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New linear π-conjugated polymers via Suzuki coupling of (1*Z*, 3*Z*)-1,4-dibromo-1,4-diaryl-buta-1,3-diene with aromatic diborates: Synthesis and photophysical properties

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ABSTRACT

A series of new linear π-conjugated polymers have been synthesized via Suzuki coupling of (1*Z*, 3*Z*)-1,4-dibromo-1,4-diaryl-buta-1,3-diene with aromatic diborates. The structures were characterized by NMR spectroscopy, IR spectroscopy and GPC. All of them exhibit good thermal stability with high decomposition temperature over 340 °C. It was found that the absorption and emission of the polymers can be adjusted through changing the side aromatic group, and the partial twisted structure of two aromatic groups at the end carbon of buta-1,3-diene unit can hinder the interchain interaction of conjugated main chain and improve the photophysical properties of the polymers in the solid state. The electrochemical and electroluminescent properties of the polymers were primarily studied.

1. Introduction

In the past decades main-chain conjugated polymers have drawn significant attention for their promising applications in many areas such as light-emitting diodes [1–6], photovoltaic cells [7–11], field-effect transistors [12–16], nonlinear optics [17–19], chemical and biological sensors [20–23] etc. Among them linear π-conjugated polymers have been widely studied due to simple synthetic steps, good solubility and well-controlled processibility and easily modified photophysical properties. However, the strong interchain interaction or intermolecular π–π stacking of linear conjugated polymers in the solid state generally results in the red-shift of absorption and emission, and weakens or even quenches the emission, and influences properties of the PLED device such as emission stability and color purity. Thus, in order to avoid these problems some strategies have been developed to prevent the intermolecular interaction of the linear π-conjugated polymers [24–30]. For example, functionalization at the 9-position of fluorene unit for polyfluorenes is a simple and effective method to suppress interchain interaction and improve photophysical properties [31–34].

Buta-1,3-diene unit has been used as a functional group in the synthesis of many organic intermediates for natural and bioactive compounds [35–39], and as a conjugated building block for the design of electro-optical materials [40,41]. Several different methods have been developed for the synthesis of 1,4-dibromo-buta-1,3-diene derivatives [42–44]. Recently, Li et al. [45] reported a simple and effective synthetic method for 1,4-dibromo-1,4-diaryl-buta-1,3-diene via the palladium-catalyzed dimerization of alkyne, which provided an easily synthetic route to design new kind of conjugated system. For example, Yamamoto et al. [46] reported the preparation of head-to-head substituted polyacetylenes by organometallic polycondensation from corresponding 1,4-dibromo-buta-1,3-diene derivatives. Due to existence of two bromide atoms at each end-carbon of buta-1,3-diene unit as reactive point, 1,4-dibromo-1,4-diaryl-buta-1,3-diene can be as a conjugation block connected with some other conjugated group to build linear π-conjugated polymers. In addition, the partially twisted structure from two aromatic groups at same carbon atom of buta-1,3-diene unit can suppress π–π intermolecular stacking and prevent interchain interaction of linear π-conjugated polymers, which may improve the photophysical properties.

Here we reported an effective route to synthesize new kind of linear π-conjugated polymers containing buta-1,3-diene unit connected with different aromatic group, and presented the synthesis and photophysical properties of the polymers.

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2. Experimental section

2.1. Materials

1,2-Dimethoxyethane was purged with nitrogen for 20–30 min before use. 2,2′-(9,9-Dihexylfluorene-2,7-diyl)bis(1,3,2-dioxaborinane) (**4**) was purchased from Acros. All other reagents and solvents were used as commercially purchased without further purification. 9-Hexyl-3,6-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-carbazole [16], 2-ethynylnaphthalene, 9,9-diethyl-2-ethynylfluorene [47,48] 3,6-dibromo-9-hexylcarbazole [49] were prepared according to the references.

