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PURIFIED POLAR POLYFLUORENE FOR LIGHT-EMITTING DIODES AND LIGHT-EMITTING ELECTROCHEMICAL CELLS[*]

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Abstract Conjugated ployfluorene with 2-(2-(2-methoxyethoxy)ethoxy)ethyl groups (EO-PF) is prepared by the palladium-catalyzed Suzuki coupling reaction. The polymer is purified carefully by a simple chemical procedure. The inductively coupled plasma (ICP) test shows palladium-catalyst in the polymer can be removed by this procedure. The thermal properties, electrochemical properties, UV-Vis absorption properties, photoluminescence properties and electroluminescent properties of the polymer without (EO-PF1) or with purification (EO-PF2) are studied. EO-PF2 shows better PL CIE coordinates in THF solutions as blue light-emitting materials and better photoluminescence stability in thin solid films. Polymer light emitting diodes and electrochemical cells based on EO-PF2 exhibit somewhat improved optoelectronic performance than control devices of EO-PF1.

Keyword: Purification; Polyfluorene; Palladium catalyst; Light-emitting electrochemical cells.

INTRODUCTION

In the last two decades, high-efficiency stable light-emitting polymers have drawn great attentions of researchers for their potential applications in full-color displays and the next-generation lighting sources[1, 2]. Due to the large band gap of polyfluorene[3−5] (PF) homopolymer, it is a highly attractive class of conjugated materials for blue light-emitting devices. However, PF-based blue light-emitting materials suffer from poor color stability due to the appearance of long-wavelength green emission in the photoluminescent (PL) or electroluminescent (EL) spectrum[6]. This phenomenon is extensively discussed in scientific literatures as a result of the formation of excimer/aggregate or ketonic defects resulting from photo- and/or electro-oxidation of polyfluorene chains[7−9].

The purity of the monomers and the polymers is crucial to polyfluorene optoelectronic properties, especially for blue light emitting polyfluorene. The monoalkylated fluorene is a typical impurity which can lead to long wavelength emission. The existence of monoalkylated fluorene in polymer backbone can be detected using potassium *tert*-butylate, due to the formation of strongly yellow colored fluoren-9-yl anions[10]. Recently researchers have found new synthetic route or specific method to purify the monomer to get high performance

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photoluminescent polyfluorene. Holmes *et al*. reported a new polyfluorene synthesis route to avoid the presence of emissive fluorenone defects formed by oxidation of monoalkylfluorene impurities[11]. Meijer *et al.* reported improving color purity and stability of dimethyloctyl-substituted polyfluorene by monomer purification[12]. 2-Bromic fluorene as end group is another type of nonemissive quencher which can be easily formed in polyfluorene by Suzuki or Yamamoto coupling reactions[13]. Now end-capping strategy is widely used in polyfluorene synthesis to avoid the possible quenching. The metal catalyst such as palladium (0) in Suzuki coupling reaction is another key factor[14]. Recently, Ma *et al*. reported that the palladium (0) catalyst residual in polfluorene after polymerization can catalyze the oxidation of fluorene with 9-H to form fluorenone which definitely affect the polyfluorene PL and EL properties[15]. Nonpolar polyfluorene can be purified by chromatography to remove most metal palladium (0) catalyst and some low molecular weight oligomers. The impurity in conjugated polymers affects not only the color stability and efficiency in PLED devices, but also the life time. How to control the impurity in conjugated polymers is an important research field in polymer electronic studies. Tsuda *et al*. have reported how to control Pd content down to ppm in polyfluorene[16].

Polar polyfluorenes with polyether-type side groups are widely used in blue light-emitting electrochemical cells and hydrophilic applications[17–20]. These polymers show relatively unstable properties for their high polarity which makes them inconvenient to be purified by chromatography. In this contribution, we report about a simple purification procedure which has good effect on the photo-electronic properties of the ployfluorene with polar 2-(2-(2-methoxyethoxy)ethoxy)ethyl side groups. After careful purification of the polymer, the palladium-catalyst and low molecular weight oligmers can be removed. The UV-Vis absorption properties, photoluminescence properties and electroluminescent properties of the polymers are studied. Preliminary results reveal that the polymers after carefully purification show relatively high light-emitting diodes (LED) and light-emitting electrochemical cells (LEC) devices performances.

