

Synthesis and Characterization of Novel Poly(arylenevinylene) Derivative

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ABSTRACT: The new poly(arylenevinylene) derivative composed naphthalene phenylene vinylene backbone was developed. The theoretical calculation showed that the model compound of the obtained polymer was highly distorted between the styryl and naphthalene units as well as between the backbone and fluorene side units. The polymer was synthesized by the palladium catalyzed Suzuki coupling reaction with 2,6-(1',2'-ethylborate)-1,5-dihexyloxy-naphthalene and 1,2-bis(4'-bromophenyl)-1-(9'',9'')-dihexyl-3-fluorenyl)ethene. The structure of the polymer was confirmed by ¹H NMR, IR, and elemental analysis. The weight-average molecular weight of the polymer is 29,800 with the

polydispersity index of 1.87. The new polymer showed good thermal stability with high T_g of 195°C. The bright blue fluorescence ($\lambda_{\text{max}} = 475$ nm) was observed both in solution and film of new polymer with naphthalene phenylene vinylene backbone. Double layer LED devices with the configuration of ITO/PEDOT/polymer/LiF/Ca/Al showed a turn-on voltage at around 4.5 V, the maximum luminance of 150 cd/m², and the maximum efficiency of 0.1 cd/A. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 2009–2015, 2008

Key words: poly(arylenevinylene); naphthalene; LED; blue fluorescence

INTRODUCTION

Conjugated polymers as promising materials for optoelectronic applications such as transistors and organic light emitting diodes including flexible displays have been attracted.^{1–14} In the field of polymer-based electroluminescent devices, much interest has been paid to the main-chain conducting polymers such as poly(phenylenevinylene) (PPV), poly(*p*-phenylene) (PPP), poly(thiophene), poly(fluorene), their copolymers and soluble derivatives because of the potential application as large band gaps, especially poly(fluorene) (PFs), have acquired much interest because they emit blue light and also enable full color via energy transfer to longer wavelength emitters.^{15–24}

One of the major problems in obtaining blue EL from PFs is the change of the emission spectra that becomes more serious upon exposure to heat, resulting in the declining color stability of the light emission from LEDs fabricated with PFs. These changes of emission spectra are attributable to the interchain interactions.^{25–27}

Recently, we reported the poly(biphenylenevinylene) derivatives and poly(terphenylenevinylene) derivatives for pure blue OLED.^{28–36} In connection with the recent report, we designed and synthesized the new poly(arylenevinylene) derivative which is composed naphthalene phenylene vinylene for blue light emitting. The high efficient blue emission can be expected by the introduction of rigid fused aromatic naphthalene unit having extended π -system when compared with phenylene.^{37,38}

On the basis of arylenevinylene backbone, the large fluorene substituents were introduced into the each vinyl bridge. The introduced bulky fluorene pendants can lead to enhance the solubility of the resulting polymer, shorten the effective conjugation length that results in pure blue emission, and inhibited the formation of excimers that results in efficient pure blue emission. It is also expected that the high efficient blue emission is due to the intramolecular energy transfer from fluorene pendant to main chain.

EXPERIMENTAL SECTION

General

¹H NMR spectra data were expressed in ppm relative to the internal standard and were obtained on a DRX 500 MHz NMR spectrometer. FTIR spectra

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were obtained with a Bomem Michelson series FTIR spectrometer, and the UV-vis absorption spectra obtained in chloroform on a Shimadzu UV 3100 spectrometer. 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 -styragel columns of 105, 104, 103, 500, and 100 Å, refractive index detectors, solvent THF). Elemental analysis was 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 $10^\circ C/min$. The photoluminescence spectra were recorded on a Perkin-Elmer LS-50 fluoremeter utilizing a lock-in power meter (Newport 818-SL). Cyclic voltammetry was carried out in a two-compartment cell with a model with platinum electrodes at a scan rate of 10 mV/s against an Ag/Ag^+ ($0.1M\text{ AgNO}_3$ in acetonitrile) reference electrode in an anhydrous and nitrogen-saturated solution of $0.1M\text{ Bu}_4\text{NBF}_4$ in acetonitrile. 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 Alpha step IQ.