2.2. Instruments

^1H NMR and ^{13}C NMR spectra were obtained on a Varian Mercury 300 spectrometer using tetramethylsilane ($\delta = 0$ ppm) as internal standard. IR spectra were recorded on a Perkin–Elmer2 spectrometer with KBr pellets. The elemental analysis was carried out on a CARLOERBA-1106 microelemental analyzer. EI-MS spectra were recorded with a VJ-ZAB-3F-Mass spectrometer. UV–vis spectra were measured with a Shimadzu UV-2550 spectrometer, and the photoluminescence (PL) spectra were obtained using a Hitachi F-4500 spectrometer. Gel permeation chromatography (GPC) analysis was performed on an Agilent 1100 series HPLC system equipped with a G1326A refractive index detector, in which polystyrene standards were used as calibration standards and THF was used as an eluent at the flow rate of 1.0 mL/min. Thermogravimetric analysis (TGA) was performed on NETZSCH STA449C thermal analyzer at a heating rate of 20 °C/min in argon. Cyclic voltammetry (CV) was carried out with a CHI voltammetric analyzer at room temperature in nitrogen–purged anhydrous acetonitrile with 0.10 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$) as the supporting electrolyte at a scanning rate of 100 mV/s. A polymer-coated platinum disc was used as working electrode, a Ag/AgCl electrode as the reference electrode, and a platinum wire as the counter electrode. Ferrocene was used for potential calibration.

2.3. Synthesis of monomer and polymer

General procedure for (1*Z*, 3*Z*)-1,4-dibromo-1,4-diaryl-buta-1,3-diene (monomer **1**–**3**) [45]: To a flask was added arylacetylene (10 mmol), $PdCl_2$ (0.25 mmol) and $CuBr_2$ (30 mmol) in toluene (50 mL) and MeCN (2 mL). The mixture was stirred at room temperature until starting material was completely consumed as monitored by TLC. After the mixture was filtered off and the solvent was evaporated, the residue was purified by column chromatography using petroleum ether as an eluent and recrystallized from hexane.

2.3.1. *(1Z, 3Z)-1,4-Dibromo-1,4-diphenylbuta-1,3-diene (**1**)*

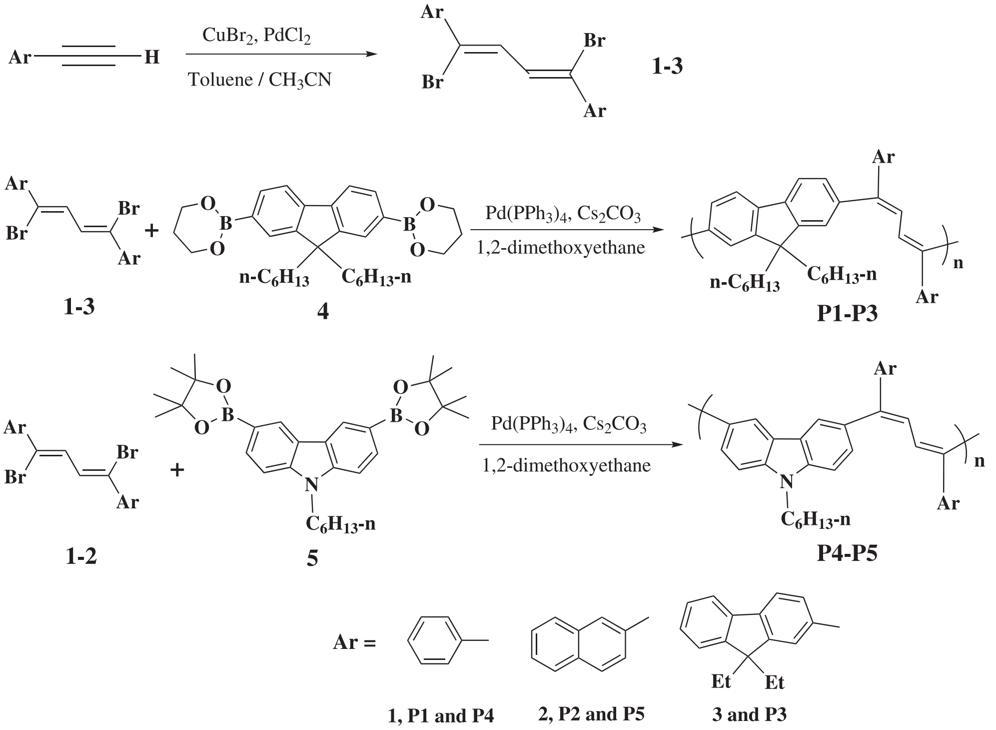
White solid. Yield: 54%. ^1H NMR (300 MHz, $CDCl_3$, δ ppm): 7.70 (d, 4H), 7.40–7.32 (m, 8H). EI-MS: $m/z = 364.0$