EXPERIMENTAL

Materials

All reagents, unless otherwise specified, were obtained from Aldrich, Acros and TCI Chemical Co. and used as received. 2-(2-(2-Methoxyethoxy)ethoxy)ethyl-4-methylbenzenesulfonate (**1**)[21] and 9,9-bis(2-(2-(2-methoxy-ethoxy)ethoxy)ethyl)-2,7-dibromo-fluorene (**2**)[20] were prepared following published procedures with modification and characterized by ^{1}H-NMR. The silica used in purification was obtained from Silicycle Inc.

Synthesis

2-(2-(2-Methoxyethoxy)ethoxy)ethyl-4-methylbenzenesulfonate (1)

A 2-L flask was charged with 2-(2-(2-methoxyethoxy)ethoxy)ethanol (0.5 mol) and 4-methylbenzene-1-sulfonyl chloride in CH_2Cl_2 (500 mL). The homogeneous mixture was kept 0°C with CO_2-acetone bath. NaOH (160 g, 4 mol) was added in small amounts under vigorous stirring. The mixture was stirred for 3 h at 0°C, then CH_2Cl_2 and ice-water were added. The organic layer was washed with water, dried ($MgSO_4$) and concentrated to get pure product **(1)** (99% yield).

^{1}H-NMR (400 MHz, $CDCl_3$, δ): 7.77 (dd, $J = 8.18$, $J = 1.12$, 2H), 7.32 (d, $J = 7.97$, 2H), 4.14 (m, 2H), 3.67 (m, 2H), 3.57 (m, 6H), 3.51 (m, 2H), 3.34 (s, 3H), 2.42 (s, 3H).

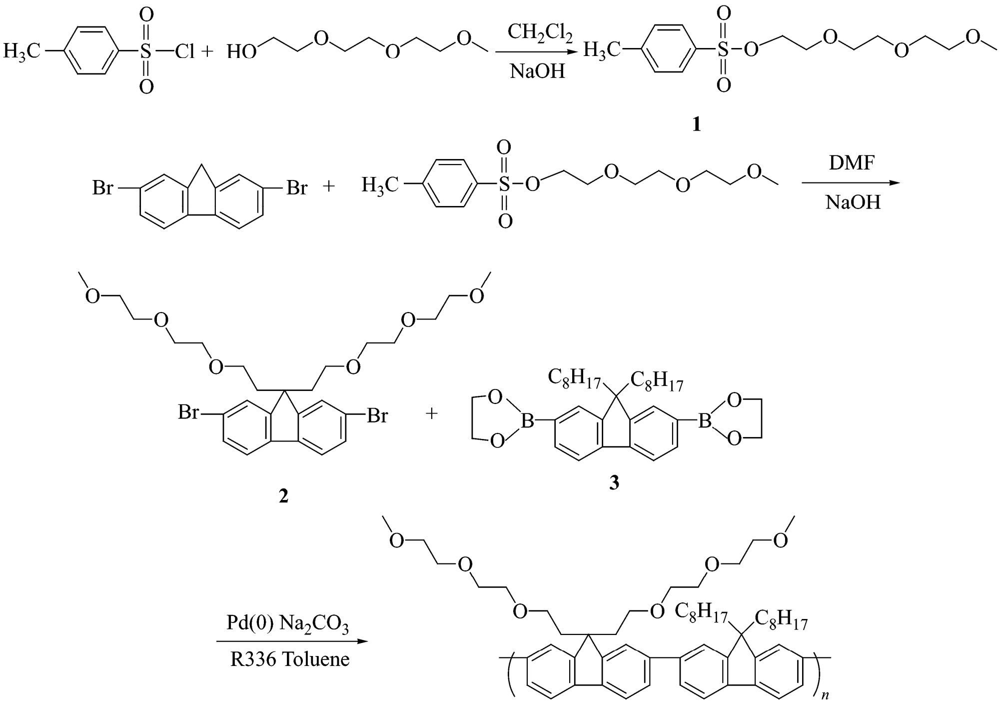
9,9-Bis(2-(2-(2-methoxyethoxy)ethoxy)ethyl)-2,7-dibromo-fluorene (2)