Fabrication of the LED

Poly(styrenesulfonate)-doped poly(3,4-ethylenedioxythiophene) (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 was spin-coated (3000 rpm , 50 s) from a filtered (0.2 m filter) $1.0\text{ wt}\%$ of polymer solution in chlorobenzene on a PEDOT layer. A thin layer of LiF (0.5 nm) and subsequently a 10 nm layer of Ca and a 150 nm layer of Al were evaporated subsequently on the top of the emissive layer under a high vacuum (below $1 \times 10^{-5}\text{ Torr}$). The active area of the device was 4 mm^2 . All fabrication steps were performed in clean room conditions. Measurements were done at room temperature in air.

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.

SYNTHESIS

Synthesis of the 2,6-dibromo-1,5-dihydroxy naphthalene ($C_{10}H_6Br_2O_2$)

The 1,5-dihydroxy naphthalene (20 g , 0.125 mol) and catalytic amount of iodine were added in the acetic acid solvent (550 mL). After the mixture was heated to $80^\circ C$, bromine (40 g , 0.25 mol) was slowly added. After the mixture was stirred for 30 min , the flask was cooled to room temperature. The crude product was filtered and washed with the petroleum ether. Yield: 57% . $^1H\text{ NMR}$ (500 MHz , $CDCl_3$) [ppm] δ 7.75 (d, 2H), 7.64 (d, 2H), 6.0 (s, 2H) FTIR (KBr, cm^{-1}): 2950 (aliphatic C-H str), 3050 (aromatic C-H str), 1180 (aromatic C-Br str).

Synthesis of the 2,6-dibromo-1,5-dihexyloxy naphthalene ($C_{22}H_{30}Br_2O_2$)

The 2,6-dibromo-1,5-dihydroxynaphthalene (5 g , 15.7 mmol), 1-bromohexane (10.38 g , 63 mmol), KOH and NaI (0.94 g , 63 mmol) were mixed in ethanol (200 mL) and refluxed for 48 h . After reflux, the flask was cooled to the room temperature, and the ethanol was removed and extracted with ether. Then the mixture was washed with 10% NaOH aqueous solution (2.2 g , 40 mmol) to remove the HBr from the residue, and washed again by the water to remove the salt. The mixture was stirred with $MgSO_4$ for drying, and filtered. The crude product was purified by column chromatography by hexane as eluent. Yield: 25% , $^1H\text{ NMR}$ (500 MHz , $CDCl_3$) [ppm] δ 7.75 (m, 2H), 7.64 (m, 2H), 4.09 (m, 4H) FTIR (KBr, cm^{-1}): 2950 (aliphatic C-H str), 3050 (aromatic C-H str), 1180 (aromatic C-Br str) M^+ : 486, Anal. Calcd for $C_{22}H_{30}Br_2O_2$: C, 54.32% ; H, 6.17% . Found: C, 54.27% ; H, 6.11% .

Synthesis of the 2,6-(1',2'-ethylborate)-1,5-dihexyloxynaphthalene

2,6-Dibromo-1,5-dihexyloxy naphthalene (1.5 g , 3.1 mmol) was dissolved in THF solvent, and then the mixture was stirred at $-78^\circ C$. $n\text{-BuLi}$ (2.052 g , 0.032 mol) was dropped, and stirred at $-78^\circ C$ for 1 h . In the mixture, 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.377 g , 7.4 mmol) was added and stirred them at room temperature for overnight. After the reaction, the mixture was worked up with water to prevent from HCl. Then the obtained crude product was purified by column chromatography using hexane as eluent. Yield: 35% , $^1H\text{ NMR}$ (500 MHz , $CDCl_3$) [ppm] δ 7.94 (m, 2H), 7.76 (m, 2H), 4.08 (m, 4H), 1.41 (s, 19H), FTIR (KBr, cm^{-1}): 3100 (aromatic C-H str), 2900 (aliphatic C-H str).