2.3.2. *(1Z, 3Z)-1,4-Dibromo-1,4-di(naphthalen-2-yl)buta-1,3-diene (**2**)*

Yellow solid. Yield: 38%. ^1H NMR (300 MHz, $CDCl_3$, δ ppm): 8.20 (s, 2H), 7.93–7.80 (m, 8H), 7.58 (s, 2H), 7.54–7.51 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 136.6, 133.8, 133.3, 130.4, 128.8, 128.6, 128.3, 128.2, 127.8, 127.2, 126.9, 124.8. Anal. Calcd for $C_{24}H_{16}Br_2$: C, 62.10; H, 3.47. Found: C, 62.02; H, 3.34. EI-MS: $m/z = 463.7$.

2.3.3. *(1Z, 3Z)-1,4-Dibromo-1,4-bis(9,9-diethylfluoren-2-yl)buta-1,3-diene (**3**)*

Yellow solid. Yield: 45%. ^1H NMR (300 MHz, $CDCl_3$, δ ppm): 7.69–7.65 (m, 8H), 7.45 (s, 2H), 7.34 (s, 6H), 2.07 (q, 8H), 0.35 (t, 12H). ^{13}C NMR (300 MHz, $CDCl_3$, δ ppm): 150.7, 150.4, 143.0, 140.8, 138.2, 130.6, 127.9, 127.8, 127.2, 127.0, 123.2, 122.5, 120.3, 119.7, 56.4, 32.8, 8.8. Anal. Calcd for $C_{38}H_{36}Br_2$: C, 69.95; H, 5.56. Found: C, 70.43; H, 6.06. EI-MS: $m/z = 652.4$.



Scheme 1. Synthetic routes for the monomers and the polymers.

Table 1
Molecular weight and thermal properties of the polymers.

Polymers	M_w ($\times 10^3$)	M_n ($\times 10^3$)	PDI	T_d (°C)
P1	8.3	4.4	1.9	363
P2	5.7	2.6	2.2	346
P3	13.3	6.3	2.1	347
P4	1.8	1.1	1.6	346
P5	2.0	1.2	1.7	410

Table 2
Optical data and fluorescence quantum yield of the polymers.

Polymer	Absorption λ_{max} (nm)		Emission λ_{max} (nm)		Φ_F (%)
	THF	Thin film	THF	Thin film	
P1	379	378	488	491	37
P2	388	398	498	502	42
P3	404	406	505	509	46
P4	365	372	484	499	21
P5	383	400	505	525	21

2.4. *General procedures for the preparation of the polymers (**P1**–**P5**)* [50]

The related monomer (**1**, **2** or **3**), Pd(PPh_3)$_4$, Cs_2CO_3 were dissolved in dimethoxyethane at argon atmosphere. The mixture was stirred at 65 °C for 3 days. Then the reaction mixture was dropped into 200 mL methanol through a cotton filter. The solid was collected and purified by precipitation into methanol for three times.

P1: 2,2′-(9,9-dihexylfluorene-2,7-diyl)-bis(1,3,2-dioxaborinane) (100.4 mg, 0.2 mmol), monomer **1** (73.2 mg, 0.2 mmol), Pd(PPh_3)$_4$ (23 mg), Cs_2CO_3 (326 mg, 1 mmol). The yellow powder 70 mg. Yield: 65%. ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 7.81 (d, 2H), 7.37–7.21 (m, 14H), 6.97 (s, 2H), 1.94 (t, 4H), 1.08–0.99 (m, 12H), 0.76 (m, 10H). ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 169.2, 151.3, 144.6, 142.8, 140.5, 130.0, 128.4, 127.8, 126.1, 125.5, 119.8, 55.4, 40.5, 31.8, 29.9, 24.2, 22.8, 14.3.