2,7-Dibromo-fluorene (3.24 g, 10 mmol), 2-(2-(2-methoxyethoxy)ethoxy)ethyl-4-ethylbenzenesulfonate (7.00 g, 22 mmol) and tetrabutylammonium bromide (0.39 g, 1.2 mmol) in 150 mL of aqueous KOH (45%) were heated at 70°C for 8 h. After the reaction mixture was cooled to room temperature, the resulting solution was extracted with CH_2Cl_2 (3 × 100 mL). The combined organic fractions were washed with water, dilute HCl and brine and dried over $MgSO_4$. The crude oily product was purified by chromatography on silica gel. First use CH_2Cl_2 and then CH_2Cl_2:ethyl acetate (3:1) as eluent. Wash the product in hexane with carbon to get colorless product. Then the monomer was re-crystallized from hexane to get good crystals (50% yield).

^{1}H-NMR (400 MHz, $CDCl_3$, δ): 7.53 (dd, J = 3.6 Hz, 2H), 7.50 (s, 2H), 7.48 (dd, J = 7.9 Hz, 2H), 3.53 (m, 4H), 3.48 (m, 4H), 3.39 (m, 4H), 3.34 (s, 6H), 3.21 (m, 4H), 2.76 (t, J = 7.6 Hz 4H), 2.34 (t, J = 7.6 Hz, 4H).

Synthesis of the polymer

2, 3 (Scheme 1), $(PPh_3)_4Pd(0)$ (0.5 mol%–1 mol%) and Aliquat336 were dissolved in a mixture of toluene and an aqueous solution of 2 mol/L Na_2CO_3. The mol ratio of **2** and **3** was kept 1:1 in the reaction. The solution was refluxed at 90°C for 24 h with vigorous stirring in an argon atmosphere. At the end of polymerization, the polymers were end-capped with **3** and bromobenzene. The organic mixture was then poured into methanol and the polymer precipitate was separated by filtration. The polymer was re-dissolved in THF, filtered through 1.5 μm filter, and precipitated in methanol. The polymer was air-dried, followed by drying in vacuum at ambient temperature. The yield of the resulting copolymer, EO-PF, was around 95%.



Scheme 1 Synthesis route of monomers and the polymer

^{1}H-NMR (400 MHz, $CDCl_3$, δ): 7.84, 7.69, 7.51, 3.53, 3.46, 3.31, 2.94, 2.59, 2.17, 1.15, 0.81. M_n = 24000, M_w = 122000.

The thermal gravimetric analysis (TGA) showed that the degradation temperature (T_d) of the polymer was around 406°C (5% decomposition).

Purification Procedure of the Polymer

The polymer toluene solution was washed with 1 mol/L HCl and DI water and treated with an organic silica gel (Silicycle Inc.) functionalized with hydroxyl, amino, carboxylic acids, nitriles or aminoacids groups. The solution was precipitated in methanol. The polymer was collected on filter paper, air-dried and finally dried in vacuum at ambient temperature.

Gel permission chromatography of the resulting polymer (EO-PF2) showed M_n = 28000 and M_w = 225000.

Measurements

Photoluminescence (PL) spectra were measured on a Photon Technology International spectrophotometer. ^{1}H-NMR spectra were recorded on a Bruker DRX 400 spectrometer operating at 400 MHz. Cyclic voltammetry was measured on a Potentiostat/Galvanostat model 283 electrochemical workstation (Princeton Applied Research) at a scan rate of 50 mV/s in a 1 mol/L $LiClO_4$ solution in acetonitrile (CH_3CN), with platinum working electrode and saturated calomel electrodes (SCE) as reference electrode. The polymer molecular weights were determined by GPC (Waters) using polystyrene standards and THF as eluent.