Synthesis of 4-bromophenyl-9'',9''-dihexyl-3-fluorene ketone

In a mixture of 9,9-dihexylfluorene (I) (5 g, 22.8 mmol), CS₂ (70 mL), and AlCl₃ (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 2N HCl aqueous solution, and then the mixture was extracted with diethyl ether. The extract was washed with 2N KOH aqueous solution and then was washed several times with water. The washed ether solution was dried over MgSO₄ and filtered. The solvent was evaporated, and the crude product was recrystallized in hexane. Yield: 69%; mp: 84°C. ¹H NMR (500 MHz, CD₂Cl₂) [ppm] δ 7.85–7.83 (m, 4H), 7.74–7.69 (m, 2H), 7.45–7.42 (m, 3H), 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) FTIR (KBr, cm⁻¹): 3047 (aromatic C–H str), 2846 (aliphatic C–H str), 1643 (C=O), 1064 (aromatic C–Br).

Synthesis of 1,2-bis(4'-bromophenyl)-1-(9'',9''-dihexyl-3-fluorenyl)ethene

In a two-necked flask equipped with a reflux condenser, a mixture of (4-bromobenzyl)triphenylphosphonium bromide (7.42 g, 14.5 mmol), sodium hydride (1.16 g, 48 mmol), and toluene (70 mL) was refluxed for 8 h. After cooling, 4-bromophenyl-9'',9''-dihexyl-3-fluorene ketone (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 MgSO₄ and filtered. The solvent was removed by evaporation. The residue was dissolved in *n*-hexane and then purified by flash column chromatography by ethyl acetate: hexane (1 : 10) as eluent. After column, recrystallization from ethanol afforded pure product. Yield: 51%, mp: 87°C. ¹H NMR (500 MHz, CD₂Cl₂) [ppm] δ 7.69–7.27 (m, 2H), 7.5 (m, 2H), 7.4–7.3 (m, 7H), 7.15–7.14 (m, 4H), 7.03–6.99 (m, 3H), 1.09–1.06 (m, 8H), 0.84–0.81 (m, 6H), 0.64–0.63 (m, 4H) FTIR (KBr, cm⁻¹): 3008 (aromatic C–H str), 2900 (aliphatic C–H str), 1064 (aromatic C–Br str) M+: 670, Anal. Calcd for C₃₉H₄₂Br₂: C, 69.85%; H, 6.27%. Found: C, 69.81%; H, 6.23%.

Synthesis of polymer

The polymer was prepared from palladium catalyzed Suzuki coupling reaction.

All handling of catalysts and polymerization was done in a nitrogen atmosphere. To a stirred solution of 2,6-(1',2'-ethylborate)-1,5-dihexyloxynaphthalene (0.15 g, 0.3 mmol) 1,2-bis(4'-bromophenyl)-1-(9'',9''-dihexyl-3-fluorenyl)ethene (0.173g, 0.25mmol) in 10 mL THF and 4 mL 2M K₂CO₃ solution in water was added catalysts, Pd(PPh₃)₄ (0.01 g, 0.008 mmol %).

The reaction mixture was heated at 80°C under nitrogen atmosphere for 48 h. Bromobenzene (0.005 g, 0.0318 mmol) was added and then phenyl boronic acid (0.005 g, 0.041 mmol) was added with small amounts of catalysts for end-capping. After 2 h, the reaction mixture was poured into methanol (50 mL) and filtered with glass filter. The residue was dissolved in CHCl₃ and washed with water. After being dried over MgSO₄, precipitation was repeated twice with chloroform/methanol. Yield = 65%. ¹H NMR (500 MHz, CDCl₃) [ppm] 7–8 (m, 19H), 3.64(m, 4H), 0.5–2 (m, 52H), FTIR (KBr, cm⁻¹): 3100 (aromatic C–H str), 2950(aliphatic C–H str).