P2: 2, 2′-(9,9-dihexylfluorene-2,7-diyl)-bis(1,3,2-dioxaborinane) (126 mg, 0.25 mmol), monomer **2** (117 mg, 0.25 mmol), Pd(PPh_3)$_4$ (29 mg), Cs_2CO_3 (407 mg, 1.25 mmol). The green powder 125 mg. Yield: 79%. ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 7.91–7.33 (m, 20H), 7.18 (s, 2H), 1.94 (4H), 1.01–0.68 (m, 22H). ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 151.4, 144.8, 140.7, 140.2, 139.2, 133.6, 130.2, 128.5, 127.9, 127.7, 126.9, 126.3, 126.2, 125.7, 125.5, 119.9, 55.5, 40.5, 31.9, 29.9, 24.4, 22.8, 14.3.

P3: 2,2′-(9,9-dihexylfluorene-2,7-diyl)-bis(1,3,2-dioxaborinane) (92 mg, 0.14 mmol), monomer **1** (70 mg, 0.14 mmol), Pd(PPh_3)$_4$ (16 mg), Cs_2CO_3 (228 mg, 0.70 mmol). The yellow powder 72 mg. Yield: 63%. ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 7.88 (s, 2H), 7.64–7.50 (m, 6H), 7.41–7.25 (m, 10H), 7.12 (s, 2H), 7.07 (s, 2H), 2.01–1.88 (m, 12H), 1.07 (s, 12H), 0.76 (s, 10H), 0.31 (t, 12H). ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 149.9, 149.2, 148.7, 143.6, 140.5, 140.1, 139.9, 139.4, 138.2, 129.1, 125.8, 125.0, 123.9, 121.8, 18.6, 118.4, 54.8, 54.2, 39.4, 31.2, 30.8, 28.8, 23.2, 21.6, 13.1, 7.5.

P4: monomer **4** (111 mg, 0.22 mmol), monomer **1** (80 mg, 0.22 mmol), Pd(PPh_3)$_4$ (25 mg), Cs_2CO_3 (359 mg, 1.10 mmol). The yellow powder 50 mg. Yield: 50%. ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 7.49–6.71 (m, 18H), 4.37 (2H), 1.98 (2H), 1.50–1.26 (m, 6H), 0.91 (3H). ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 140.5, 128.7, 128.4, 128.2, 128.1, 128.0, 127.5, 127.2, 126.6, 123.0, 122.9, 108.6, 43.7, 31.8, 29.4, 27.3, 22.8, 14.2,

P5: monomer **4** (91 mg, 0.18 mmol), monomer **1** (84 mg, 0.18 mmol), Pd(PPh_3)$_4$ (21 mg), Cs_2CO_3 (293 mg, 0.90 mmol). The yellow powder 70 mg. Yield: 70%. ^{1}H NMR (300 MHz, $CDCl_3$, δ ppm): 8.16–6.91 (m, 22H), 4.37 (2H), 2.00–1.89 (2H), 1.33–1.28 (m, 6H), 0.94–0.87 (3H). ^{13}C NMR (75 MHz, $CDCl_3$, δ ppm): 140.6, 133.4, 132.8, 128.4, 128.3, 128.1, 127.8, 127.6, 127.3, 126.5, 126.4, 126.2, 126.0, 125.8, 123.1, 108.7, 43.7, 31.8, 29.4, 27.3, 22.8, 14.2.

