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Synthesis and Light-Emitting Properties of a New Conjugated Polymer Containing Carbazole and Quinoxaline Moieties

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ABSTRACT

A conjugated polymer consisting of carbazole and quinoxaline units (**5**) is synthesized in a high isolation yield (95%). The polymer possesses a high molecular weight (~22 kDa) and is completely soluble in common solvents. When **5** is used as an emitting layer in a multilayer electroluminescence (EL) device, it emits a blue light of 484 nm and exhibits a current efficiency of ~0.7 cd/A.

Key Words: Conjugated polymer; Photo- and electroluminescence; Light-emitting diodes.

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INTRODUCTION

Significant progress in the development of polymer light-emitting diodes (PLEDs) has been made since the discovery of electroluminescence (EL) in a conjugated polymer, poly(*p*-phenylenevinylene).[1,2] The PLEDs possess some important advantages over their low molecular weight counterparts, i.e., organic light-emitting diodes (OLEDs). They are flexible and can be easily processed into a large-area, robust thin films by spin-coating, screen-printing, or ink-jet printing techniques.[3] They can emit different colors once their molecular structures are varied.[4] To achieve full-color display, polymers capable of emitting red, green, and blue lights need to be developed. Synthesis of blue light-emitting polymer is of particular interest because blue can be readily converted to green or red or any other color by a "color converter."[5]

In our previous work, we studied light-emitting properties of a series of disubstituted polyacetylenes and found that most of them emitted strong blue EL.[6–9] In this work, we synthesized a new conjugated polymer (**5**; Sch. 1), which emitted a blue light of 484 nm when used as an active layer in a multilayer EL device with configuration shown in Chart 1.

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RESULTS AND DISCUSSION

Polymer Synthesis

The synthetic route to polymer **5** is shown in Sch. 1.

The *bis*(phenylglyoxalyl)carbazole derivative **4** was synthesized in four steps. First, carbazole was brominated at room temperature in dichloromethane by using appropriate quantity of *N*-bromosuccinimide (NBS) in the presence of silica gel to give 3,6-dibromocarbazole **1** in 72% yield.[10] Then, alkylation using sodium hydride in dry DMF and 2-ethylhexylbromide at 60°C afforded 3,6-dibromo-9-(2-ethylhexyl)carbazole **2** in 76% yield.[11] The symmetrical acetylenic carbazole (**3**) was synthesized via Sonogashira cross-coupling reaction between **2** and phenylacetylene in 83% yield[12] and **3** was finally converted to the corresponding *bis*(phenylglyoxalyl) derivative (**4**) by oxidation with iodine in DMSO at 155°C in 80% yield.[13]

Polycondensation between **4** and 3,3′-diaminobenzidine produced a highly fluorescent and conjugated polymer, poly[*N*-(2-ethylhexyl)carbazole-3,6-diyl-*bis*-quinoxaline] (**5**). The reaction was carried out in *m*-cresol at room temperature for 48 hr, giving **5** in high yield (95%). The polymer was completely soluble in common organic solvents such as chloroform and THF. The purified polymer product was characterized spectroscopically and satisfactory analysis corresponding to its expected molecular structure was obtained (see "Experimental" section for details). The number-average molecular weight of **5** was ~22,000 Da and its polydispersity index was ~1.7, as estimated by a GPC analysis calibrated with polystyrene standards in THF.

Light-Emitting Properties

The photoluminescence (PL) spectrum of a thin solid film of **5** and the EL spectrum of an LED device of **5** are shown in Fig. 1. The device structure is given in