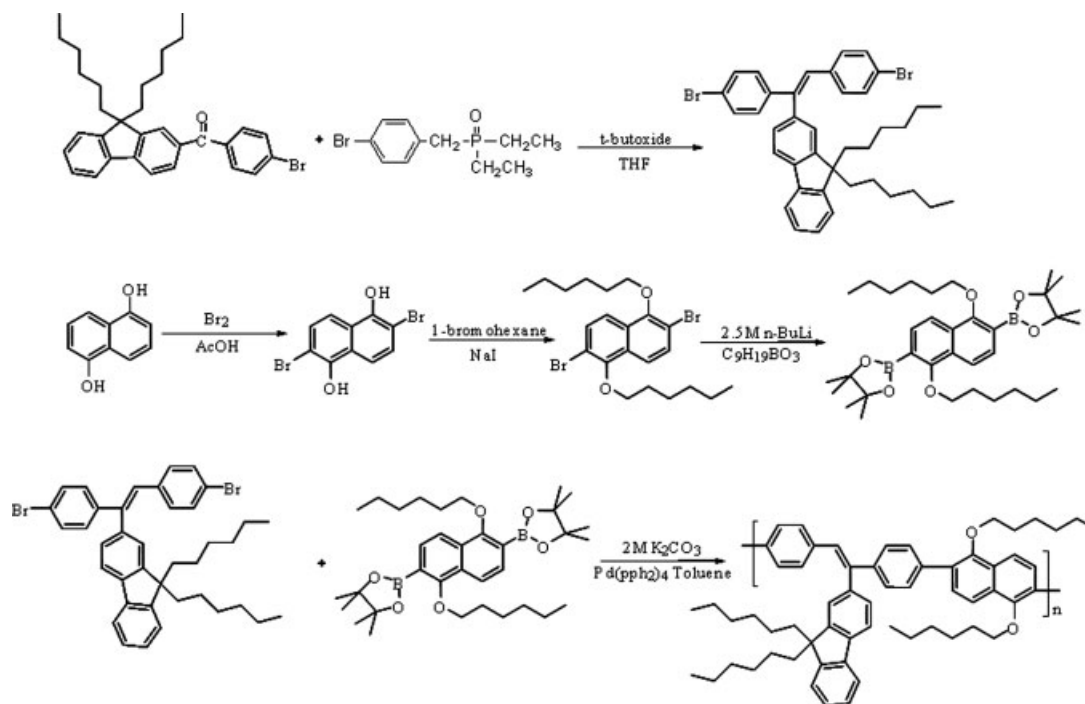
Anal. Calcd for C₆₁H₇₂O₂: C, 87.56%; H, 8.61%. Found: C, 87.12%; H, 8.37%.

RESULTS AND DISCUSSION

The method for preparing this polymer is outlined in Scheme 1. The Friedel-Crafts acylation of 9,9-dihexylfluorene was carried out with 4-bromobenzoyl chloride. The resulting 9',9'-dihexyl-3-fluorenyl-4'-bromomethyl ketone, which was removed after recrystallization in good yield, was converted to monomer 1 by the Wittig reaction. The alkylation was followed by bromination of 1,5-dihydroxy naphthalene afforded to 2,6-dibromo-1,5-dihexyloxy naphthalene. The polymerization was carried out by the palladium catalyzed Suzuki coupling reaction with 2,6-(1',2'-ethylborate)-1,5-dihexyloxynaphthalene and 1,2-bis(4'-bromophenyl)-1-(9'',9''-dihexyl-3-fluorenyl)ethene.

Theoretical calculations using PM3 parameterization in the HyperChem 5.0 program (Hypercube), to fully optimize the molecular structure, was carried out for the characterization of three-dimensional structures.³⁹ Figure 1 shows the stereostructure of the model compound derived from calculative analyses. From the result, it can be suggested that the obtained polymer is highly distorted between the styryl and naphthalene units as well as between the backbone and fluorene side units.