3. Results and discussion

3.1. *Synthesis and characterization*

As shown in Scheme 1, (1*Z*, 3*Z*)-1,4-dibromo-1,4-diaryl-buta-1,3-diene (monomer **1**–**3**) was synthesized by the dimerization of corresponding arylacetylene in the presence of $PdCl_2$ and $CuBr_2$ in the mixed solvents of toluene and MeCN [45]. In order to adjust the photophysical properties, we selected three aromatic acetylene as starting material to prepare the corresponding (1*Z*, 3*Z*)-1,4-dibromo-1,4-diaryl-buta-1,3-diene. In addition, two aromatic diborates, 2,2′-(9,9-dihexylfluorene-2,7-diyl)-bis(1,3,2-dioxaborinane) (**4**) and 9-hexyl-3,6-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-9*H*-carbazole (**5**), were chosen to react with monomer **1**–**3**. All the new monomers were characterized by NMR, MS and elemental analysis as in the Experimental section.

The Suzuki reaction was applied to synthesize the polymers (**P1**–**P5**) through the C–C coupling of (1*Z*, 3*Z*)-1,4-dibromo-1,4-diaryl-buta-1,3-diene (**1**–**3**) with corresponding aromatic diborates

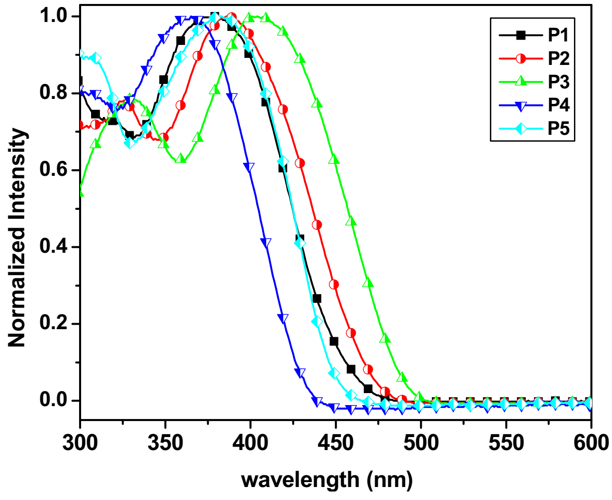


Fig. 1. UV–vis absorption spectra of the polymers in THF (1.0×10^{-6} mol/L).

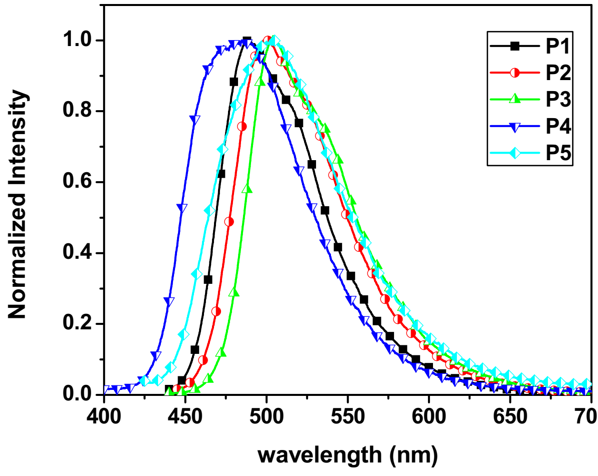


Fig. 2. Photoluminescence spectra of the polymers in THF (1.0×10^{-6} mol/L).

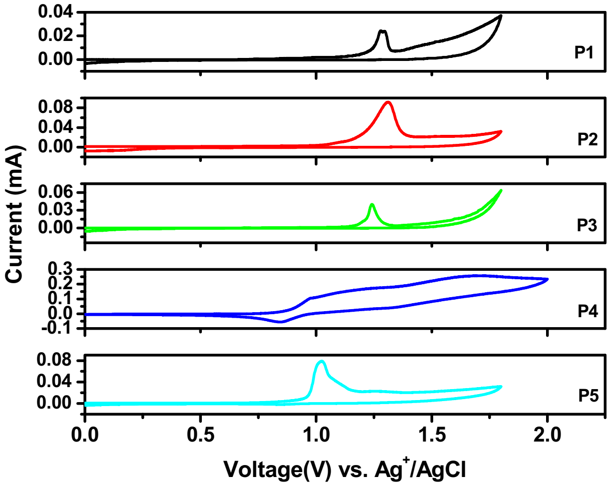


Fig. 3. The cyclic voltammograms of the polymers.