Devices

Light emitting diodes (LED) was fabricated on pre-patterned indium-tin oxide (ITO) with a sheet resistance of 10–20 Ω/□. The substrate was ultrasonically cleaned with acetone, detergent, deionized water and 2-propanol subsequently. Oxygen plasma treatment was made for 10 min as the final step of substrate cleaning to improve the contact angle just before film coating. Onto the ITO glass a 50 nm thick layer of polyethylenedioxythiophene-polystyrene sulfonic acid (PEDOT:PSS) film was spin-coated from its aqueous dispersion (Baytron P 4083, Bayer AG). This PEDOT:PSS layer was included to improve the hole injection and reduce the possibility of leakage. It was dried at 80°C for 2 h in a vacuum oven. Then the solution of the copolymer was spin-coated on top of the ITO/PEDOT:PSS surface. The typical thickness of the emitting layer was 70–80 nm. A thin layer of barium as an electron injection cathode and a 200 nm thick aluminum layer were successively deposited by vacuum thermal evaporation through a mask. The vacuum pressure was below 2×10^{-4} Pa. The deposition speed and thickness of the barium and aluminum layers were monitored by a thickness/rate meter (model STM-100, Sycon). The active area of the devices in this study was 0.17 cm^2. The spin coating of the EL layer and device testing were carried out within a glove box (Vacuum Atmosphere Co.) with nitrogen circulation. Current-luminance-voltage (*I-L-V*) characteristics were measured with a computer-controlled Keithley 236 Source Measure Unit and calibrated by Si photodiode. Luminance was calibrated by PR705 spectragraph-photometer after the encapsulation of the devices with UV-curing epoxy and thin cover glass. EL spectra were taken by InstaSpec™ IV CCD spectragraph.

LECs were fabricated similar to the PLED devices except that the LECs have a layered configuration of ITO/PEDOT:PSS/EO-PF:lithium trifluoromethanesulfonate blend/Al. The polymer and lithium trifluoromethanesulfonate (Aldrich) blend (weight ratio of (5.2–5.6):1) was spin-coated from a cyclopentanone solution. The coating thickness was 50–80 nm.

RESULTS AND DISCUSSION

Synthesis and Characterization

The general synthetic route to the polymer (EO-PF) is outlined in Scheme 1. The obtained polymer is readily soluble in common organic solvents, such as THF and chloroform. ^{1}H-NMR chemical shifts at *ca.* $\delta = 3.53$, 3.46, 3.31, 2.94 and 2.59 are assigned to the polar 2-(2-(2-methoxyethoxy)ethoxy)ethyl side group. The polymer can not be purified by silica chromatography using toluene as eluent as usual polyfluorene for the highly polar 2-(2-(2-methoxyethoxy)ethoxy)ethyl side group, so we have used one suite purification method to purify this polymer as we mentioned in the experiment section. The polymers without and with this purification method were named EO-PF1 and EO-PF2. During the purification procedure the polymer solution is treated with functionalized silica gel (Silicycle Inc.). The functional groups such as hydroxyl, amino, carboxylic acids, nitriles or aminoacids groups can form a very strong affinity for the residual Pd metal to remove it from the products. It has been reported to be effective to remove Pd catalyst in polar reaction product[22]. The ICP tests show that the Pd catalyst in EO-PF1 is 149×10^{-6}. The ICP tests of EO-PF2 show no response. (Indicates palladium content in polymer EO-PF2 solution is below the detection limits of the instrument (10×10^{-9}) with polymer concentration 0.14 mg/mL). ICP testing results of EO-PF1 and EO-PF2 show that after purification Pd catalyst can be removed. Although the Pd catalyst scavenging conditions described in this paper are unoptimised and scavenging efficiency is affected by a number of experimental factors, including contact time, temperature and concentration, as well as the nature of the structural groups present within the functionalised silica scavenger[22], our purification method is very efficiency for Pd catalyst scavenging. The number-average and weight-average molecular weight of EO-PF1 and EO-PF2 determined by GPC using a polystyrene standard is

M_n = 24000, M_w = 122000 and M_n = 28000 and M_w = 225000, respectively. After purification, the polymer shows relatively higher molecular weight, which might be the evidence for removal of some low molecular weight component.

