

Conjugated Polymers for Optoelectronic Applications

Shengang Xu, Yingliang Liu, Shaokui Cao*

Summary: Novel conjugated polymers containing carbazole, phenothiazine or tri-phenylamine units in the main chain were designed and synthesized via Wittig, Knoevenagel or Heck condensations respectively. A majority of them have good solubility in common organic solvents, high thermal stability and good hole-injection ability. Their diluted solutions in THF showed strong absorption with the absorption maximum in the range of 294~470 nm and the optic band gaps located in the range of 1.90~2.75 eV. When irradiated by ultraviolet or visible light, the diluted solutions in THF of the polymers emitted light from purple to yellow color with the emission maximum in the range of 347~597 nm and the full width at half maximum located in the range of 59~119 nm. Several polymeric light-emitting diodes (PLEDs) devices were fabricated using these polymers as light-emitting materials, and a double-layer device composed of ITO/PEDOT:PSS/PQTN/Mg:Ag showed a good performance, in which the maximum brightness was measured as 2434.0 cd/m² under a 11.0 V forward bias voltage. Photovoltaic devices were also investigated using these polymers as an active layer, and a device composed of ITO/PNB/PTCDI-C₁₃/Al showed a good performance, which was estimated to have external quantum efficiency at around 1% at 330 nm. From these preliminary experimental results, we may infer that these polymers are good light-emitting materials for PLEDs; while for photovoltaic applications, their absorption spectra need to be further improved to match the solar illumination.

Keywords: conjugated polymer; light-emitting; photovoltaic; polycondensation

Introduction

The initial report of polymeric light-emitting diodes (PLEDs) based on poly(p-phenylenevinylene) (PPV)^[1] gave birth to an intense research effort in conjugated polymers, primarily focused on the development of optoelectronic and electrochemical devices such as light emitting diodes (LEDs), photovoltaic devices (PVDs), field effect transistors (FETs), and electrochromic devices (ECDs). For any application, it is the predominant advantage of the conjugated polymers to tailor their electronic structure and material properties via tailoring their chemical structures. For optoelectronic applications, it is especially important to control the electronic structure of the polymer to

achieve a desired band gap. Developing methodologies to attain such precise control over the polymer electronic structure is a key aspect for the advancement of the field.

Significant developments in modern synthetic chemistry, especially the chemistry of carbon-carbon bond formation (through Kumada, Stille, Heck, Yamamoto, Suzuki, and Sonogashira couplings, etc.), have allowed the synthesis of various well-defined conjugated polymers and oligomers with optimized physical properties. While, these reactions normally require a transition metal catalyst, which results in a problem in materials purification that is harmful to materials physical properties. On the other hand, Wittig and Knoevenagel condensations are also famous reactions for the formation of carbon-carbon double bond, which can proceed without a transition metal catalyst.

Based on the above considerations, we have primarily focused our effort on the

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conjugated polymer preparation through Wittig and Knoevenagel condensations. Some conjugated polymers containing charge transporting groups or fluorescent dye in main chain were synthesized via these condensations. The photophysical and electrochemical properties of these conjugated polymers were investigated. Using some polymers as light-emitting materials or photovoltaic materials, the single-, double- or multi-layer light-emitting diodes or photovoltaic devices were fabricated. In the present article, the progress of the polymers preparation and their application in optoelectronic fields in our group^[2–7] are presented.

