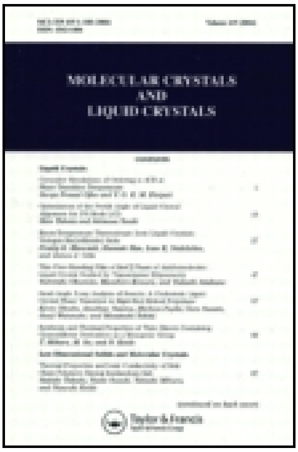

Molecular Crystals and Liquid Crystals
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl20

Synthesis and Characterization of Carbazole-based Copolymers Containing Benzothiadiazole Derivative for Polymer Light-Emitting Diodes

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To cite this article: Jin Su Park , Sung-Ho Jin , Yeong-Soon Gal , Jun Hee Lee & Jae Wook Lee (2012): Synthesis and Characterization of Carbazole-based Copolymers Containing Benzothiadiazole Derivative for Polymer Light-Emitting Diodes, Molecular Crystals and Liquid Crystals, 567:1, 102-109

To link to this article: http://dx.doi.org/10.1080/15421406.2012.702439

Mol. Cryst. Liq. Cryst., Vol. 567: pp. 102–109, 2012
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ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2012.702439

Synthesis and Characterization of Carbazole-based Copolymers Containing Benzothiadiazole Derivative for Polymer Light-Emitting Diodes

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*A new thermally robust electroluminescent (EL) carbazole-based π-conjugated copolymer, including poly[3,7-(N-hexylcarbazole)-co-4,7-{5,6-bis(3,7-dimethyloctyloxymethyl)-2,1,3-(benzothiadiazole)}] (**PCz-co-P2C$_{10}$BT**) was synthesized and used to fabricate the efficient polymer light-emitting diodes (PLEDs). The glass transition temperature of the **PCz-co-P2C$_{10}$BT** (105°C) was found to be higher than that of poly(9,9-dialkylfluorene) derivatives. We fabricated PLEDs in ITO/PEDOT/light-emitting polymer/Alq$_3$/LiF/Al configuration. The new copolymer was found to have green emission color (523 nm). The maximum brightness and external quantum efficiency of **PCz-co-P2C$_{10}$BT** were 260 cd/m^2 at 14 V and 0.22%, respectively.*

Keywords benzothiadiazole; carbazole; polymer light-emitting diodes; PhOLEDs;

Introduction

Ever since Tang and Vanslyke constructed organic light-emitting diodes (OLEDs) using organic materials as emitters, OLEDs continue to attract intensive interest because of their numerous applications in full-color flat-panel displays and other lighting sources [1,2].

Polymer light-emitting diodes (PLEDs) have received remarkable scientific and industrial attention due to their potential applications in large area flat-panel displays [3]. π-Conjugated polymers are attractive because of their applicability to PLEDs [4]. Among the various types of π-conjugated polymers reported, poly(N-alkylcarbazole) (PCz) and its derivatives exhibit the most promise for PLED applications and have been widely used, because of their chemical and thermal stability, high photoluminescent (PL) quantum efficiency, and ease of property tuning by copolymerization with various comonomers.

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PCz derivatives have strong absorption in the near UV region and a low redox potential. The electrochemical and spectroscopic properties of PCz derivatives have been extensively investigated [5]. Normally, PCz derivatives emit blue light with a wide band gap. However, by incorporating a narrow band gap comonomers into the carbazole backbone, the emission colors of PCz derivatives could be tuned over the entire visible range [6]. The most commonly used comonomer is the electron deficient 2,1,3-benzothiadiazole (BT) unit, which emits the green color in PLEDs [7,8]. In this article, we report the synthesis and characterization of a new EL polymer, consisting of poly[3,7-(N-Hexylcarbazole)-*co*-4,7-{5,6-bis(3,7-dimethyloctyloxymethyl)-2,1,3-(benzothiadiazole)}] (**PCz-*co*-P2C$_{10}$BT**), aimed at increasing the solubility of resulting copolymer, tuning the emission color from blue to green, and improving the PLED performance. The new copolymer was synthesized with palladium-catalyzed Suzuki polymerizations and was fully characterized. The new EL polymer was used to fabricate efficient PLEDs using **PCz-*co*-P2C$_{10}$BT** as an emitting layer with ITO/PEDOT/polymer/Alq$_3$/LiF/Al configuration.