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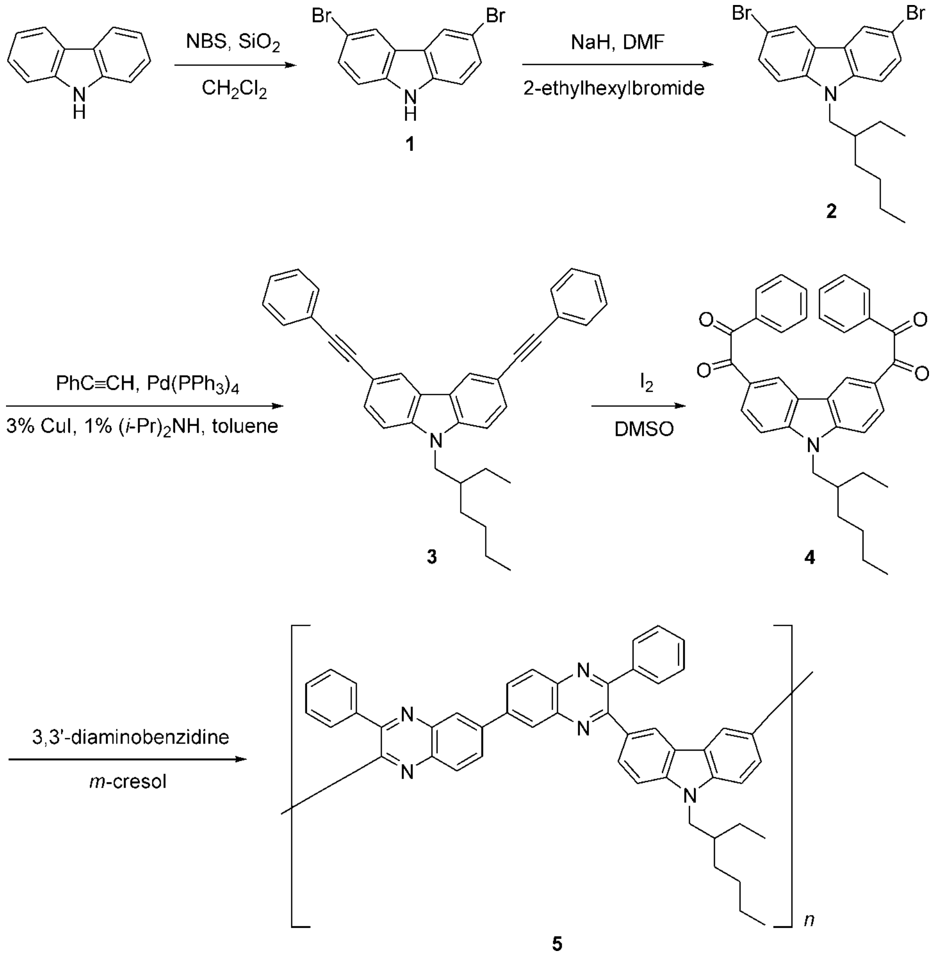

Scheme 1. Synthetic route to the new polymer (**5**).

Chart 1. Both the PL and EL spectra peaked at 484 nm, indicating that the blue light is truly emitted from **5** and that the PL and EL originate from the same singlet excitons induced by photoexcitation or charge recombination. When the voltage applied to the EL device was increased from 16 to 28 V, the emission peak experienced no red-shift, suggesting that the well-separated polymer chains have prevented the aggregation from occurring.

The current density–voltage and brightness–voltage characteristics of the EL device are shown in Fig. 2. Both brightness and current densities are low before the device is turned on at 16 V. Afterwards, they increase exponentially with voltage. The plots of current density–voltage and brightness–voltage are almost superposed in profile, suggesting that the charge transport is balanced. It can be seen from Fig. 2 that a maximum brightness of 380 cd/m^2 is achieved at 28 V and 754 A/m^2, which is higher than the brightness (314 cd/m^2) of our carbazole-containing polyacetylene.[8]