The structure of the obtained polymer was confirmed by ¹H NMR, IR, and elemental analysis. The Figure 2 shows the ¹H NMR and IR spectra of polymer. The polymer composition can be estimated by the integration area of the peaks from the ¹H NMR. The peak at 3.6 ppm is attributed to the protons connected to oxygen in the naphthalene unit. The peak at 2.0 ppm is attributed to the protons adjacent to the ninth position of fluorene. The obtained polymer was fully soluble in common organic solvents such as chloroform, chlorobenzene, toluene, and tetrahydrofuran. The molecular weight has been determined by gel permeation chromatography with THF as an eluent and monodisperse polystyrene as the



Scheme 1 Synthetic scheme of monomers and polymer.

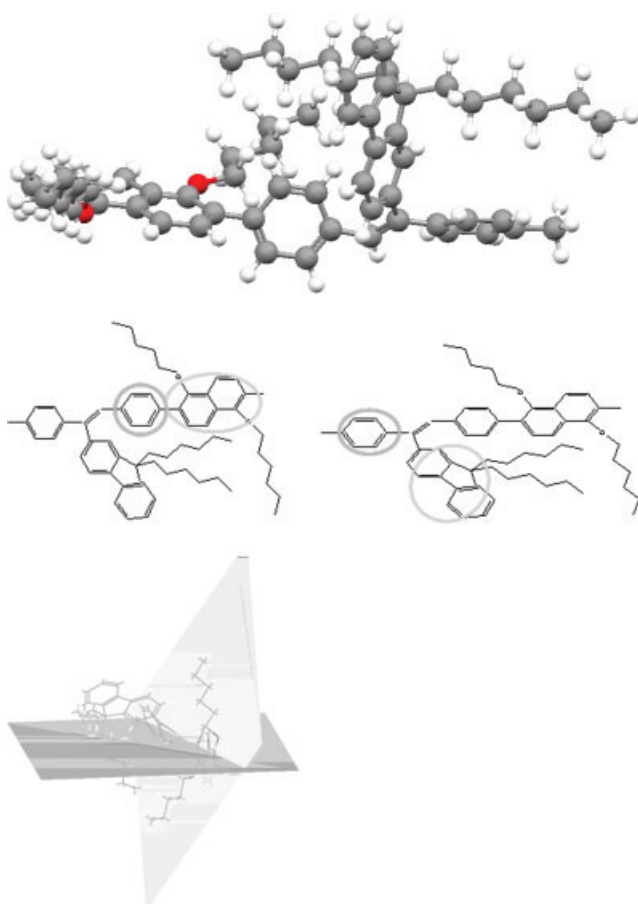


Figure 1 Synthetic scheme of monomer and polymer. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

standard. The weight average molecular weight of polymer is 29,800 and the polydispersity index is 1.87. The thermal stability of polymer under nitrogen atmosphere has been evaluated by thermogravimetric analysis, and the decomposition temperature is 439°C, as determined at a 5 wt % loss (Fig. 2). The polymer displays a glass transition temperature (T_g) at 195°C, which means high thermal stable amorphous nature of material, in the differential scanning calorimetry thermogram (Fig. 3).

Figure 4 shows the optical absorption and photoluminescence spectra of polymer in both solution and solid. The polymer in a chloroform solution exhibited the maximum absorption at 368 nm because of the $\Pi \rightarrow \Pi^*$ transition of the conjugated polymer backbone with a shoulder at 310 nm, which is a characteristic peak of fluorene unit. The absorption of thin film also shows its maximum at 375 nm with the absorption edge at 424 nm.