Experimental

Characterization

Carbazole, N-bromosuceinimide (NBS), sodium hydride, 1-bromohexane, n-butyl lithium, 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane, Alq$_3$, LiF, and aluminum were purchased from Aldrich Co., and used without further purification unless otherwise noted. Solvents were dried and purified by fractional distillation over sodium/benzophenone and handled in a moisture-free atmosphere. The moisture content of THF was below 10 ppm. Column chromatography was performed using silica gel (Merck, 250–430 mesh). The ^{1}H-NMR spectra were recorded on an Inova-500 spectrometer and the chemical shifts were recorded in ppm units with chloroform as the internal standard. The UV-visible and the emission spectra were recorded with a Shimadzu UV-3100 and Hitachi F-4500 fluorescence spectrophotometers. The solid state emission measurements were carried out by supporting each film on a quartz substrate, which was mounted to receive front-face excitation at an angle $< 45^o$. Polymer film was excited with several portions of the visible spectrum using a xenon lamp. The molecular weight and polydispersity of the polymer were determined by gel permeation chromatography (GPC) using a PL gel 5 μm MIXED-C column on an Agilent 1100 series liquid chromatography system with THF as an eluent and calibration with polystyrene standards. Thermal analyses were carried out on a Mettler Toledo TGA/SDTA 851, DSC 822 analyzer under an N$_2$ atmosphere at a heating rate of 10°C/min. Cyclic voltammetry (CV) was carried out in a 0.1 M solution of tetrabutylammonium tetrafluoroborate (Bu$_4$NBF$_4$) in anhydrous chloroform using a CHI600C at a potential scan rate of 50–100 mV/s. Polymer film was coated onto a Pt disc electrode (diameter: 0.2 cm) by dipping the electrode into the polymer solution (10 mg/mL). A platinum wire was used as the counter electrode and Ag/AgNO$_3$ electrode was used as the reference electrode. All electrochemical experiments were performed at room temperature in a glove box in an Ar atmosphere.

Synthesis of 3,6-Dibromo-9H-Carbazole (1)

To a solution of carbazole (5 g, 29.9 mmol) in anhydrous tetrahydrofurane (THF) was added NBS (10.64 g, 59.8 mmol) at room temperature. The reaction mixture was stirred at

85°C for 48 h. Then the reaction mixture was diluted with water and extracted with ethyl acetate. The combined organic layers were washed with brine, dried anhydrous $MgSO_4$ and concentrated. The product was purified by recrystallization. (1.9 g, 98%) ^{1}H-NMR (Acetone-d_6, δ ppm): 7.20–7.31 (m, 4H), 8.41 (s, 2H); ^{13}C-NMR ($CDCl_3$, δ ppm): 129.2, 121.1, 121.0, 119.7, 117.0, 113.3. Anal. Calcd for ($C_{12}H_7Br_2N$) : C 44.35, H 2.17, N 4.31 Found : C 46.12, H 2.24, N 4.13.

Synthesis of 3,6-Dibromo-N-Hexylcarbazole (2)

60 mL of dried THF in a 100 mL three neck flask was purged with a continuous N_2 flow. Sodium hydride (0.2 g, 7.6 mmol) was added into the flask to be stirred. 3,6-Dibromo-9H-carbazole (3 g, 9.2 mmol) was added slowly into the mixture. 1-Bromohexane (1.1 g, 7.6 mmol) diluted by THF (10 mL) was added dropwise into the flask and the content in the flask was refluxed for 12 h. The reactant was cooled to room temperature and then the solvent was stripped off by evaporation. The residue was mixed with H_2O (60 mL) and extracted with ethyl acetate. The extracted solution was dried with anhydrous $MgSO_4$ and then filtrate. Yellowish viscous liquid was obtained by evaporation of the solvent and was purified by employing a silica gel column with an eluent of an ethyl acetate:hexane:methylene chloride (1:7:2). The product was purified by recrystallization (4.36 g, 80%). ^{1}H-NMR ($CDCl_3$, δ ppm): 0.84 (m, 3H), 1.33 (m, 6H), 1.81 (m, 2H), 4.20 (t, 2H), 7.24 (d, 2H), 7.56 (q, 2H), 7.76 (d, 1H), 7.86 (s, 1H), 8.17 (s, 1H). ^{13}C-NMR ($CDCl_3$, δ ppm): 130.3, 121.1, 121.0, 120.0, 117.0, 113.3, 60.9, 31.5, 30.4, 27.0, 22.7, 14.01. Anal. Calcd for ($C_{18}H_{19}Br_2N$) : C 52.84, H 4.68, N 3.42 Found : C 50.94, H 4.86, N 3.55.