Experimental Part

Synthesis of Monomers

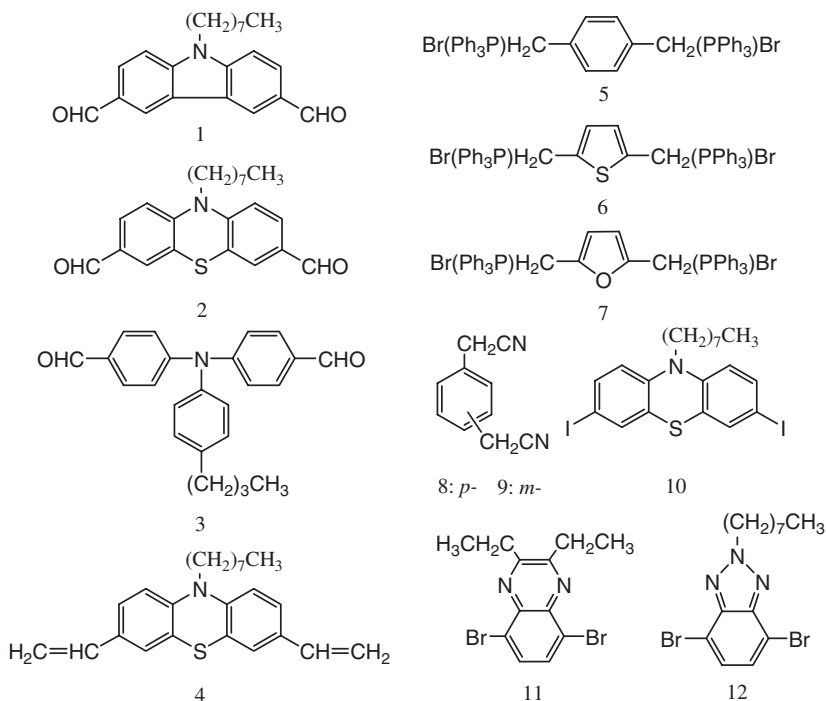
Scheme 1 shows the chemical structures of the monomers used for polymer synthesis. Except for monomer **8** and **9** used as

received, the other monomers were synthesized in our laboratory according to literatures.^[8–17] Their chemical structures were confirmed through spectroscopic technologies.

Polymers Synthesis

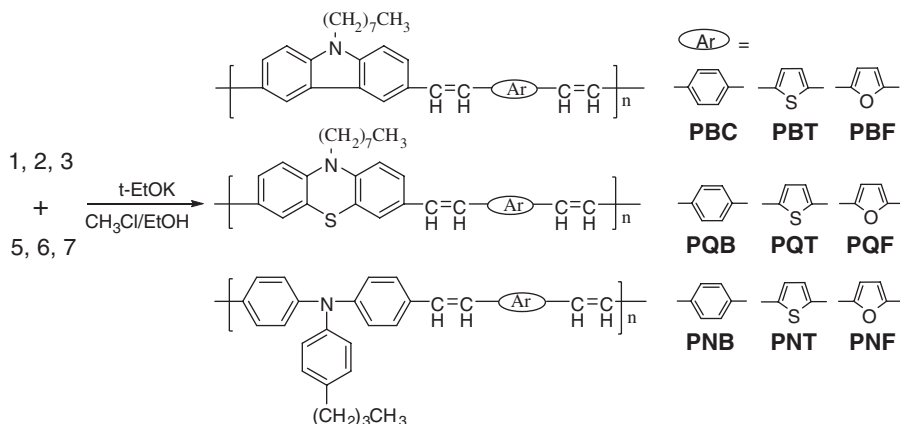
Synthesis of Polymers PBC~PNF^[8]

As shown in Scheme 2, the nine polymers PBC~PNF were synthesized by Wittig condensation of the dialdehyde **1**, **2** and **3** with the phosphorus ylide **5**, **6** and **7**, respectively. As a typical example, polymer PBC was synthesized as follows: monomer **1** (0.3354 g, 1.0 mmol) and compound **4** (0.3672 g, 1.0 mmol) was added to a 25 mL 3-necked round-bottom flask, then 10 mL mixture solvent of ethanol and chloroform (volume ratio: 1:2) was added too. After the monomers completely dissolved, a solution of potassium tert-butoxide (*t*-BuOK) (0.590 g, 5.26 mmol) in a 10 mL above mixture solvent was dropwise added. The mixture



Scheme 1.

Chemical structures of monomers.