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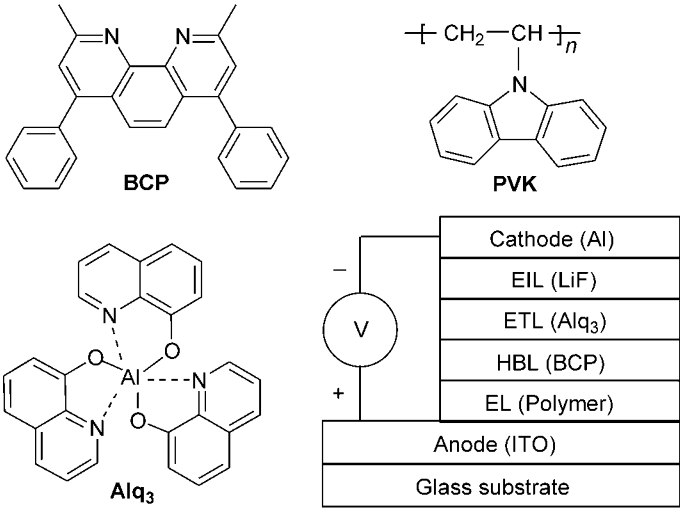

Chart 1. Configuration of a multilayer EL device of polymer **5** (see "Experimental" section for the definitions of the abbreviations).

Figure 3 shows the changes of current and power efficiencies with the applied voltage in the EL device. The current efficiency of **5** initially increased with voltage and reached a maximum value of 0.69 cd/A at 22 V. Afterwards it dropped with a further increase in the applied voltage. The effect of the applied voltage on the power efficiency of **5** was similar to that on the current efficiency, and the maximum power efficiency of **5** was 0.10 lm/W at 22 V. The maximum external quantum efficiency of the device was 0.37% (at 22 V

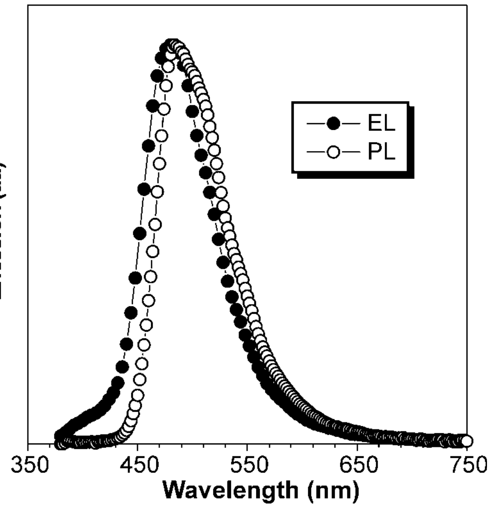

Figure 1. Photo- (PL) and electroluminescence (EL) spectra of polymer **5**. Excitation wavelength for the PL spectrum: 337 nm. EL device configuration: ITO/**5**:PVK (1 : 4 by weight) (46 nm)/BCP (20 nm)/Alq$_3$ (20 nm)/LiF (1 nm)/Al.

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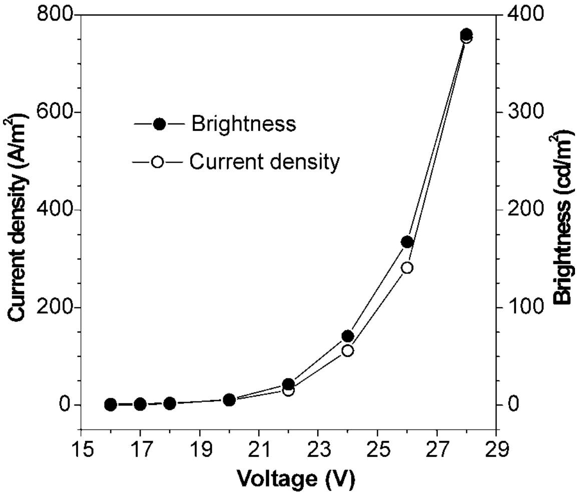

Figure 2. Effect of applied voltage on current density and brightness of the EL device of ITO/**5**:PVK (1 : 4 by weight) (46 nm)/BCP (20 nm)/Alq_3 (20 nm)/LiF (1 nm)/Al.

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under 30.9 A/m^2). The EL efficiency are comparable to those of our carbazole-containing polyacetylene.[8]

EXPERIMENTAL

Materials

Carbazole was purchased from Acros, Geel, Belgium and used as received.