Synthesis of 3,6-bis(4,4,5,5-Tetramethyl-1,3,2-Tioxaborolan-2-yl)-N-Hexylcarbazole (3)

To a solution of 3,6-dibromo-N-hexylcarbazole (0.2 g, 0.49 mmol) in anhydrous THF (10 mL) at −78°C, *n*-butillithium was added by syringe. The mixture was stirred at −78°C for 2 hr. 2-Isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (0.2 ml, 1.1 mmol) was added slowly to the solution and the mixture was stirred for 2 h at −78°C. The resulting mixture was warmed to room temperature and stirred for 24 h. The mixture was poured into water and was extracted with diethyl ether. The organic layer was washed with brine and dried over anhydrous $MgSO_4$. The solvent was removed by distillation and the residue was purified by column chromatography. The product was white solid in 43% yield. ^{1}H-NMR ($CDCl_3$, δ ppm): 0.86 (t, 3H), 1.41 (m, 30H), 1.83 (t, 2H), 4.29 (t, 2H), 7.39 (d, 2H), 7.98 (d, 2H), 8.78 (s, 2H). ^{13}C-NMR ($CDCl_3$, δ ppm): 130.8, 122.0, 121.0, 120.0, 118.9, 111.1, 83.1, 60.9, 31.5, 30.4, 27.0, 22.7, 21.3, 14.1. Anal. Calcd for ($C_{30}H_{43}B_2NO_4$) : C 71.59, H 8.61, N 2.78, O 12.72 Found : C 74.31, H 8.95, N 2.89.

4,7-Dibromo-5,6-bis(Decyloxymethyl)Benzo[c][1,2,5]thiadiazole (4)

^{1}H-NMR ($CDCl_3$, δ ppm): 0.92 (m, 6H), 1.31–1.36 (m, 30H), 1.86–1.88 (m, 4H), 3.38 (t, 4H), 4.72 (d, 4H). ^{13}C-NMR ($CDCl_3$, δ ppm): 159.7, 142.3, 114.2, 72.0, 68.2, 31.8, 30.0, 29.6, 29.3, 25.9, 22.7, 14.1. Anal. Calcd for ($C_{28}H_{46}Br_2N_2O_2S$) : C 53.0, H 7.31, N 4.41, O 5.04 Found : C 51.04, H 7.60, N 4.56.

*Synthesis of Poly[3,7-(N-Hexylcarbazole)-*co*-4,7-{5,6-bis(3,7-Dimethyloctyloxymethyl)-2,1,3-(Benzothiadiazole)}] (PCz-*co*-P2C$_{10}$BT) (1:1 mol%)*