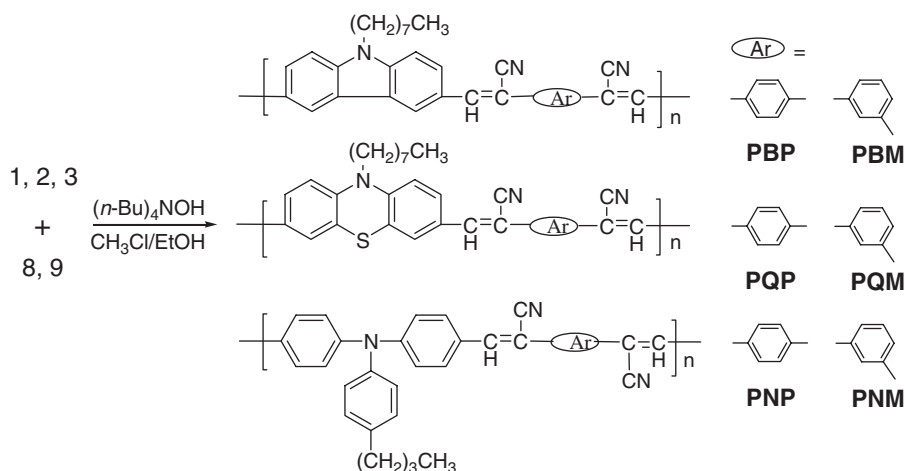
**Scheme 2.**

Synthesis of PBC~PNF via Wittig condensation.

was stirred for 48 hr under refluxing. Solvents were removed under reduced pressure, and the residual was dissolved in THF and the insoluble substances were removed through filtration. The filtrate was precipitated from methanol (200 mL). Upon drying in vacuo, PBC was obtained as yellow powders with 41% yield. The other eight polymers were synthesized similar to that of PBC with a yield from 37% to 65%. Ft-IR spectroscopy revealed that the C=C double bond in these polymers is in a *trans*- configuration.

Synthesis of Polymers PBP~PNM^[9]

As shown in Scheme 3, the six polymers PBP~PNM were synthesized by Knoevenagel condensation of the dialdehyde **1**, **2** and **3** with the diacetonitrile **8** and **9** respectively. As a typical example, polymer PNP was synthesized as follows: monomer **3** (0.7140 g, 2 mmol) and monomer **8** (0.3120 g, 2 mmol) were added to a 25 mL 3-necked round-bottom flask, then 5 mL THF as solvent was added too. After the monomers completely dissolved, 0.5 mL (n-Bu)₄NOH was dropwise added to the

**Scheme 3.**

Synthesis of PBP~PNM via Knoevenagel condensation.

mixture solution. Then, the reaction solution was heated to 60 °C under the protection of nitrogen. After the reaction was carried on for 4 hr, the mixture was cooled to room temperature and precipitated with methanol (100 mL). The precipitate was filtered and dried in vacuo. The amaranthine powder was obtained with 65% yield. The other five polymers were synthesized similar to that of PNP with a yield from 54% to 65%.

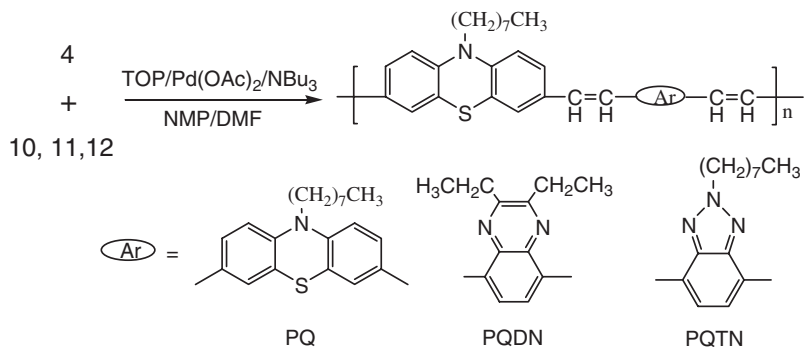
Synthesis of Polymers PQ~PQTN^[7]

As shown in Scheme 4, the three polymers PQ~PQTN were synthesized from the dialkene **4** with the dichloride **10**, **11** and **12** via Heck condensation. As a typical procedure, PQTN was synthesized as follows: A mixture of monomer **4** (0.5454 g, 1.5 mmol), monomer **12** (0.5835 g, 1.5 mmol), Pd(OAc)₂ (0.0168 g, 0.075 mmol), tri-(*o*-tolyl)phosphine (0.1368 g, 0.45 mmol), DMF (8 mL), tri-*n*-butylamine (5 mL), and *N*-methylpyrrolidone (4 mL) were placed in a 25 mL 3-necked round-bottom flask. The mixture was heated at 120 °C for 48 hr with stirring. After cooling to room temperature, the solution was poured into methanol and the precipitate was filtered, washed with methanol. The polymer was purified by dissolution in THF, filtrated and precipitated in methanol again. The polymer was obtained as a red powder with 78% yield. Then PQ and PQDN were synthesized similar to that of PQTN. Ft-IR spectroscopy revealed that the C=C double

bond in these polymers is also in a *trans*-configuration.