(**4** or **5**) in the presence of Pd(0)-catalyst under argon atmosphere. 1,4-Diaryl-buta-1,3-diene unit was connected with 2,7-(9,9-dihexylfluorene) group as a bridge to yield **P1**–**P3**. The linkage of 2,7-(9,9-dihexylfluorene) group was replaced by 3,6-(*N*-hexylcarbazole) group to produce **P4** and **P5**. All the polymers (**P1**–**P5**) were purified by precipitation in methanol with good yields. The molecular weights were determined by GPC with polystyrene as a standard and THF as solvent. The corresponding data were collected in Table 1. As can be seen, **P1**–**P3** show larger average molecular weight than **P4**–**P5**, which is in agreement with the solubility of the linkage, since 9,9-dihexylfluorene group exhibits better solubility than *N*-hexylcarbazole group. The thermal stability of the polymers was investigated by thermo-gravimetric analysis (TGA) and the corresponding data were collected in Table 1. All polymers show high decomposition temperature (T_d) over 340 °C, indicating that they have high thermal stability.

3.2. Optical properties

The UV–vis absorption spectra of the polymers in THF solution are shown in Fig. 1 and the corresponding data are summarized in Table 2. As can be seen, all the polymers exhibit a broad absorption maxima in the range of 360–410 nm that can be attributed to π–π^* electron-transition contribution. In the same series, such as **P1**–**P3** with fluorene unit as a bridge, the order of UV–vis absorption maxima (404 nm (**P3**) > 388 nm (**P2**) > 379 nm (**P1**)) corresponds to the order of the conjugation length of the side-group: fluorene > naphthyl > phenyl, indicating that UV–vis absorption is mainly related to the conjugation length. Similar phenomenon is also observed for **P4**–**P5** with carbazole unit as a bridge. In addition, the UV–vis absorption spectra in the thin film were also measured (see Fig. S17). Compared with the solution, **P1**–**P4** exhibit little red-shift of UV–vis absorption in the thin film (Figs. S18–21). Only **P5** exhibits an obvious red-shift absorption in the thin film (Fig. S22).

Table 3
Electrochemical Properties of the polymers.

Polymer	E_{ox}^{onset} (V)	E_{HOMO} (eV)	E_{LUMO} (eV)	E_g^{opt} (eV)
P1	1.18	–5.40	–2.84	2.66
P2	1.12	–5.44	–2.83	2.61
P3	1.17	–5.49	–2.95	2.54
P4	0.86	–5.18	–2.37	2.81
P5	0.93	–5.25	–2.60	2.65

Table 4
EL performances of **P1**, **P3** and **P5** in devices with the configuration of ITO/polymer (35 nm)/TPBI (40 nm)/LiF/Al.

Polymer	λ_{ELmax} (λ_{PLmax}) (nm)	$V_{turn\text{-}on}$ (V)	J_{max} (mA/cm^2)	L_{max} (cd/m^2)	η_{max} (cd/A)	EQE (%)
P1	496 (491)	12.5	109	144	0.19	0.09
P3	512 (509)	8.9	242	520	0.27	0.07
P5	528, 596 (525)	11.5	200	324	0.20	0.08

Fig. 2 shows the photoluminescence (PL) emission spectra of the polymers in THF solution. It can be seen that all the polymers exhibit blue or green emission in solution. **P1**–**P3** show the maximum emission at 488, 498 and 505 nm and **P4**–**P5** at 484 and 505 nm, respectively, which is also in agreement with the order of conjugation length of the side group: fluorene > naphthyl > phenyl. We also investigated PL properties of the polymers in thin film (see Fig. S23). **P1**–**P3** in thin film exhibit small red-shift emission (3–4 nm) compared with the solution. However, **P4**–**P5** exhibit larger red-shifted emission (15–20 nm) in thin film than that in solution. In addition, the quantum yield of **P1**–**P3** is obviously higher than that of **P4**–**P5** in solution. These suggest that interchain interaction exists in solid state for **P4**–**P5**, but it has been effectively suppressed in **P1**–**P3**. The possible reason is that the co-planarity of carbazole group with 1,3-buta-1,3-diene unit as well as a long-chain alkyl group at N atom of carbazole group in **P4**–**P5** is favorable to the π–π stacking of main chain that results in aggregation of the π-conjugated system. Unlike **P4**–**P5**, two long-chain alkyl groups at 9 position of fluorene group in **P1**–**P3** hinder the interchain interaction or intermolecular aggregation.

