Intense green light from a silyl-substituted poly(*p*-phenylenevinylene)-based light-emitting diode with air-stable cathode

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A silicon-containing poly(p-phenylenevinylene) derivative, poly[2,5-bis(butyldimethylsilyl)-1,4phenylenevinylene] (BS-PPV), was synthesized *via* the Gilch reaction. The polymer is fully solution processable with high thermal stability. The UV-Vis absorption and fluorescent emission spectra demonstrate that BS-PPV is a promising green emissive material for light-emitting device application. Cyclic voltammetric measurements indicate that it can be reversibly n-doped and irreversibly p-doped with the onset oxidation and reduction potentials of 1.16 and -1.81 V, respectively. The HOMO and LUMO energy levels of BS-PPV were estimated to be 5.56 and 2.59 eV, respectively. Single layer devices with the configuration ITO/BS-PPV/Al were fabricated, which showed a turn-on voltage of 6 V and intense green light was observed at around 7.5 V. The performance is better than that of devices fabricated with other silicon-containing PPV-based polymers.

Introduction

Enormous efforts have been made to develop conjugated electroluminescent polymers for application in light-emitting diodes (LEDs) in the past decade.¹⁻¹² Polymer LEDs (PLEDs) have demonstrated some attractive advantages such as low cost processing through simple spin-coating or casting techniques, color tuning by way of chemical modification of the active polymers and ease of realization of larger area flat panel displays over traditional inorganic LEDs. It is recognized that good processability, high photoluminescent quantum efficiency and high thermal and optical stability are basic prerequisites for electroluminescent polymers to be commercialized for LED displays. Poly(p-phenylene vinylene) (PPV) and its derivatives are typical examples and are the most extensively investigated materials for PLEDs. Among PPV derivatives, increasing attention is being paid to silylsubstituted PPVs since the first silicon-containing PPV was reported by Wudl and co-workers in 1994.13,14 Silvlsubstituted PPVs are attractive owing to their extremely high photoluminescent (PL) quantum efficiencies compared with PPV itself or alkoxy-substituted PPVs. Very high PL efficiency (60% or above for thin film samples) and EL efficiency based on silyl-substituted PPVs have been achieved.^{15,16} The high EL performance is attributed to a good balance for hole and electron injections into the emissive material and the low barrier energies between the polymer film and the electrodes.¹⁵ In addition, silicon-containing PPVs have demonstrated very good solubility and uniform film morphology without any crystalline features in the film state.¹⁵

In our previous work, a silicon-containing PPV-based copolymer with an alternating structure showed that the PL quantum efficiency could be as high as 65%.¹⁷ In this paper we report a facile way for the synthesis of a new PPV derivative, poly[2,5-bis(butyldimethylsilyl)-1,4-phenylenevinylene] (BS-PPV), through introducing two silyl groups as the substituents on the phenylene ring, aiming at developing a more

readily processable material with high PL and EL quantum efficiencies. Single layer devices using BS-PPV as the emissive layer and air stable Al as the cathode with the configuration of ITO/BS-PPV/Al were fabricated. The characteristics of the single layer devices will be discussed.

Experimental

Measurements

NMR spectra were collected on a Bruker ACF 300 spectrometer with chloroform-d as solvent and tetramethylsilane as internal standard. FT-IR spectra were recorded on a Bio-Rad FTS 165 spectrometer by dispersing samples in KBr disks. UV-Vis and fluorescence spectra were obtained on a Shimadzu UV-NIR 3100 spectrophotometer and on a Perkin-Elmer LS 50B luminescence spectrometer, respectively. Thermogravimetric analysis (TGA) was conducted on a DuPont Thermal Analyst 2100 system with a TGA 2950 thermogravimetric analyzer at a heating rate of $20 \,^{\circ}\text{C} \, \text{min}^{-1}$ and an air flow rate of 75 ml min⁻¹. Elemental microanalyses were performed on a Perkin-Elmer 2400 elemental analyzer for C, H, N and S determination. Cyclic voltammetry (CV) was performed on an EG&G Parc Model 273A potentiostat/ galvanostat system with a three-electrode cell in a solution of Bu_4NBF_4 (0.10 M) in acetonitrile at a scan rate of 10 mV s⁻¹. Polymer films were coated on a square Pt electrode (0.50 cm^2) by dipping the electrode into the corresponding solutions and then drying in air. A Pt wire was used as the counter electrode and a Ag/AgNO₃ (0.10 M in acetonitrile) electrode was used as the reference electrode. Prior to each series of measurements the cell was deoxygenated with argon. Gel permeation chromatographic (GPC) analysis was conducted on a Perkin-Elmer Model 200 HPLC system equipped with Phenogel MXL and MXM columns using polystyrene as standard and THF as eluent.