Instruments

Melting point was determined using an X-5A melting point measurement instrument. ¹H NMR spectra were recorded on a Bruker DPX-400 spectrometer (400 MHz). Ft-IR spectra were measured on a Nicolet Protégé 460 infrared spectrophotometer. Weight molecular weight (Mw) and molecular weight distribution (MWD) were determined by gel permeation chromatography (GPC) on a Waters M515 instrument. Thermogravimetric (TG) analysis was conducted on a NETZSCH TG-209 calorimeter. UV-vis spectra were measured on a Shimadzu UV-3010 instrument. The fluorescent and electroluminescent spectra were recorded on a Hitachi F-4500 fluorescence spectrophotometer. Cyclic voltammetry (CV) was measured on a CHI 650 electrochemistry workstation. Polymer films were prepared by spin-coating on a KW-4A spin-coater. Polymer film thickness was measured by a Jobin Yvon UVISEL ellipsometer. For PLEDs, the current-voltage (I-V) and luminance-voltage (L-V) characteristics were simultaneously measured using a programmable Array 3645A power supply and a PR650 SpectraScan spectrophotometer (Photo Research). Furthermore, as for photovoltaic devices, the dark and illuminated J-V characteristics of the devices were measured using a Keithley 2410 source meter. And the output



Scheme 4.

Synthesis of PQ~PQTN via Heck condensation.

light power was estimated with a calibrated optical meter (Newport 1830C).

Results and Discussion

The chemical structures of the intermediates, monomers and polymers were confirmed by melting point, ^1H NMR, Ft-IR, etc.

Solubility, Molecular Weight and Thermal Properties of Polymers

The solubility of all polymers was tested in some common organic solvents at room temperature, and the results are summarized in Table 1. The nine polymers PBC~PNF, which were synthesized by Wittig condensation, have good solubility in common organic solvents such as tetrahydrofuran (THF), chloroform, dichloroethane (DCE), acetone, *N*, *N*-dimethylformamide (DMF). And the three polymers PQ~PQTN also have good solubility in THF, chloroform, DCE, acetone and DMF. While for the six polymers PBP~PNM, although they have good

solubility in THF, they are partly soluble in chloroform, DCE, acetone and DMF.

The molecular weight of all the polymers were measured by GPC at 30 °C using THF as eluent and standard polystyrene as reference. The GPC results are also summarized in Table 1. The Mw and MWD of them are in the range of $2.1\sim 48.2 \times 10^3$ and 1.40~4.19, respectively.

The thermal properties of polymers were determined by thermogravimetric analysis (TGA) in nitrogen atmosphere at a heating rate of 10 °C/min. The onset temperatures of TGA curves (T_d) were summarized in Table 1 also, which are in the range of 350.2~456.0 °C, indicating that all of the polymers have good thermal stability.

Absorption, Fluorescence and Electrochemical Properties of Polymers

The absorption, fluorescence emission and electrochemical properties of the polymers were measured by UV-vis absorption spectroscopy, fluorescence (FL) spectroscopy and cyclic voltammetry (CV), respectively.

Table 1.
Solubility, GPC results and thermal properties of the polymers.

Polymers	Solvents and Solubility ^a					GPC results ^b		T_d /°C
	THF	Chloroform	DCE	Acetone	DMF	Mw/ 10^3	MWD	
PBC	+	+	+	+	+	3.2	1.60	448.9
PBT	+	+	+	+	+	2.1	2.41	401.2
PBF	+	+	+	+	+	2.5	1.91	383.3
PQB	+	+	+	+	+	6.4	2.55	383.6
PQT	+	+	+	+	+	4.2	1.42	372.3
PQF	+	+	+	+	+	5.7	1.96	406.5
PNB	+	+	+	+	+	14.4	3.68	456.0
PNT	+	+	+	+	+	2.6	1.60	413.5
PNF	+	+	+	+	+	3.1	1.40	398.4
PBP	+	±	±	±	+	48.2	4.19	409.1
PBM	+	±	±	±	+	9.7	2.01	455.2
PQP	+	±	±	±	±	10.7	3.81	383.1
PQM	+	±	±	±	±	3.6	1.93	392.4
PNP	+	±	±	±	±	30.7	3.66	350.2
PNM	+	±	±	±	±	14.1	2.29	385.9
PQ	+	+	+	+	+	7.7	1.74	370.7
PQDN	+	+	+	+	+	7.3	2.14	398.6
PQTN	+	+	+	+	+	17.2	2.25	419.2

^a Solubility: + soluble, ± partly soluble;

^b Measured at 30 °C using THF as eluent and standard polystyrene as reference;

^c T_d : the onset temperatures on TGA curves.