Optical Properties and Electrochemical Properties

The normalized UV-Vis absorption spectra of EO-PF in THF solution (Fig. 1.) show a peak at 390 nm attributable to π-π^* transition for polyfluorene backbone. The full width at half maximum is 73 nm and 51 nm respectively for EO-PF1 and EO-PF2. The optical band gap (E_g) estimated from the onset wavelength of UV-Vis spectra of the copolymer solid films is 2.88 eV and 2.98 eV, respectively for EO-PF1 and EO-PF2.

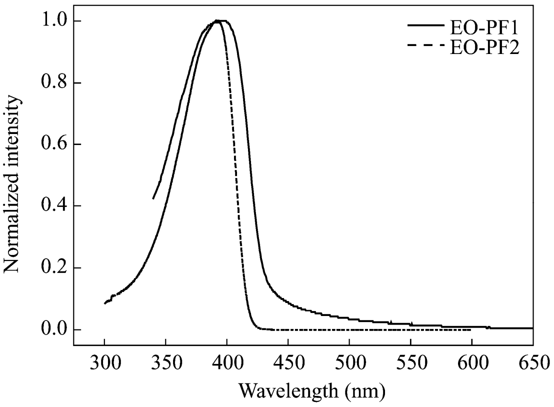


Fig. 1 UV-Vis absorption spectra of EO-PF1 and EO-PF2 in THF solutions

The electrochemical properties of EO-PF were investigated by cyclic voltammetry (CV) which shows one p-doping process in the polymer. The onset potentials of the p-doping are 1.2 V. Using the empirical formula[23] $E_{\mathrm{HOMO}} = -(E_{\mathrm{ox}} + 4.4)$ (eV), the HOMO of EO-PF is calculated with $E_{\mathrm{HOMO}} = -5.6$ eV. The LUMO level is estimated from its HOMO level and optical energy gap with $E_{\mathrm{LUMO}} = -2.62$ eV which is similar with the E_{LUMO} of poly(dioctylfluorene) 2.12–2.6 eV[24, 25].

Photoluminescence Properties

The PL spectra of EO-PF1 and EO-PF2 in THF solution excited by 350 nm laser are shown in Fig. 2. The peak at 429 and 448 nm is attributed to the emission of polyfluorene backbone. The EO-PF2 PL intensity decreases

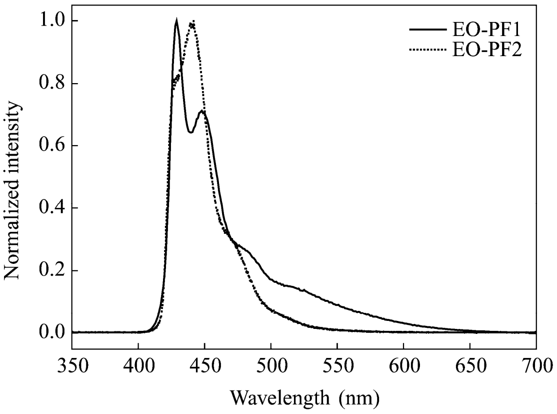


Fig. 2 PL spectra of EO-PF1 and EO-PF2 in THF solutions

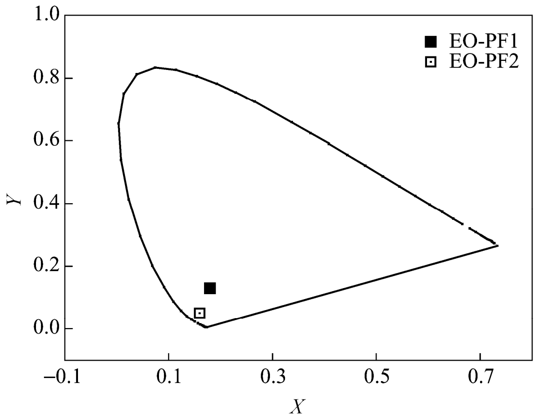


Fig. 3 CIE (1931) color coordinates of PL spectrum of EO-PF1 (black squares) and EO-PF2 (white squares) in THF solution