Poly(*N*-vinylcarbazole) (PVK), 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline or bathocuproine (BCP), *tris*(8-hydroxyquinolinato)aluminum(III) (Alq_3), LiF, and Al were all purchased from Aldrich, St. Louis, MO, USA and used without further purification. The molecular structures of these compounds are shown in Chart 1.

Device Fabrication [14–16]

The ITO coated glass substrates were irradiated by ultraviolet (UV) light in a UVO Cleaner (Jelight 144AX-220, Irvine, CA, USA) for 10 min. A 5 mg/mL chloroform solution of a blend of **5**/PVK (1 : 4 by weight) [46 nm; emitting layer (EL)] was spun onto the cleaned ITO glass substrates by a casting equipment (CEE Co., Rolla, MO, USA). The polymer blend layer was allowed to dry for 2 hr at 54°C in a vacuum oven (Shell Lab 1430D, San Juan Capistrano, CA, USA). BCP [20 nm; hole-blocking layer (HBL)], Alq_3 [20 nm; electron-transport layer (ETL)], LiF [1 nm; electron injection layer (EIL)], and Al (150 nm) layers were deposited in turn by a thermal evaporator (Denton Vacuum DV-502A, Moorestown, CA, USA) at a pressure of 10^{-6} mbar and at different evaporation rates. The thickness of each layer was measured in situ by a quartz crystal monitor and checked with a stylus profiler (Tencor P-10, Milpitas, CA, USA).[17,18]

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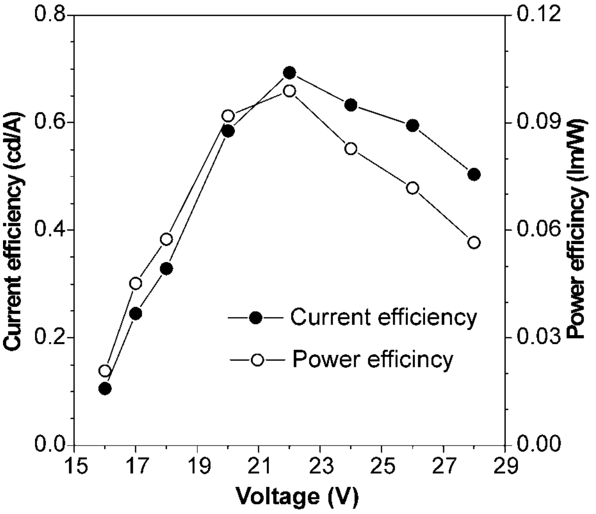

Figure 3. Effect of applied voltage on current efficiency and power efficiency of the EL device of ITO/**1**:PVK (1 : 4 by weight) (46 nm)/BCP (20 nm)/Alq_3 (20 nm)/LiF (1 nm)/Al.

Instrumentation

The current–voltage changes were measured using a current source (Fluke 45; Everett, WA, USA) and a DC voltage source (Advantest R6145; Shinjuku-ku, Tokyo, Japan). The spectra, luminance, and photon density of the light emitting from the EL device were recorded on a photospectrometer (SpectroScan PR650; Chatsworth, CA, USA). The measured area of the EL device was 12.6 mm^2. An He–Ne laser generator (Laser Science VSL-337ND-S, Cambridge, MA, USA) was used as excitation source ($\lambda_{ex} = 337$ nm) for the PL measurements and the PL spectra were recorded on a photospectrometer (SpectroScan PR750; Chatsworth, CA, USA).

Synthesis of 3,6-Dibromocarbazole 1

To a stirred mixture of carbazole (0.835 g, 5 mmol) in dichloromethane (DCM; 100 mL) containing 20 g of silica was added dropwise a solution of NBS (1.78 g, 10 mmol) in DCM (150 mL). The reaction mixture was stirred for 24 hr in the absence of light at room temperature. The reaction mixture was then filtered and the silica was washed with DCM (3 × 30 mL). The combined extracts were washed with water (200 mL) and the organic layer was dried over $MgSO_4$ and evaporated under reduced pressure.