Figure 1, Figure 2 and Figure 3 show the UV-vis absorption and FL spectra of PBC~PNF, PBP~PNM and PQ~PQTN in diluted THF solution, respectively. The absorption maximum ($\lambda_{\text{max}}^{\text{abs}}$), the onset absorption wavelength ($\lambda_{\text{onset}}^{\text{abs}}$) and the band gaps (E_g^{opt}) summarized from the UV-vis absorption spectra are listed in Table 2. Moreover, the excitation wavelength (λ_{ex}), the emission maximum ($\lambda_{\text{em}}^{\text{max}}$) and the full width at half maximum (FWHM) of the FL spectra are also listed in Table 2.

As shown in Table 2, all of the polymers show the absorption maximum in the range of 294~470 nm, the onset absorption wavelength in the range of 451~652 nm and the optic band gaps (E_g^{opt}) calculated from the formula: $E_g^{\text{opt}} = 1240/\lambda_{\text{onset}}^{\text{abs}}$ eV in the range of 1.90~2.75 eV. Comparing the nine polymers PBC~PNF, the band gap values of the polymers prepared from monomers **1**, **2** and **3** show the similar orders of thiophene-

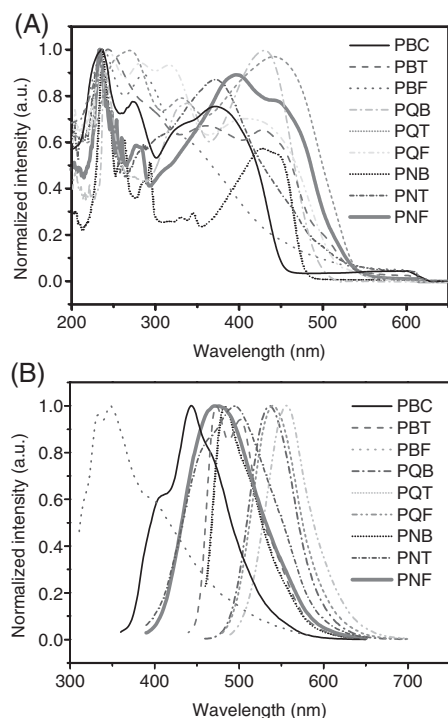


Figure 1. UV-vis absorption (A) and FL (B) spectra of polymers PBC~PNF diluted THF solutions.

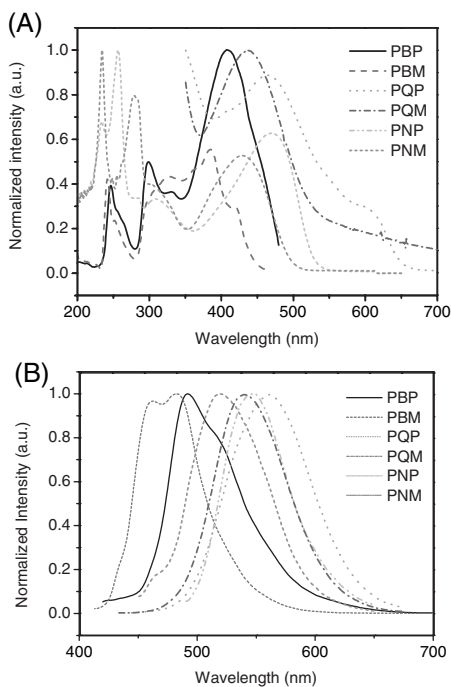


Figure 2. UV-vis absorption (A) and FL (B) spectra of polymers PBP~PNM diluted THF solution.

polymer > furan-polymer > phenylene-polymers, which indicate that the introduction of thiophene units into the conjugated polymers main chain to decrease their band gap is more effective than that of phenylene units or furan units. As for the six polymers PBP~PNM, the band gap of *p*-polymer is smaller than that of the corresponding *m*-polymers, which may be aroused from the effect of a quinoid structure formation. Furthermore, comparing PQDN and PQTN with PQ, 45 nm red shift of UV-vis absorption and 0.3 eV reduce of the band gap were observed, which indicate that the method of donor-acceptor alternant arrangement can effectively decrease the band gap of conjugated polymer.

