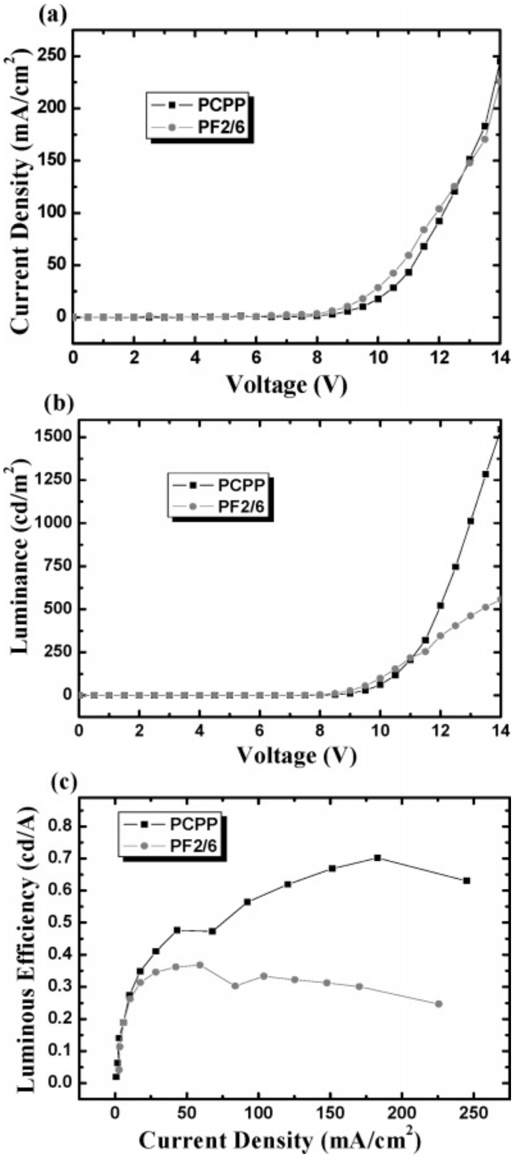


Figure 4. Current density−voltage−luminescence ($J-V-L$) characteristics and efficiencies of PLEDs of PCPP and PF2/6 with a configuration of ITO/PEDOT:PSS/polymer/Al. (a) Current density−voltage characteristics, (b) luminescence−voltage characteristics, and (c) efficiencies.

from the values of the band gap and HOMO energy level, is 2.59 eV.

Electroluminescent Properties and Current−Voltage−Luminance. 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. The PEDOT:PSS was spin-coated from aqueous solution with isopropyl alcohol (10 wt %) on the surface-treated ITO substrate. On top of the PEDOT layer, the emissive polymer film was obtained by spin-casting an ODCB (*o*-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 a vacuum, and the aluminum electrode were deposited on the top of the polymer film through a mask by vacuum evaporation at pressure below about 10^{-6} mbar, yielding an active area of 4 mm^2. The current density−voltage and luminescence−voltage characteristics were measured using a Keithley 236 source measure unit. All processing steps and measurements mentioned above were carried out in air and at room temperature.

The EL spectrum of PCPP in the ITO/PEDOT:PSS/PCPP/Al configuration exhibited a maximum peak at 400 nm, which corresponds to the blue light, and was also blue-shifted about 20 nm as compared to that of PF2/6. The voltage−current density characteristics of the devices fabricated from PCPP revealed that the turn-on voltage was ≈6.0 V. The maximum brightness of PCPP and PF2/6 was 1500 and 550 cd/m^2 at 14 V, respectively. The maximum luminescence efficiencies of the polymer LEDs with PCPP and PF2/6 at room temperature were 0.70 cd/A at 180 mA/cm^2 and 0.36 cd/A at 60 mA/cm^2, respectively. The 1931 Commission Internationale de L'Eclairage CIE coordinates of the EL spectra of PCPP were $x = 0.17$, $y = 0.12$ without any filtering.

The EL spectra of OLEDs fabricated from in the ITO/PEDOT:PSS/polymer/Al configuration were obtained after annealing the device for up to 18 h at 150 °C in air. As shown Figure 2b, the EL spectra of the OLED from PCPP were not changing without showing any peak in the region of 500−600 nm even after annealing for 18 h at 150 °C in air. The change of the EL spectrum on operation of the ITO/PEDOT:PSS/polymer/Al device is shown in Figure 2c. After operation of the device for up to 40 min, the initial EL spectrum of the OLED from PCPP was cleanly preserved without showing any peak in the region of 500−600 nm even after operation of the device for 40 min in air.

Although the reason for the stable blue emission of PCPP is unclear, there are several possible speculations possible. Complete removal of the monoalkylated byproduct, depending on 500 MHz proton NMR, eliminated the possibility of keto-defect formation. Or, it could be possible that PCPP may have more difficulty to form the aggregates.

Conclusion

In conclusion, we present here the synthesis of a new conjugated polymer, PCPP. The PL spectra of PCPP thin film did not show any peak in the long wavelength region even after annealing for 18 h at 150 °C in air. The OLED with the configuration of ITO/PEDOT:PSS/PCPP/Al generates EL emission with high maximum brightness (1500 cd/m^2 at 14 V), high efficiency (0.70 cd/A at 180 mA/cm^2), maximum peak at 400 nm, low turn-on voltage (6.0 V), and excellent CIE coordinates ($x = 0.17$, $y = 0.12$) for the blue color without any filtering. Moreover, this polymer displays stabilized blue EL emission without any low-energy emission band in the region of 500−600 nm even after annealing for 18 h at 150 °C or operation of the device for 40 min.

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