Single layer LED devices were fabricated using an indium tin oxide (ITO)-coated glass substrate with a sheet resistance of about 250 $\Omega \square^{-1}$. The substrate was cleaned by sonicating in hexane and propan-2-ol successively prior to use. A uniform polymer film was obtained by spin coating at room temperature from 10 mg ml⁻¹ of xylene solution at 2000 rev min⁻¹ with thickness of about 1000 Å. The film was dried at room temperature in a vacuum oven for 24 h before the cathode electrode was deposited. An aluminum electrode with thickness of about 1000 Å was evaporated (JEOL-400 vacuum evaporator) through a mask at a pressure of 3×10^{-7} Torr and a rate of 1 Å s⁻¹ (TM-200R thickness monitor, Maxtek). The active area of the LED was approximately 7 mm². The electrical contacts were fixed using a conductive epoxy 14G adhesive. All of the sample processing, handling and opticelectronic measurements were performed under ambient conditions. The determination of current-voltage (I-V)characteristics was carried out using a Keithley 238 High Current Source Measure unit.

Materials

Tetrahydrofuran (THF) was distilled over sodiumbenzophenone. 2,5-Dibromo-*p*-xylene, potassium *tert*butoxide (1.0 M solution in THF), *N*-bromosuccinimide (NBS) and magnesium turnings, were purchased from Aldrich and butyldimethylsilyl chloride from Fluka. All chemicals were used without further purification.

2,5-Bis(butyldimethylsilyl)-p-xylene. The Grignard reagent 2,5-bisbromomagnesium-p-xylene was prepared by refluxing the mixture of 2,5-dibromo-p-xylene (5.28 g, 20 mmol) and magnesium turnings (1.06 g, 44 mmol) in 20 ml of anhydrous THF for 4 h. To this solution, cooled in an ice-bath, was added a solution of butyldimethylsilyl chloride (6.63 g, 44 mmol) in 20 ml of THF. The mixture was refluxed for 24 h and then cooled in an ice-bath. After it had been quenched with saturated ammonium chloride aqueous solution, THF was evaporated and the residue was extracted twice with 30 ml of hexane. The combined organic layer was washed with water, brine and then dried over anhydrous magnesium sulfate. After the solvent had been evaporated under reduced pressure, the residue was purified by silica gel chromatography with hexane as eluent. A 3.54 g amount of colorless liquid was obtained with a yield of 53%. MS, m/z 334. ¹H NMR (CDCl₃, ppm), δ 7.19 (2H, s), 2.42 (6H, s), 1.42–1.27 [8H, m, -(CH₂)₂-], 0.93-0.79 (10H, m, -SiCH₂- and -CH₃), 0.30 [12H, s, $-Si(CH_3)_2$]. Anal. Calcd. for $C_{20}H_{38}Si_2$: C, 71.77; H, 11.44; Found: C, 71.93; H, 11.50%.

2,5-Bis(butyldimethylsilyl)-1,4-bisbromomethylbenzene. A 5.01 g (15.0 mmol) amount of 2,5-bisbutyldimethylsilyl-pxylene, 5.40 g (30.0 mmol) of NBS, a catalytic amount of benzyl peroxide (BPO) and 150 ml of benzene were charged into a flask. The reaction mixture was stirred at ambient temperature and irradiated with a tungsten lamp for 2 h. The solution was then washed with water three times followed by brine. The organic phase was dried over anhydrous magnesium sulfate. After filtration and evaporation of the solvent, the crude product was purified by silica gel chromatography to give 3.01 g of colorless liquid product (41% yield). MS, m/z492. FT-IR (KBr), v_{max}/cm^{-1} 3075, 2949, 2929, 2848, 1469, 1413, 1376, 1343, 1249, 1208, 1191, 1168, 1126, 910, 877, 865, 842, 826, 810, 789, 753, 719, 701, 681, 659, 538, 482, 458, 439. ¹H NMR (CDCl₃, ppm), δ 7.49 (2H, s), 4.58 (4H, s, -CH₂Br), 1.44-1.27 [8H, m, -(CH₂)₂-], 0.92-0.85 (10H, m, -SiCH₂- and -CH₃), 0.39 [12H, s, -Si(CH₃)₂]. ¹³C NMR (CDCl₃, ppm), δ 142.0, 140.0, 137.6, 34.1, 26.4, 26.0, 16.0, 13.7, 1.6. Anal. Calcd. for $C_{20}H_{36}Br_2Si_2$: C, 48.77; H, 7.37; Br, 32.45; Found: C, 48.53; H, 7.42; Br, 32.29%.