When the polymer diluted solutions were excited by 289~477 nm light, they emitted light from purple to yellow color with the emission maximum in the range of 347~597 nm. Their FWHM is in the range of 59~119 nm, which are relatively narrow for the emissive spectra of conjugated

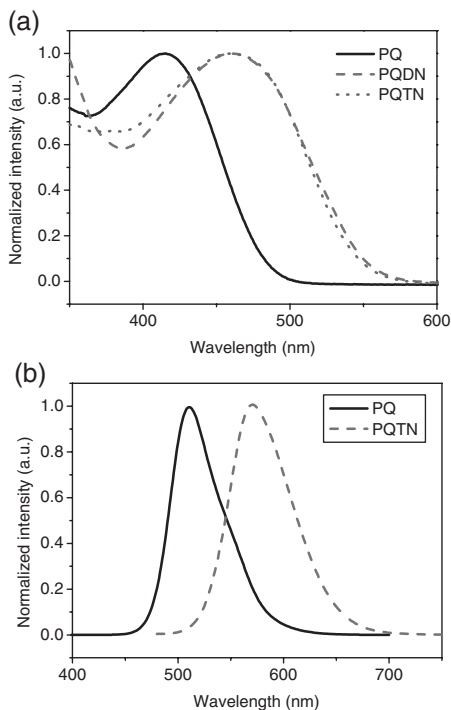


Figure 3. UV-vis absorption (a) and PL (b) spectra of polymers PQ~PQTN diluted THF solutions.

polymers. Especially for the six polymers PBP~PNM synthesized via Knoevenagel condensation, their FWHM are in the range of 64~76 nm.

The electrochemical property of the polymers was measured by cyclic voltammetry (CV) at room temperature. The CV curves of PQP, PQM and PQTN are shown in Figure 4. The onset oxidation potentials of PQP, PQM and PQT are 0.28 V, 0.48 V (vs Ag/AgCl) and 0.49 V (vs SCE), respectively, which suggested that the hole-injection ability of the polymers is very well. Especially, the redox reversibility at the anodic scan of PQP and PQM is also very well.

PLEDs Fabricated from the Polymers

For PLEDs fabrication, ITO-coated glass (15 Ω/\square) and magnesium, silver alloy (weight ratio: approximately 10:1) were used as the anode and cathode, respectively. *N,N'*-diphenyl-*N,N'*-bis(3-methylphenyl)-[1,1'-biphenyl]-4,4'-diamine (TPD) was used as hole-transporting and electron-blocking material. 2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline (BCP) and tris-(8-hydro-

Table 2. UV-vis absorption and FL data of the polymers.

Polymers	UV-vis ^a			FL ^b		
	$\lambda_{\text{max}}^{\text{abs}}/\text{nm}$	$\lambda_{\text{onset}}^{\text{abs}}/\text{nm}$	$E_g^{\text{opt}}/\text{eV}$	λ_{ex}	$\lambda_{\text{em}}^{\text{max}}$	FWHM/nm
PBC	375	451	2.75	347	444	96
PBT	427	531	2.34	428	502	105
PBF	294	458	2.71	289	348	68
PQB	429	491	2.52	441	537	61
PQT	441	538	2.30	471	557	59
PQF	407	516	2.40	461	541	63
PNB	428	478	2.59	447	483	59
PNT	397	520	2.38	372	495	119
PNF	374	507	2.45	374	471	99
PBP	407	487	2.55	409	492	67
PBM	385	444	2.79	381	482	64
PQP	465	652	1.90	477	597	76
PQM	437	521	2.38	452	578	66
PNP	470	526	2.36	474	544	65
PNM	429	495	2.51	431	519	76
PQ	415	501	2.48	365	510	56
PQDN	460	571	2.17			
PQTN	461	570	2.18	460	571	69

^a UV-vis: $\lambda_{\text{max}}^{\text{abs}}$, the absorption maximum; $\lambda_{\text{onset}}^{\text{abs}}$, the onset absorption wavelength; E_g^{opt} : calculated from the formula, $E_g^{\text{opt}} = 1240/\lambda_{\text{onset}}^{\text{abs}}$ eV.