The residue was purified by chromatography over silica-gel with a mixture of hexane–AcOEt (9 : 1) and recrystallized from a mixture of DCM–hexane to afford 1.15 g of a solid in 72% yield; mp 211°C, mp (lit): 212–213°C.[10] ^{1}H NMR (δ, DMSO, 300 MHz): 8.44 (d, $J = 1.8$ Hz, 2H); 7.54 (dd, $J = 1.8, 8.7$ Hz, 2H); 7.48 (d, $J = 8.7$ Hz, 2H); 3.35 (br s, NH). ^{13}C NMR (δ, DMSO, 75 MHz): 139.13, 129.05, 123.71, 123.65, 113.53, 111.32. MS (CI) m/z [assignment, relative intensity (%)]: 324/326/328 (MH, 50/100/51), 245/247 (M − Br, 53/52).

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Synthesis of 3,6-Dibromo-9-(2-ethylhexyl)carbazole 2

To a solution of 3,6-dibromocarbazole (2.93 g, 9 mmol) in dry DMF was added 0.54 g of a 80% dispersion in mineral oil (18 mmol) of sodium hydride. The mixture was heated for 1 hr at 60°C and then cooled to room temperature. 2-Ethylhexyl bromide (4.689 g, 24.3 mmol) was added dropwise. After heating for 14 hr at 60°C, the reaction mixture was poured in ice-water (200 mL) and extracted with DCM (300 mL). The crude product was purified by column chromatography on silica-gel with a mixture of heptane–chloroform (95 : 5) as eluent to give 3 g of a colorless liquid in 76% yield. ^{1}H NMR (δ, $CDCl_3$, 400 MHz): 8.12 (d, $J = 1.65$ Hz, 2H); 7.53 (dd, $J = 1.76$, 8.7 Hz, 2H); 7.23 (d, $J = 8.7$ Hz, 2H); 4.08 (d, $J = 7.72$ Hz, 2H); 1.98 (m, 1H); 1.28 (m, 8H); 0.88 (t, 3H); 0.84 (t, 3H). ^{13}C NMR (δ, $CDCl_3$, 400 MHz): 139.78, 128.98, 123.42, 123.20, 111.94, 110.65, 47.67, 39.32, 30.97, 28.75, 24.36, 22.96, 13.94, 10.84. MS (CI) m/z [assignment, relative intensity (%)]: 436/438/440 (MH, 53/100/49), 357/359 (M − Br, 38/38).

Synthesis of 3,6-*bis*(Phenylethynyl)-9-(2-ethylhexyl)carbazole 3

In a dry flask under argon pressure was placed 3,6-dibromo-9-(2-ethylhexyl)carbazole (2.62 g, 6 mmol), dry diisopropylamine (25 mL), dry toluene (15 mL), $Pd(PPh_3)_4$ (0.416 g, 0.36 mmol), and CuI (0.0457 g, 0.24 mmol). Argon was bubbled through the reaction mixture for 5 min and this mixture was then heated to 60°C. Phenylacetylene (1.224 g, 12 mmol) was added. The reaction was stirred under argon at 60°C for 12 hr. The flask was then allowed to cool to room temperature and ethylacetate (100 mL) was added. The mixture was washed successively with 10% HCl, H_2O, and saturated aqueous NaCl and then dried. The solvent was evaporated and the residue was chromatographed over silica-gel with a mixture of heptane–chloroform (7 : 1) as eluent to yield (2.381 g, 83%) of a slightly yellow solid; mp 106–110°C. ^{1}H NMR (δ, $CDCl_3$, 400 MHz): 8.27 (d, $J = 1.13$ Hz, 2H); 7.63 (dd, $J = 1.52$, 8.7 Hz, 2H); 7.57 (d, $J = 8.7$ Hz, 2H); 7.34 (m, 8H); 4.14 (d, $J = 7.51$ Hz, 2H); 2.04 (m, 1H); 1.27 (m, 8H); 0.91 (t, 3H); 0.84 (t, 3H). ^{13}C NMR (δ, $CDCl_3$, 400 MHz): 140.91, 131.50, 129.67, 128.32, 127.83, 124.11, 123.86, 122.50, 113.90, 109.27, 90.56, 87.85, 47.66, 39.36, 31.00, 28.74, 24.40, 22.97, 13.95, 10.87. MS (CI) m/z [assignment, relative intensity (%)]: 479 (MH, 8), 380 (M − C_7H_{15}, 2).