Poly[2,5-bis(butyldimethylsilyl)-1,4-phenylenevinylene] (BS-PPV). A solution of 0.984 g (2.0 mmol) of 2,5-bis(butyldimethylsilyl)-1,4-bisbromomethylbenzene in 30 ml of anhydrous THF was charged into a 100 ml flask. To this solution were added dropwise 12 ml of a 1.0 M solution of potassium tert-butoxide (12.0 mmol) in anhydrous THF at room temperature with stirring. The mixture was continuously stirred for 24 h and then poured into 300 ml of methanol. The resulting yellow precipitate was washed with de-ionized water and dried under vacuum. The crude polymer was dissolved in chloroform and reprecipitated in methanol twice. After Soxhlet extraction with methanol and acetone for 12 h successively, the polymer was finally dried under vacuum. A 0.290 g amount (44% yield) of yellow solid was obtained. FT-IR (KBr), v_{max}/cm^{-1} 3044, 2961, 2923, 2855, 1467, 1250, 1194, 1164, 1108, 1078, 1064, 959, 836, 810, 769, 720, 641, 464. ¹H NMR (CDCl₃, ppm), δ 7.96–7.80 (br, 1H), 7.78–7.62 (br, 1H), 7.58-7.36 (br, 2H), 1.47-1.20 (br, 8H), 1.04-0.72 (br, 10H), 0.56–0.24 (br, 12H). Anal. Calcd. for $(C_{20}H_{34}Si_2)_n$: C, 72.65; H, 10.36; Found: C, 72.59; H, 10.24%.

Results and discussion

The synthetic route for BS-PPV is facile and the detailed conditions are outlined in Scheme 1. The monomer was prepared by radical bromination of 2,5-butyldimethylsilyl-p-xylene, which was synthesized through coupling of the Grignard reagent 2,5-dibromomagnesium-*p*-xylene and butvldimethylsilyl chloride in THF. The monomer was polymerized through the Gilch route in the presence of potassium tertbutoxide in THF. The polymer thus obtained is a yellow solid after precipitation of its chloroform solution twice in methanol. The polymer is fully soluble in common organic solvents such as chloroform, THF, toluene, and xylene. Its molecular weight was measured by means of GPC using THF as eluent and polystyrene as standard to be $M_{\rm n}=73\,400$ and $M_{\rm w}=$ 187400 with a polydispersity index of 2.55. The degree of polymerization of the polymer was calculated as 222. A high molecular weight and long conjugated chain of the backbone are good for the preparation of smooth and uniform films for polymer LED device fabrication and can lower the speed of crystallization of the thin films in LED devices.

The thermal stability of the polymer in air was evaluated by TGA and differential thermal analysis (DTA). The TGA results showed that the polymer has high thermal stability. The thermogram is depicted in Fig. 1. The onset of degradation of the polymer is at about $312 \,^{\circ}$ C. It can be seen that there are two weight loss steps for the polymer. The first step is from 312 to $532 \,^{\circ}$ C with a maximum weight loss rate at



Scheme 1 The synthetic route for BS-PPV. Reagents and conditions: (i) Mg-THF-C₄H₉(CH₃)₂SiCl; (ii) NBS-benzene-BPO-hv; (iii) *tert*-butoxide-THF.



484 °C. The second step is in the range 532-680 °C and the maximum weight loss rate occurs at 624 °C. At 400 °C, the weight loss in air is just about 5%. Above 680 °C, no further weight loss takes place and there is about 20% of residue remaining, which may be charcoal and silicon oxide produced during the degradation. From the percentage weight loss, it can be deduced that the first weight loss is related to the sidechain degradation and the second weight loss corresponds to the decomposition of the polymer backbone.