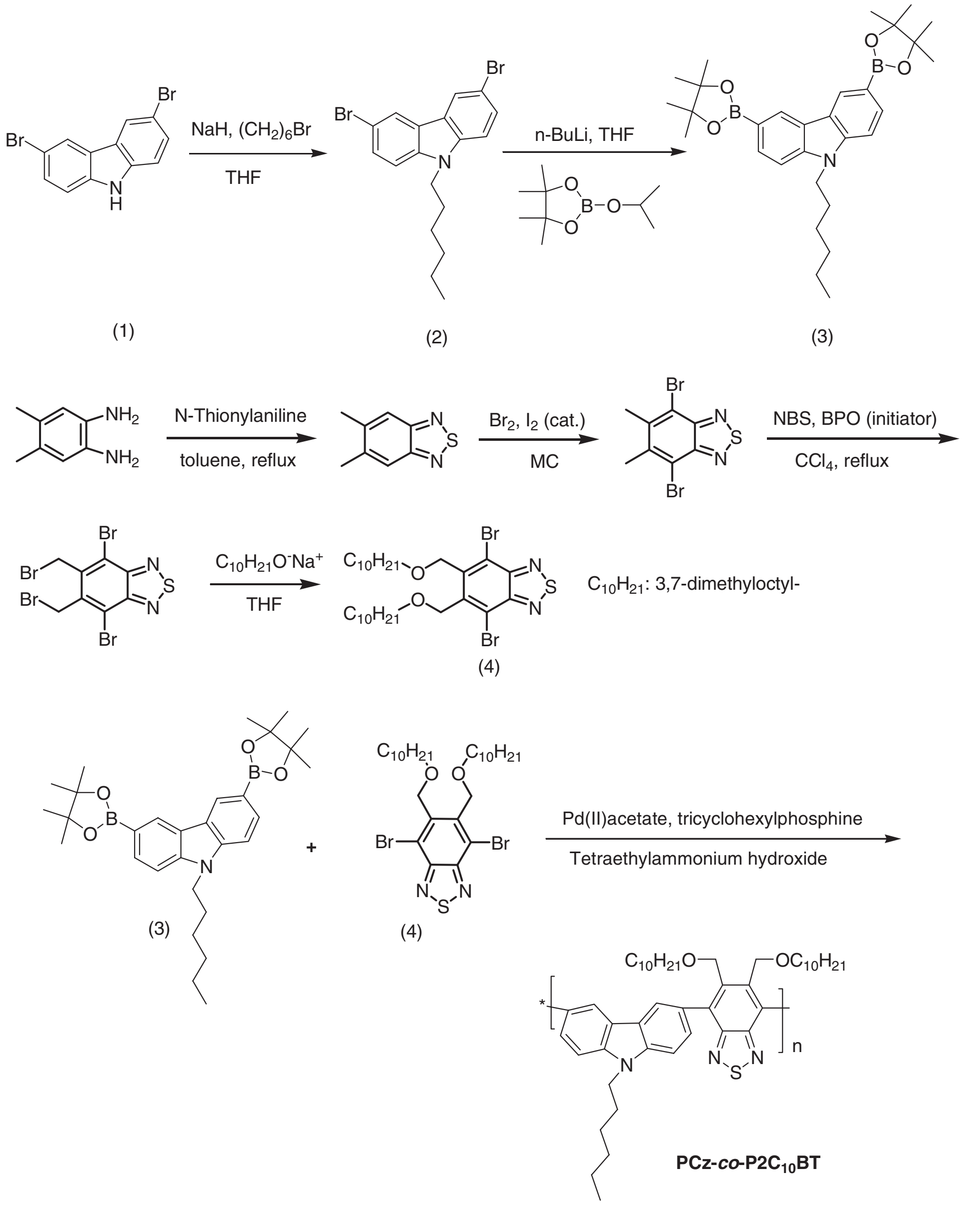
The copolymer was prepared by the Suzuki coupling reaction between 3,6-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-N-hexylcarbazole (3) and 5,6-bis(3,7-dimethyloctyloxymethyl)-4,7-dibromo-2,1,3-benzothiadiazole (4). A mixture of compound 3 (0.3 g, 0.6 mmol), compound 4 (0.38 g, 0.6 mmol) and Pd(PPh$_3$)$_4$ (20 mg, 0.025 mmol) in toluene (5 mL) was stirred at 95°C. To this flask, aqueous 2M Na$_2$CO$_3$ (0.5 mL) was added and the mixture was stirred for 48 h at 100°C. The polymer was end-capped by adding a small amount of bromobenzene to remove the boronic ester end group, followed by stirring for 2 h. The polymerization solution was poured into methanol (500 mL) and the crude polymer was successively Soxhlet extracted with methanol, isopropyl alcohol and hexane to remove the unreacted monomers, impurities and oligomers. The collected powder was dried under vacuum overnight at 40°C to afford **PCz-*co*-P2C$_{10}$BT** (70 mg, 37%) as a dark purple powder. ^{1}H-NMR (CDCl$_3$, δ ppm): 8.0–7.4 (br, 6H, aromatic protons), 4.8–4.5 (br, 4H, methoxy protons next to oxygen), 4.5–4.2 (br, 2H, methoxy protons next to nitrogen), 3.6–3.2 (br, 4H, methylene protons next to oxygen), 1.8–0.6 (br, 47H, aliphatic protons). Anal. Calcd for $(C_{46}H_{67}N_3O_2S)_n$: C, 79.58; H, 9.26; N, 6.33; S, 4.83 Found : C, 73.35; H, 5.40; N, 8.65: S, 3.72.