significantly in the region from 470 nm to 650 nm compared with that of EO-PF1. The full width at half maximum of the polymer PL spectrum decreases by 3.5 nm after purification. The CIE coordinates of the PL spectrum of EO-PF1 and EO-PF2 are respectively (0.18, 0.13) and (0.16, 0.05) (Fig. 3). After simply careful purification, the CIE of EO-PF2 turns to be deeper blue than the CIE blue (0.155, 0.086). The normalized PL spectra of EO-PF1 and EO-PF2 in thin solid films after being annealed at 80°C and 120°C are shown in Fig. 4. The EO-PF2 polymer shows relatively better PL thermal stability.

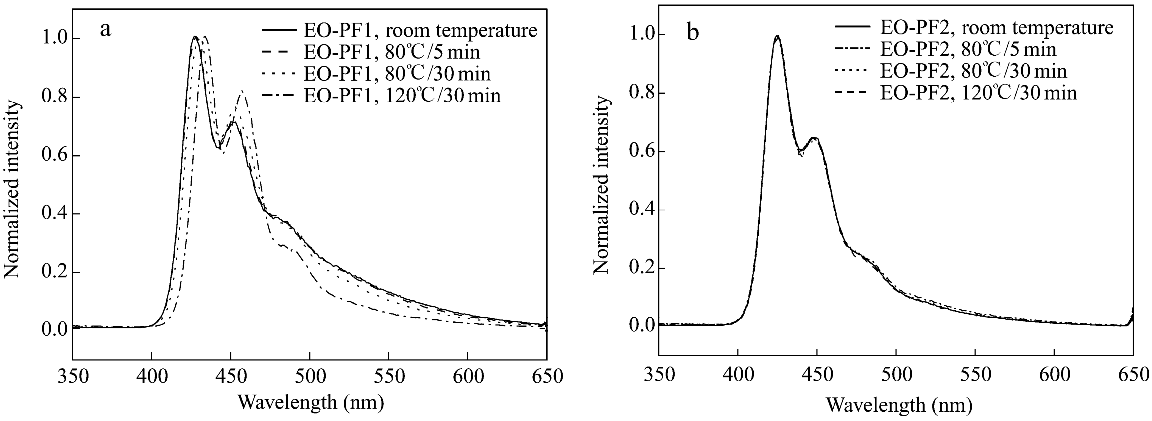


Fig. 4 Normalized PL spectra of EO-PF1 (a) and EO-PF2 (b) in thin solid films at room temperature and after annealing (80°C/5 min, 80°C/30 min and 120°C/30 min)

Electroluminescent Properties

Polymer light emitting diodes (PLEDs) were fabricated using spin-cast EO-PF films as the emitting layer. A PEDOT:PSS layer was used to improve the hole injection and reduce current leakage. The device configurations used were ITO/PEDOT:PSS/EO-PF/Ba/Al. Figure 5 shows the EL spectra of the two LEDs based on polymers EO-PF1 and EO-PF2. The polymer EO-PF2 shows relatively low emission intensity in the region of 500–600 nm. The EO-PF1 and EO-PF2 based light emitting diodes show EL CIE (0.19, 0.22) and (0.18, 0.15) and maximum luminance 324 cd/m^2 and 1323 cd/m^2, respectively. The LED performances are further tabulated in Table 1.

Light emitting electrochemical cells (LECs) was fabricated in a similar manner as the LEDs with a device configuration of ITO/PEDOT:PSS/Polymer:Li Salt Blend/Al. The LEC devices have a slow turn-on at 4.4 V and 3.6 V for EO-PF1 and EO-PF2, respectively. When operated at a constant current density of 29.4 mA/cm^2, the devices turned on in 82 and 14 s for EO-PF1 and EO-PF2, respectively. Figure 6 shows EL spectra of polymers in such devices. The EO-PF1 and EO-PF2 based LEC devices show EL CIE (0.18, 0.14) and (0.17, 0.13). The maximum luminance of the LEC device is around 1300 cd/m^2 as shown in Table 2. This kind of polymer after purifications shows relatively good light emitting properties than the LEC device with similar chemical structure reported by Yang[17].