The maximum absorption peak of the polymer having naphthalene phenylene vinylene backbone is 60–70 nm blue shifted when compared with that of PPV, 20–30 nm red shifted as compared with that of PPP, and 5 nm red shifted as compared with biphenylenevinylene units. Under excitation of 365 nm, bright blue fluorescence was observed both in solution and film of new polymer with naphthalene phenylene vinylene backbone. Moreover, the PL spectrum of the film was almost consistent with that of dilute solution with the maximum emission peak of 475 nm, indicating that the intermolecular interaction is inhibited due to distorted backbone and steric

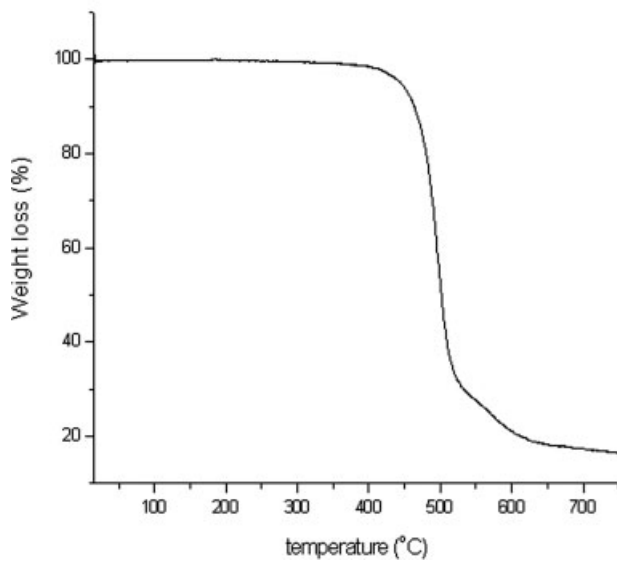
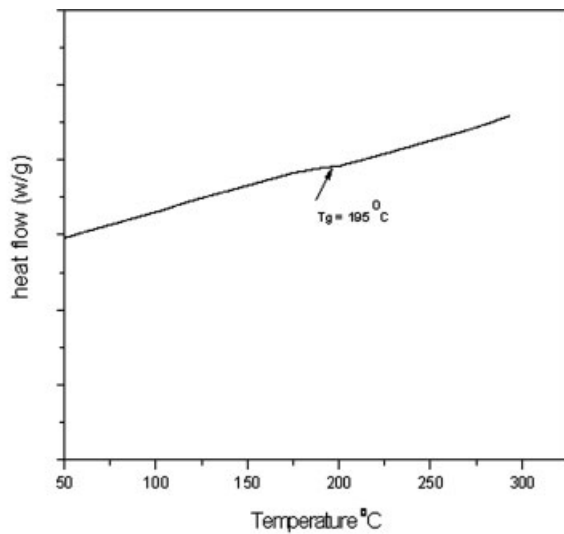


Figure 3 TGA and DSC thermogram of polymer.

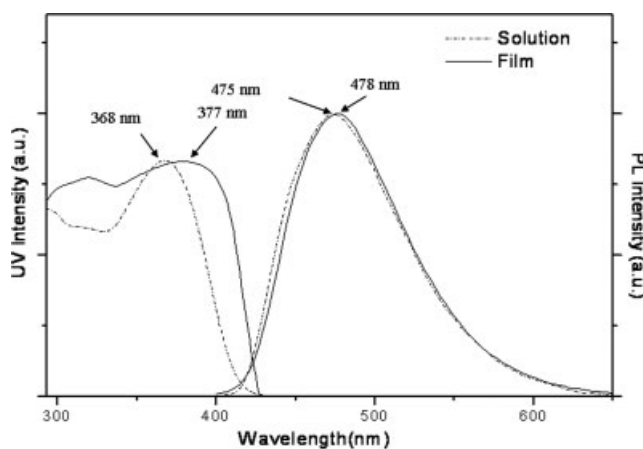


Figure 4 The optical absorption and photoluminescence spectra of polymer in both solution and solid.