Fabrication of PLED and Measurements

Optical thin film of the **PCz-*co*-P2C$_{10}$BT** was obtained by spin-coating from their toluene solutions. Polymer solution was filtered with 0.45 μm PP syringe filter (Whatman) prior to spin-coating. Film for optical absorption and PL measurement was spin-coated onto quartz substrates and dried over 10 min at 120°C under N$_2$ atmosphere. Glass substrate coated with ITO, whose conductivity was 15 Ω/cm^2 (Samsung Corning) served as anode electrode. The ITO substrates were cleaned successive rinsing with acetone and wiping with non-woven tissues to remove adhering glass particle from cutting and ultrasonic treatment with distilled water, acetone, isopropyl alcohol, and finally distilled water and dried with N$_2$ gas. The PEDOT/PSS (Baytron CH8000, 30 nm) as a hole injection and decreasing roughness of ITO surface was spin-coated from aqueous solution with 10 wt% of isopropyl alcohol. A solution of **PCz-*co*-P2C$_{10}$BT** (0.5–1.5 wt% in toluene) was filtered through 0.45 μm PP syringe filter and deposited by spin coating onto the PEDOT/PSS layer with uniform and pinhole free films as a thickness around 100 nm. Finally, cathode electrode, Alq$_3$ (15 nm)/LiF (1 nm)/Al (200 nm) triple layer, was vacuum (1×10^{-6} torr) evaporated onto the resulting emitting layer. The active area of the PLED was approximately 4 mm^2. The thickness of the active layer was measured using a KLA Tencor Alpha-step IQ surface profilometer. For the measurements of device characteristics, current density-voltage-luminescence (*J-V-L*) characteristics were measured using a current/voltage source (Keithly 238) and an optical power meter (CS-1000, LS-100). All processes and measurements mentioned above were carried out in the open air at room temperature.

Results and Discussion

Carbazole-based polymers have many advantages such as good hole injection and transporting properties, thermal stability and tuning the emission colors. Its unique chemical properties are effectively raise the highest occupied molecular orbital (HOMO) energy level and reduce the barrier height for hole injection between the anode layer and the emitting



Scheme 1. Synthetic process of the PCz-*co*-P2C_{10}BT

layer in PLEDs. Scheme 1 outlines the synthetic routes of the monomer and the **PCz-*co*-P2C_{10}BT**. Compound (2) was prepared from bromination of carbazole, followed by alkylation. Finally we introduced boronic acid at the position of 3- and 6- in compound (3) for Suzuki polymerization by treating *n*-BuLi. 5,6-Bis(3,7-dimethyloctyloxymethyl)-4,7-dibromo-2,1,3-benzothiadiazole (4) was synthesized according to the published procedures [9]. The polymerization was carried out using palladium(II) acetate, tricyclohexylphosphine, tetraethylammonium hydroxide aqueous solution (20%w/w) in dried toluene under

N_2 atmosphere for 48–72 h. During the polymerization, the fluorescent light was observed without any precipitation. The **PCz-*co*-P2C$_{10}$BT** was completely soluble in various organic solvents such as chloroform, chlorobenzene, toluene, and THF. Molecular structures of the monomer and the **PCz-*co*-P2C$_{10}$BT** were identified by ^{1}H- and ^{13}C-NMR and elemental analysis. To improve color purity and PLED performance, the **PCz-*co*-P2C$_{10}$BT** was further purified by multiple soxhlet extraction with different solvents including methanol, acetone and finally chloroform to remove unreacted monomers and inorganic impurities. The solution of **PCz-*co*-P2C$_{10}$BT** was filtered through the methanol precipitation and then dried in vacuo. From these processes, the highly purified and narrow polydispersity of **PCz-*co*-P2C$_{10}$BT** was obtained. The weight-average molecular weight (Mw) and the polydispersity of the **PCz-*co*-P2C$_{10}$BT** were 5,570 and 1.21, respectively. The glass transition temperature of **PCz-*co*-P2C$_{10}$BT** was 105°C. TGA thermogram was measured at temperature of 5% weight loss for **PCz-*co*-P2C$_{10}$BT** and showed the stable up to 279°C. Higher thermal stability of the light emitting polymer is one of the most critical requirements to operate the PLED especially for blue light emitting it needs higher turn on voltage compared to other color light emitting polymers.