Table 1. PLED device performances of the polymer (ITO/PEDOT:PSS/polymer/Ba/Al)

Polymers	Hole transport layer	V^a (V)	J^b (mA/cm^2)	LE^c (cd/A)	$L_{max}{}^d$ (cd/m^2)	CIE(x, y)
EO-PF1	PEDOT:PSS	5.2	23	0.3	324	0.19, 0.22
EO-PF2	PEDOT:PSS	4.6	35	0.4	1323	0.18, 0.15

^{a} Voltage at maximum luminance efficiency; ^{b} Current density at maximum luminance efficiency;
^{c} Maximum luminance efficiency; ^{d} Max luminance

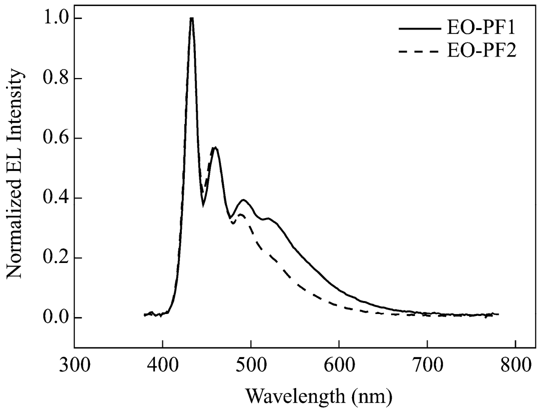


Fig. 5 Normalized EL spectra with LED device configuration ITO/PEDOT:PSS/polymer (EO-PF1 or EO-PF2)/Ba/Al

Table 2. LEC device performances of the polymer (ITO/hole transport layer/polymer/Al)

Polymers	V_{on} [a] (V)	Turn on time[b] (s)	V^c (V)	J^d (mA/cm^2)	LE[e] (cd/A)	L_{max} [f] (cd /m^2)	CIE(x, y)
EO-PF1	4.4	71	7.0	318	0.43	1359	0.18，0.14
EO-PF2	3.6	14	6.3	163	0.45	1311	0.17，0.13

[a] Turn on voltage; [b] Turn on time at constant current density 29.4 mA/cm^2;
[c] Voltage at maximum luminance efficiency; [d] Current density at maximum luminance efficiency;
[e] Maximum luminance efficiency; [f] Max luminance

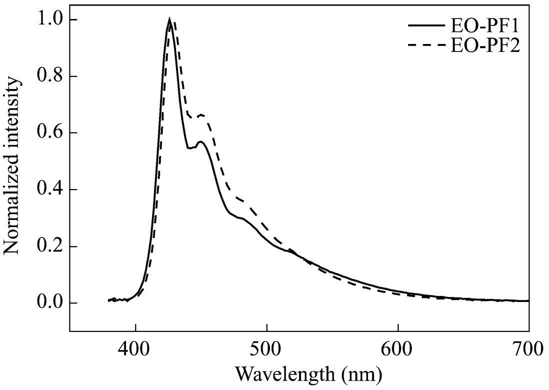


Fig. 6 Normalized EL spectra with LEC device configuration ITO/PEDOT:PSS/polymer (EO-PF1 or EO-PF2):Li Salt Blend/Al

CONCLUSIONS

Conjugated ployfluorene with polar 2-(2-(2-methoxyethoxy)ethoxy)ethyl groups has been prepared by the palladium-catalyzed Suzuki coupling reaction and purified by a suite of simple procedure. This procedure can remove Pd catalyst and some of the low molecular weight fractions in the polymer. The EO-PF2 shows better PL CIE coordinates in THF solutions as blue light-emitting materials, better PL thermal stability in thin solid film and higher maximum luminance in PLED devices than EO-PF1. The LEC devices based on EO-PF2 show relatively low turn-on voltage and short turn-on time. More careful device fabrication and characterization are needed to fully take advantage of the enhanced optoelectronic performances of EO-PF2.