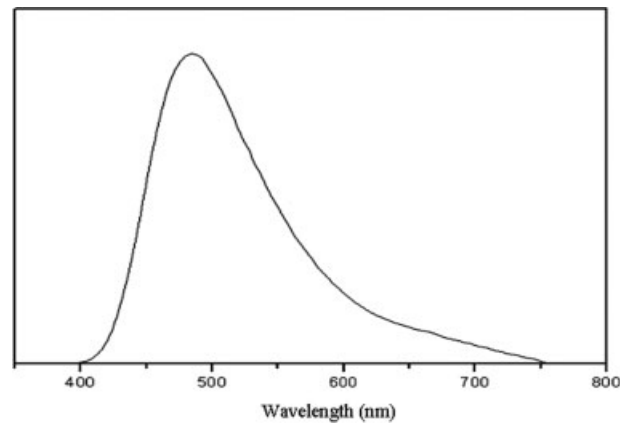


Figure 5 EL spectrum of ITO/PEDOT/polymer/LiF/Ca/Al structured device.

charge transport ability such as poly(9-vinylcarbazole) will be necessary.

CONCLUSIONS

We designed and synthesized the new poly(arylene-vinylene) derivative which is composed naphthalene phenylene vinylene for blue light emitting. The theoretical calculation of model compound of obtained

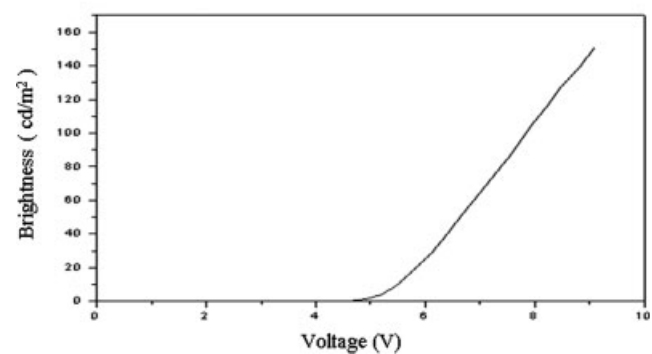
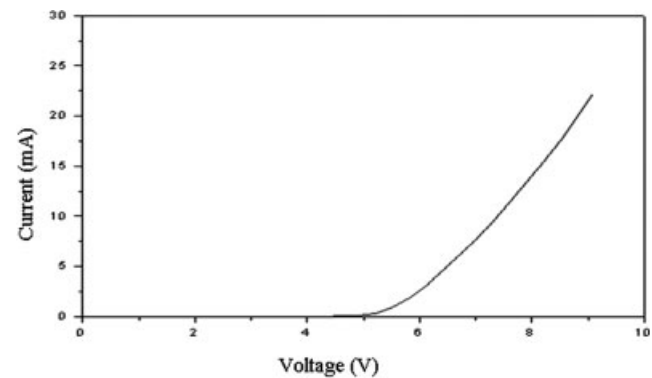


Figure 6 Current-voltage-luminescence (I-V-L) and current efficiency characteristic of ITO/PEDOT/polymer/LiF/Ca/Al structured device.

polymer showed highly distorted structure. The structure of the polymer which was obtained by palladium catalyzed Suzuki coupling reaction with 2,6-(1',2'-ethylborate)-1,5-dihexyloxynaphtalene and 1,2-bis(4'-bromophenyl)-1-(9'',9''-dihexyl-3-fluorenyl)ethane, was confirmed by ¹H NMR, IR, and elemental analysis. The new polymer with naphthalene phenylene vinylene backbone and fluorene pendant showed maximum emission at 475 nm both in solution and thin film, indicating that the intermolecular interaction is inhibited due to distorted backbone and steric hindrance by introduced bulky 9,9-dihexylfluorene substituents. Double layer LED devices with the configuration of ITO/PEDOT/polymer/LiF/Ca/Al showed a turn-on voltage at around 4.5 V, the maximum luminance of 150 cd/m² and the maximum current efficiency of 0.1 cd/A.

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