The absorption and PL spectra of the **PCz-*co*-P2C$_{10}$BT** in $CHCl_3$ solution and the polymer film spin-coated onto quartz with $CHCl_3$ solution were measured at room temperature. Figure 1 shows the optical absorption, PL spectrum of **PCz-*co*-P2C$_{10}$BT** in solution state and EL spectra in film state. The **PCz-*co*-P2C$_{10}$BT** exhibited two absorption peaks at 287 nm and 419 nm which can be attributed to the carbazole and 2,1,3-benzothiadiazole segments in the polymer. The absorption and emission peaks in film state were slightly red shifted comparing with the solution state because of the interchain aggregation. The absorption, emission maxima and energy band gap of **PCz-*co*-P2C$_{10}$BT** in solution and film states are summarized in Table 1.

To investigate the charge injection and transport in **PCz-*co*-P2C$_{10}$BT**, it is very important to determine the energy band diagrams of the HOMO and LUMO binding energy levels with respect to the ferrocene standard by using cyclic voltammetry (CV) measurements and the band gaps obtained from the optical absorption spectrum. The ionization potential

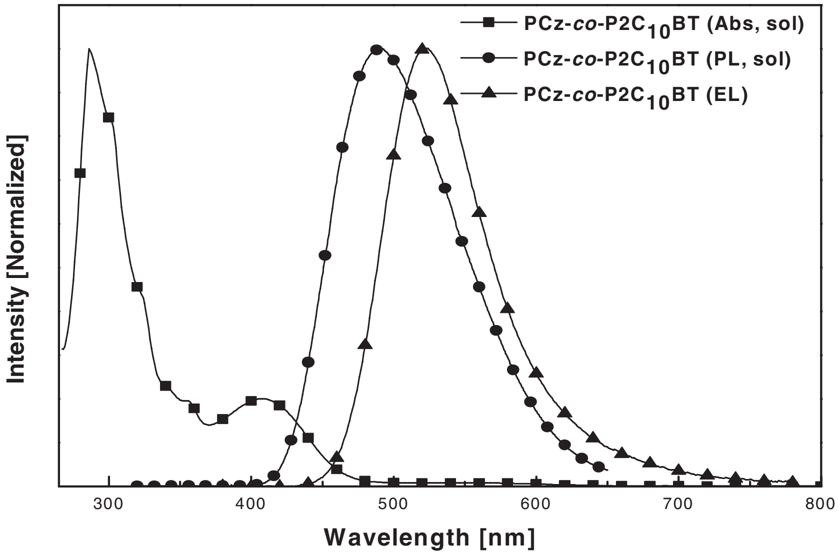


Figure 1. UV-visible absorption, PL and EL spectra of **PCz-*co*-P2C$_{10}$BT**.

Table 1. Electro-Optical properties of **PCz-*co*-P2C$_{10}$BT**.

	Abs (nm)[a]		PL (nm)[a]			
Polymer	Solution	Film	Solution	Film	EL(nm)[b]	Eg(eV)[c]
PCz-*co*-P2C$_{10}$BT	407	419	489	515	522	2.64

[a] Measured in the thin film onto the quartz substrate.
[b] EL spectras were measured cathode using by LiF/Al as cathode.
[c] Band gap estimated from the onset wavelength of the optical absorption.

(IP) and electron affinity (EA) or their associated HOMO and LUMO energy levels were estimated from the onset oxidation data and optical band edges. IP/HOMO = E_{ox}+4.8 eV and EA/LUMO = E_{HOMO}+E_g, where an SCE energy level of -4.8 eV below the vacuum level was used [10]. The HOMO energy level and optical band gap of **PCz-*co*-P2C$_{10}$BT** were 5.55 eV and 2.63 eV, respectively. The major carriers in **PCz-*co*-P2C$_{10}$BT** are holes rather than electrons, due to the lower band offset between the ITO and HOMO energy levels. The band offset was 0.80 eV for hole injection at the interface of the ITO/HOMO state.