Fig. 2 shows the UV-Vis absorption and photoluminescence spectra of BS-PPV. The UV-Vis absorption spectra of the film and solution samples are similar, with the same wavelength of maximum absorption at 434 nm. The spectrum of the film sample is slightly broader than that of the solution, which is associated with more $\pi - \pi^*$ transition energy levels along the polymer backbone in the film state. The bandgap can be estimated to be 2.45 eV from the absorption edge of the film sample. The photoluminescence spectra of solution and film samples reveal some differences. The main peak for the solution sample is at 494 nm with a shoulder at 526 nm, whereas the maximum emission for the film samples takes place at 512 nm with a well resolved side peak at 543 nm, which means the emission area lies in the green region. The side peaks of the solution and film samples are associated with vibronic coupling of excitons. Using quinine sulfate (ca. 1×10^{-5} M) in 0.1 M H_2SO_4 as the standard, the quantum yield of BS-PPV in chloroform (ca. 1×10^{-6} M) was measured.¹⁸ The absolute quantum yield is as high as 72%, which is much higher than that of MEH-PPV (14%) measured under the same conditions in our laboratory.

The electrochemical properties of BS-PPV were investigated by CV. The electrochemical redox behavior of the polymer film is an important parameter for its consideration as a light-emitting device. In addition, the energy levels of the lowest unoccupied molecular orbital (LUMO) and the highest occupied molecular orbital (HOMO) of the polymer can be determined from the oxidation and reduction potentials of the polymer films. Fig. 3 presents the cyclic voltammogram of BS-PPV in 0.10 M tetrabutylammonium tetrafluoroborate



Fig. 2 UV-Vis absorption and PL spectra of BS-PPV in solution and in the solid state. (a) UV, solution; (b) UV, film; (c) PL, solution; (d) PL, film.

(Bu₄NBF₄) in acetonitrile, which was obtained in a threeelectrode cell. The polymer films exhibited a partially reversible n-doping process when a cathodic scan was performed. The cathodic peak occurred at -1.95 V with a corresponding anodic peak at -1.78 V (vs. SCE), which represents a charging and discharging process. Along with the potential scan, the color of the films changed from green to purple at first, then to brown after oxidation. The onset for the ndoping process is -1.81 V. It is noted that BS-PPV also shows a p-doping process. The peak potential is 1.37 V, accompanied by a color change from green to blue-green and to yellow-green after reduction. The onset of p-doping took place at 1.16 V. The negative sweep of the polymer film could be repeated for at least five cycles without any significant change in the CV curves, whereas for the positive sweep the corresponding current intensity gradually decreased with the repeated cycles.

From the onset potentials of the oxidation and reduction processes, it can be estimated that the bandgap of BS-PPV is 2.97 eV, which is higher than the value obtained from the optical absorption spectrum. The difference may be caused by the interface barrier between the polymer film and the electrode surface. The optical value corresponds to the energy gap between the valence band and the conduction band, while the electrochemical value may be a combination of the results of the optical bandgap coupled with the interface barrier for charge injection, which makes it larger. However, the electrochemical system is much closer to the practical PLEDs device configuration. Hence the bandgap obtained should be more meaningful as a reference for PLED device design.

According to the empirical relationship obtained by fitting of the experimental data through the valence effective Hamiltonian (VEH) technique, the solid state ionization potential (I_p) and electron affinity (E_a) has a correlation with the E'_{ox} and E'_{red} of the onset potentials for oxidation and reduction.¹⁹ These onset potentials are indicated in Fig. 3. The correlation can be expressed as: $I_p = E'_{ox} + 4.4$ eV and $E_a = E'_{red} + 4.4$ eV. The electrochemical measurement determined that E'_{ox} and E'_{red} are 1.16 and -1.81 V, respectively, and I_p and E_a of the polymer can be roughly estimated as 5.56 and 2.59 eV, respectively.

The I_p and E_a values, optical bandgap, energy barriers of holes injected from ITO (ΔE_h) and electrons from Al (ΔE_e) into the polymer and the imbalance of the energy barrier (ΔE_e $-\Delta E_h$) are listed in Table 1. For the sake of comparison, the data related to PPV and MEH-PPV are also included. The work functions of ITO and Al are 4.6 and 4.2 eV, respectively.^{20,21} The energy barriers for hole and electron injection from ITO and Al into BS-PPV film are 0.96 and 1.61 eV, respectively. It can be seen that there is a 0.65 eV barrier difference ($\Delta E_e - \Delta E_h$) for the injection of the two kinds of elec-



Fig. 3 Cyclic voltammograms of BS-PPV films coated on platinum plate electrodes in acetonitrile containing 0.10 M Bu_4NBF_4 . Platinum wire as the counter electrode. The potentials are referenced against Ag/0.10 M AgNO₃ in acetonitrile. Scan rate: 10 mV s⁻¹.