Synthesis of 3,6-*bis*(Phenylglyoxaloyl)-9-(2-ethylhexyl)carbazole 4

A mixture of 3,6-*bis*(phenylethynyl)-9-(2-ethylhexyl)carbazole (0.479 g, 1 mmol), I_2 (0.254 g, 1 mmol) and DMSO (10 mL) was heated at 155°C for 20 hr. The solution was then poured into 1% aq. $Na_2S_2O_3$ (8 mL). The precipitate was filtered, washed with water, and dried. The residue was chromatographed over silica-gel with a mixture of heptane–chloroform (2 : 8) as eluent to give 0.43 g (80%) of an orange solid in 80% yield; mp 134–136°C. ^{1}H NMR (δ, $CDCl_3$, 400 MHz): 8.27 (d, $J = 1.13$ Hz, 2H); 7.63 (dd, $J = 1.52$, 8.48 Hz, 2H); 7.57 (d, $J = 8.7$ Hz, 2H); 7.34 (m, 8H); 4.14 (d, $J = 7.51$ Hz, 2H); 2.04 (m, 1H); 1.27 (m, 8H); 0.91 (t, 3H); 0.84 (t, 3H). ^{13}C NMR (δ, $CDCl_3$, 400 MHz): 194.93, 193.79, 145.36, 134.78, 133.29, 130.06, 129.00, 128.34, 125.83, 124.35, 123.11,

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110.20, 48.07, 39.44, 30.92, 28.71, 24.33, 22.90, 13.91, 10.80. MS (CI) m/z [assignment, relative intensity (%)]: 544 (MH, 22), 438 (M − PhCO, 10).

Synthesis of Polymer 5

A mixture of 3,6-*bis*(phenylglyoxaloyl)-9-(2-ethylhexyl)carbazole (0.135 g, 0.25 mmol), 3,3′-diaminobenzidine (0.053 g, 0.25 mmol) in *m*-cresol (1 mL) was stirred at room temperature for 24 hr at room temperature. An additional amount of *m*-cresol was then added and the mixture was dropped into methanol in a flask. After filtration, the polymer was dissolved in chloroform and dropped once again in methanol. The last procedure was repeated several times and the polymer was filtered off and dried in air to give 0.162 g (95%) of yellow solid. ^{1}H NMR (δ, $CDCl_3$, 300 MHz): 8.59 (br s, 2H-carbazole); 8.28 (m, 6H-quinoxaline); 7.59 [br s, 2H-carbazole + 4H-phenyl (ortho)]; 7.31 [br s, 2H-carbazole + 6H-phenyl (meta and para)]; 4.13 (br s, 2H); 2.01 (br s, 1H); 1.25 (br s, 8H); 0.82 (br s, 6H). ^{13}C NMR (δ, $CDCl_3$, 75 MHz): 154.3, 153.9, 141.9, 141.4, 14.0, 140.6, 139.1, 129.9, 129.5, 129.0, 128.4, 127.3, 126.9, 123.2, 122.7, 109.0, 47.7, 39.3, 31.0, 28.7, 24.4, 22.9, 14.0, 10.9.

CONCLUSION

A new conjugated polymer (**5**) was synthesized by a condensation polymerization. The PL and EL properties of **5** were studied. The thin solid film of **5** emitted blue PL peaked at 484 nm. The device with a configuration of ITO/**5**:PVK (1 : 4 by weight) (46 nm)/BCP (20 nm)/Alq_3 (20 nm)/LiF (1 nm)/Al emitted blue EL also peaked at 484 nm. The EL device showed a brightness of 380 cd/m^2. The maximum current, power, and external quantum efficiencies of the device were 0.69 cd/A, 0.10 lm/W, and 0.37%, respectively.

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