To investigate the PLED performance of **PCz-*co*-P2C$_{10}$BT** as an emitting layer, we fabricated PLED with a configuration of ITO/PEDOT/**PCz-*co*-P2C$_{10}$BT**/Alq$_3$/LiF (1 nm)/Al (120 nm). The maximum EL peak of **PCz-*co*-P2C$_{10}$BT** was slightly red-shifted with respect to the PL peak of film state as shown in Figure 1. The red-shifted EL spectrum was attributed to the injected current density, which extended the effective conjugation length. The maximum emission peak of **PCz-*co*-P2C$_{10}$BT** was at 523 nm, which corresponded to a green color.

Figure 2 shows the current density-voltage-luminescence characteristics of the ITO/PEDOT/**PCz-*co*-P2C$_{10}$BT**/Alq$_3$/LiF (1 nm)/Al (120 nm) device. The turn-on voltage of **PCz-*co*-P2C$_{10}$BT** was 10 V. The PLED performances of **PCz-*co*-P2C$_{10}$BT** are

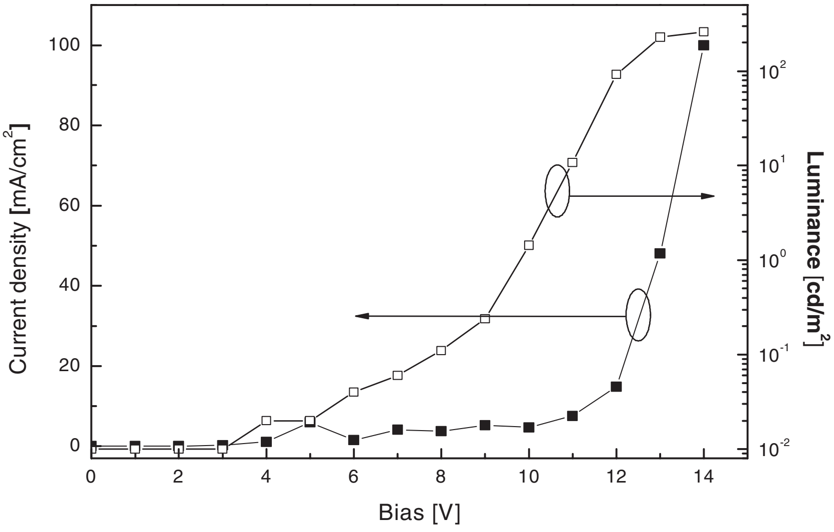


Figure 2. Current density-Luminescence-voltage (*J-V-L*) characteristic of **PCz-*co*-P2C$_{10}$BT**.

Table 2. PLED Performance of PCz-*co*-P2C_{10}BT.

Polymer	Turn-on (V)	PE[a] (lm/W)	Lmax[b] (cd/m^2)	EQE (%)
PCz-*co*-P2C_{10}BT	10	0.18	260	0.22

[a]Maximum power efficiency.
[b]Maximum luminescence.

summarized in Table 2. The maximum brightness, power efficiency and external quantum efficiency of **PCz-*co*-P2C_{10}BT** were 260 cd/m^2, 0.18 lm/W and 0.22%, respectively.

Conclusions

This study focused on the molecular design and synthesis of bipolar structure of poly[3,7-(N-Hexylcarbazole)-*co*-4,7-{5,6-bis(3,7-dimethyloctyloxymethyl)-2,1,3-(benzothiadiazole)}] (**PCz-*co*-P2C_{10}BT**) for PLED applications. The resulting EL polymer was highly soluble in common organic solvents, which allowed being easily spin-coated onto glass substrates with high quality optical thin films. We fabricated PLEDs in ITO/PEDOT/**PCz-*co*-P2C_{10}BT**/Alq$_3$/LiF (1 nm)/Al (120 nm) configuration. The maximum brightness and power efficiency were 260 cd/m^2 at 14 V and 0.18 lm/W at 11 V, respectively.

Acknowledgements

This work was supported by Basic Science Research Program through the National Research Foundation of Korea (NRF) grant funded from the Ministry of Education, Science and Technology (MEST) of Korea for the Center for Next Generation Dye-sensitized Solar Cells (No. 2011–0001055) and the New & Renewable Energy program of the Korea Institute of Energy Technology Evaluation and Planning (KETEP) grant (No. 20103020010050) funded by the Ministry of Knowledge Economy, Republic of Korea.

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