Table 1 Comparison of optical bandgap, ionization potential (I_p) , electron affinity (E_a) , the energy barrier and barrier imbalance of charge injection of BS-PPV, PPV and MEH-PPV

Polymer	E _g (optical)/ eV	${{I_{\rm p}}/\over{\rm eV^a}}$	${E_{ m a}}/{ m eV^a}$	$\frac{\Delta E_{\rm h}}{{\rm eV^b}}$	$\frac{\Delta E_{\rm e}}{\rm eV^b}$	$\Delta E_{\rm e} - \Delta E_{\rm h} / eV$
BS-PPV	2.45	5.56	2.59	0.96	1.61	0.65
PPV°	2.5	5.0	2.5	0.4	1.7	1.3
MEH-PPV°	2.1	4.9	2.8	0.3	1.4	1.1

^{*a*} The ionization potential (I_p) and electron affinity (E_a) values of BS-PPV were measured using an electrochemical method. ^{*b*} ΔE_h and ΔE_e are the energy barrier of holes and electrons injected into the polymer from ITO and Al, respectively. ^{*c*} The data for PPV, MEH-PPV and the work functions of Al and ITO were taken from the literature.^{20–22}

trical charges. However, this difference is much lower than those of PPV²⁰ and MEH-PPV,²² which are 1.3 and 1.1 eV, respectively as shown in Table 1. The relatively smaller barrier difference for injection of electrons and holes may provide a chance to fabricate devices using an air stable Al cathode with more balanced carriers and high electroluminescence quantum efficiency. From Table 1, we can see that the difference in the E_a values of the three polymers is in a small range, whereas I_p of BS-PPV is much lower than those of PPV and MEH-PPV, which leads to a small barrier difference between ΔE_e and ΔE_h . This could be attributed to the silyl groups introduced into the polymer as the side-chains which increase the oxidation potential of the resultant polymer.

Single layer LEDs with the configuration ITO/BS-PPV/A1 were successfully fabricated. The polymer was dissolved in xylene at a concentration of about 10 mg ml⁻¹ and uniform polymer films were prepared by spin coating the solution on to an ITO coated substrate with a thickness of about 100 nm. The stable Al electrode was used as the cathode. After the cathode had been deposited, the following processing and measuring were all performed under ambient conditions. The I-V characteristics measured for the device is depicted in Fig. 4. A forward bias current was obtained when the ITO electrode was the positive electrode and the Al electrode was grounded. The current density increased with increase in the forward bias and the curve showed a typical diode character. Under a forward bias, the device showed a turn-on voltage at about 6 V and green light became visible at 7.5 V at a current density of about 1.2 mA cm⁻². This result is much better than that of devices using other silyl-substituted PPVs as the active layers with similar configurations, which demonstrated turn-on voltages at 15¹⁶ and 22 V,¹⁴ respectively. The devices showed internal quantum efficiencies of 0.1-0.2%, which are comparable to the reported values.^{14,16} This comparison is very encouraging and we think that this polymeric material may be valuable for green light-emitting device application. It can be expected that the performance would be better if the devices were fabricated and measured under an inert atmosphere and more carefully controlled experimental conditions.



Fig. 4 Current density vs. bias voltage characteristics of an ITO/BS-PPV/Al device.

Conclusions

Fully soluble poly(p-phenylenevinylene) containing two silvl groups as pendants was synthesized through a polymerization reaction. TGA indicated that the polymer is stable below 300 °C in air. UV-Vis spectra of the polymer solution and film samples exhibited the same maximum absorption. The optical bandgap was measured to be 2.45 eV from the absorption edge of the film samples. PL spectra indicated that the polymer emits intense green light. A quantum efficiency of 72% of the polymer solution was observed. CV indicated that the polymer is electrochemically active, and can be both n-doped and p-doped. The n-doping process is partially reversible. However, the p-doping cycle is irreversible, which could be attributed to the instability of the doped state. The onset potentials of the oxidation and reduction processes were determined and the HOMO and LUMO energy levels were estimated to be 5.56 and 2.59 eV, respectively. The HOMO is much lower than those of PPV and MEH-PPV which can be ascribed to the introduction of silvl side-chains into the polymer. Single layer LEDs which deployed BS-PPV as the emissive layer sandwiched between ITO and Al were fabricated. The turn-on voltage of the device is about 6 V and visible green light can be observed at above 7.5 V. Internal quantum efficiencies of 0.1-0.2% were obtained based on the single layer devices.

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