3.3. Electrochemical and electroluminescent properties

In order to investigate the electrochemical properties of the polymers and estimate their HOMO and LUMO energy levels, cyclic voltammetry (CV) was performed using a platinum disk as working electrode and a silver wire as a reference electrode. CV measurements were conducted in a 0.10 M $Bu_4N \cdot PF_6$ acetonitrile solution at room temperature under argon with a scan rate of 100 mV/s, which was calibrated against an internal standard, ferrocene (Fc) with the ionization (IP) value of –4.8 eV of the Fc/Fc$^+$ redox system below the vacuum level [51]. The formal potential of Fc was measured to be 0.48 eV against Ag electrode. Therefore, the HOMO levels of copolymers can be calculated by formula $E_{HOMO}/\mathrm{eV} = -e(E_{ox} + 4.32)$ [52]. The optical band gap (E_g^{opt}) was used to estimate

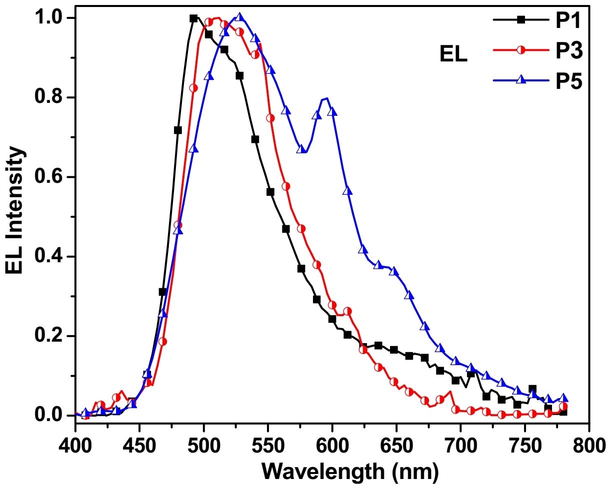


Fig. 4. The EL spectra of **P1**, **P3** and **P5** in ITO/polymer/TPBI/LiF/Al.

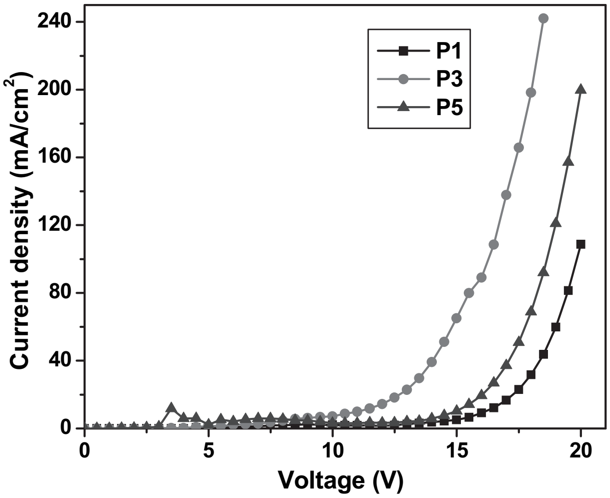


Fig. 5. I–V curve of **P1**, **P3** and **P5** in ITO/polymer/TPBI/LiF/Al.

the LUMO energy level of the polymers, where E_g^{opt} was obtained from the absorption edge of UV–vis spectrum of the film [53,54].