^b FL: λ_{ex} , the excitation wavelength; $\lambda_{\text{em}}^{\text{max}}$, the emission maximum; FWHM: the full width at half maximum.

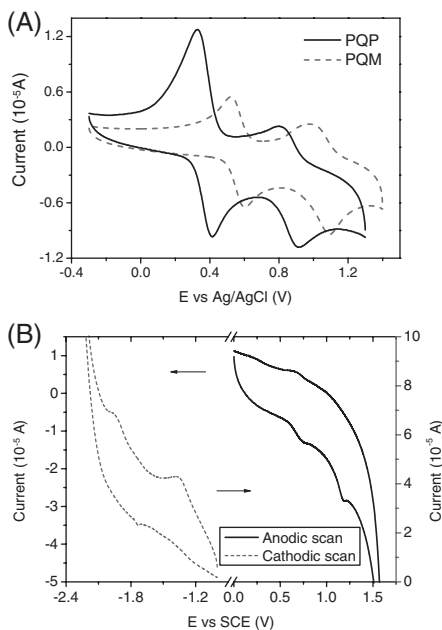


Figure 4. CV curves of the polymers. (A) PQM, PQP; (B) PQTN.

xyquinoline) aluminum (Alq) were used as electron-transporting materials and hole-blocking materials. Using these new polymers as light-emitting materials and combining suitable carrier transporting materials, the single-, double- and multi-layer configuration PLEDs devices were fabricated. The electroluminescent (EL) devices were not encapsulated, and all measurements were carried out in air and at the room temperature.

For example, using polymer PQB as the light-emitting material, the single-layer device (ITO/PQB/Mg/Mg:Ag) and multi-layer device (ITO/PQB/BCP/Alq/Mg/Mg:Ag) were fabricated. Figure 5 and Figure 6 show the current-brightness-voltage (I-B-V) and electroluminescence (EL) spectra of the single- and multi-layer device, respectively. When the forward bias voltage increased, both the current and brightness of them increased, which showed typical rectifying behavior. The highest brightness of the single-layer device was measured to be 307.7 cd/m², while the multi-layer device was measured as 1282.0 cd/m². Furthermore, the wavelength at the

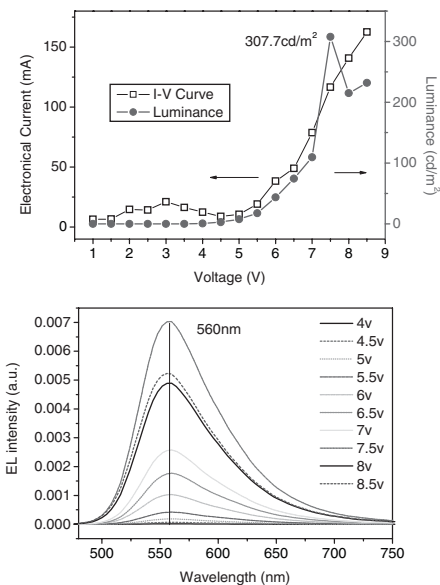


Figure 5. I-B-V characteristics and EL spectra of the device ITO/PQB/Mg/Mg:Ag.

maximum brightness of the single- and multi-layer device was 560 nm and 568 nm, respectively. With the increase of the forward bias voltage, the position of the central wavelength of their EL spectra did

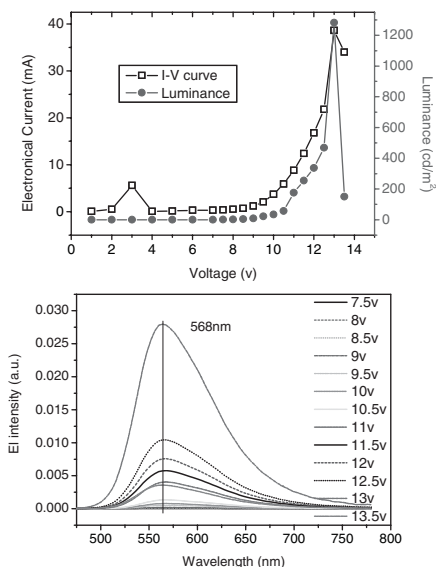


Figure 6. I-B-V characteristics and EL spectra of the device ITO/PQB/BCP/Alq/Mg/Mg:Ag.