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REFERENCES

1 Lo, S.C. and Burn, P.L., Chem. Rev., 2007, 107: 1097

2 Friend, R.H., Gymer, R.W. and Holmes, A.B., Nature, 1999, 397: 121

3 Li, C., Li, Z., Liu, J.G., Yang, H.X. and Yang, S.Y., Chinese J. Polym. Sci., 2010, 28(6): 971

4 Liu, J.J., Qin, H. and Ba, X.W., Acta Polymerica Sinica (in Chinese), 2011, (2): 217

5 Li, H., Zhao, B., Shen, P., Wan, M.X. and Tan, S.T., Acta Polymerica Sinica (in Chinese), 2010, (5): 501

6 Jiang, H.J., Wan, J.H. and Huang, W., Sci. China Ser. B-Chem., 2008, 51: 497

7 Bliznlyk, V.N., Carter, S., Scott, J.C., Klärnar, G., Miller, R.D. and Miller. D.C., Macromolecules, 1999, 32: 361

8 List, E.J.W., Guentne, R., Freitas, P.S. and Scherf, U., Adv. Mater., 2002, 14: 374

9 Pogantsch, A., Wenzl, F.P., List, E.J.W., Leising, G., Grimsdale, A.C. and Müllen, K., Adv. Mater., 2002, 14: 1061

10 Jacob, J., Sax, S., Piok, T., List, E.J.W., Grimsdale, A.C. and Müllen, K., J. Am. Chem. Soc., 2004, 126: 6987

11 Cho, S.Y., Grimsdale, A.C., Jones, D.J., Watkins, S.E. and Holmes, A.B., J. Am. Chem. Soc., 2007, 129: 11910

12 Craig, M.R., de Kok, M.M., Hofstraat, J.W., Schenning, A.P.H.J. and Meijer, E.W., J. Mater. Chem., 2003, 13: 2861

13 Chen, X.W., Tseng, H.E., Liao, J.L. and Chen, S.A., J. Phys. Chem. B, 2005, 109: 17496

14 Scherf, U. and List, E.J.W., Adv. Mater., 2002, 14 : 477

15 Liu, L.L., Yang, B., Zhang, H.Y., Tang, S., Xie, Z.Q., Wang, H.P., Wang, Z.M., Lu, P. and Ma, Y.G., J. Phys. Chem. C, 2008, 112: 10273

16 Tsuda, Y., Morishita, Y., Nomura, S., Tai, S., Hoshi, Y., Funyuu, S., Motamedi, F.J. and Wang, L.S., 2006, U.S. Patent, 0247474 A1

17 Yang, Y. and Pei, Q.B., J. Appl. Phys., 1997, 81: 3294

18 Oh, S.H., Vak, D., Na, S.I., Lee, T.W. and Kim, D.Y., Adv. Mater., 2008, 20: 1624

19 Oh, S.H., Na, S.I., Nah, Y.C., Vak, D., Kim, S.S. and Kim, D.Y., Organic Electronics, 2007, 8: 773

20 Wang, F.K. and Bazan, G.C., J. Am. Chem. Soc., 2006, 128: 15786

21 Keegstra, E.M.D., Zwikker, J.W., Roest, M.R. and Jenneskens, L.W., J. Org. Chem., 1992, 57 : 6678

22 Galaffu, N., Man, S.P., Wilkes, R.D. and Wilson, J.R.H., Org. Process Res. Dev., 2007, 11: 406

23 Li, Y.F., Cao, Y., Gao, J., Wang, D.L., Yu, G. and Heeger, A.J., Synth. Met., 1999, 99: 243

24 Liao, L.S., Fung, M.K., Lee, C.S., Lee, S.T., Inbasekaran, M., Woo, E.P. and Wu, W.W., Appl. Phys. Lett., 2000, 76: 3582

25 Janietz, S., Bradley, D.D.C., Grell, M., Giebler, C., Inbasekaran, M. and Woo, E.P., Appl. Phys. Lett., 1998, 73: 2453