The oxidation processes (p-doping) of the polymers are shown in Fig. 3. As can be seen, the polymers obviously display irreversible redox behavior. The partially twisted conjugated main chain is not favorable to the charge transport between the electrode and the polymer that stops the p-dedoping process. This electrochemical behavior is much similar to that of the head-to-head substituted polyacetylenes prepared from 1,4-dibromo-buta-1,3-diene derivatives [46]. For **P1**–**P3**, they have the same backbone structure of buta-1,3-diene unit connected with fluorene group, so they show the similar electrochemical behavior. Compared with **P1**–**P3**, **P4** and **P5** have different backbone structure, in which the electron-richer carbazole group replaces the fluorene group, so they show lower oxidative voltage than **P1**–**P3**. The HOMO levels are calculated according to the above empirical formula. However, the reduction signal of the polymers was not detected in the CV scans. Therefore, the LUMO level is estimated from the difference between the HOMO level and the optical band gap as reported in some references [53,54]. The calculated results are summarized in Table 3. As can be seen, the polymers with carbazole group as a bridge (**P4** and **P5**) have much lower energy levels than that of the polymers with fluorene group as a bridge (**P1**–**P3**), indicating that the energy level of the polymer can be modified by the introduction of the different aromatic group to connect buta-1,3-diene unit.

The PLED devices with the configuration of ITO/polymer (35 nm)/TPBI (40 nm)/LiF/Al for **P1**, **P3** and **P5** were fabricated to investigate the EL properties of the polymers, in which 1,3,5-tris(1-phenyl-1*H*-benzimidazol-2-yl)benzene (TPBI) was selected as hole blocking materials and the polymer was applied as emission materials. The data for their EL performances are summarized in Table 4. Fig. 4 presents the EL spectra of **P1**, **P3** and **P5**. As can be seen, the EL spectra of **P1** and **P3** is similar to that of the PL spectra in film state, but **P5** shows another EL emission peak at 596 nm compared with its PL emission. Figs. 5 and 6 show the I–V curve and light output of **P1**, **P3** and **P5** in ITO/polymer (35 nm)/TPBI (40 nm)/LiF/Al. For example, the light-on voltage of the PLED device for **P3** is about 8.9 V and the brightness reaches 540 cd/m^2 at 18.5 V. Generally, the factors that influence the EL performances of the polymers are related to the molecular structure, molecular weight, solubility in organic solvents, quality of the solid film and the device configuration. Therefore, in order to achieve a higher EL performance, the attempt for enhancing molecular weight, improving the solubility of the polymers and optimizing the device structure should be further studied.

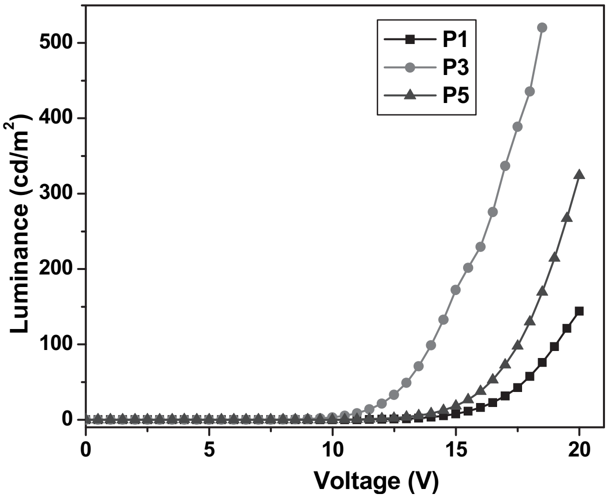


Fig. 6. The light output of **P1**, **P3** and **P5** in ITO/polymer/TPBI/LiF/Al.

4. Conclusions

A simple synthetic approach has been developed for a new kind of linear π-conjugated polymers containing buta-1,3-diene unit. Through changing the main-chain linkage and side aromatic group the optical properties can be adjusted. The introduction of twisted conjugation structure can suppress interchain interaction or intermolecular aggregation for these polymers. The EL performances for the non-optimized PLED devices with the configuration of ITO/polymer (35 nm) indicate that this new kind of polymers can be applied as emission materials for PLED application.

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Appendix. Supplementary information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.polymer.2010.06.003.

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