Table 3.Data of PLEDs fabricated from conjugated polymers^a.

Polymers	Device configuration	V_{on}/V	$V_{max}V$	λ_{max}^{EL}/nm	$B_{max}/cd/m^2$
PBC	ITO/PBC/Mg/Mg:Ag	5.0	7.0	496	65.4
PBT	ITO/PBT/BCP/Alq/Mg/Mg:Ag	7.0	11.5	584	11.5
PQB	ITO/PQB/Mg/Mg:Ag	4.0	7.5	560	307.7
PQB	ITO/PQB/BCP/Alq/Mg/Mg:Ag	7.5	13.0	568	1282.0
PQF	ITO/PQF/BCP/Alq/Mg/Mg:Ag	10.0	12.0	588	8.1
PQT	ITO/PQF/BCP/Alq/Mg/Mg:Ag	10.5	12.5	612	71.8
PNB	TO/PNB/BCP/Mg/Mg:Ag	7.0	13.0	532	286.0
PNF	ITO/PNF/Mg/Mg:Ag	13.0	13.0	544	0.6
PNT	ITO/PNF/Mg/Mg:Ag	10.0	11.0	592	24.5
PQP	TO/PQP/Mg/Mg:Ag	6.0	8.0	656	11.6
PQP	ITO/PQP:TPD/BCP/Alq/Mg/Mg:Ag	11.0	15.0	660	20.2
PQM	TO/PQM/Mg/Mg:Ag	5.0	8.0	612	47.9
PQM	ITO/PQM:TPD/BCP/Alq/Mg/Mg:Ag	8.0	14.0	608	150.0
PQTN	ITO/PQTN/Mg:Ag	4.3	9.0	612	492.0
PQTN	ITO/PEDOT:PSS/PQTN/Mg:Ag	4.1	11.0	616	2434.0

^a V_{on} : the turn-on voltage; V_{max} : the voltage at the maximum brightness; λ_{max}^{EL} : the wavelength at the maximum brightness; B_{max} : the maximum brightness.

not change. As their EL spectra have the similar peak shape with the PL spectrum of PQB THF solution, we can infer that the EL spectra originated from the emission of PQB.

Except for PQB, the other polymers were also used as light-emitting materials to fabricate PLEDs devices. The electroluminescent properties of the devices were investigated and the data were summarized in Table 3. The turn-on voltage of these devices was in the range of 4.1–13.0 V, and the maximum brightness was in the range of 0.6–2434 cd/m^2 . In general, the maximum brightness of the multi-layer device was larger than that of the corresponding single-layer device; however, the turn-on voltage of the multi-layer device was higher than that of the corresponding single-layer device. Among these devices, the performance of the device fabricated from PQTN is the best, in which the maximum bright-

ness is 2434.0 cd/m^2 under an 11.0 V forward bias voltage.

Photovoltaic Devices Fabricated from the Polymers

ITO-coated glass and aluminum were chosen as anode and cathode, respectively. Using PNB as an active layer and copper phthalocyanine (CuPc) or *N,N'*-ditridecylperylene-3,4,9,10-tetracarboxylic diimide (PTCDI- C_{13}) as an additional layer, the single- and double-layer polymeric photovoltaic devices were fabricated. Photovoltaic properties of these devices were observed under a 35 W white arc lamp illumination. The main parameters are listed in Table 4. For the single-layer device, the short-circuit current and open circuit voltage are 0.005 mA/cm^2 and 0.26 V, respectively. Double-layered devices showed a much better open circuit voltages around 0.4–0.8 V, while the short-

Table 4.Characteristics of PNB photovoltaic devices^a.

Cell structure	J_{sc} (mA/cm^2)	V_{oc} (V)	FF
ITO/PNB (100 nm)/Al	0.005	0.26	0.28
ITO/CuPc (45 nm)/PNB (40 nm)/Al	0.006	0.80	0.19
ITO/PNB (50 nm)/CuPc (45 nm)/Al	0.011	0.60	0.18
ITO/PTCDI- C_{13} (20 nm)/PNB(70 nm)/Al	0.00873	0.65	0.21
ITO/PNB (70 nm)/PTCDI- C_{13} (20 nm)/Al	0.00944	0.48	0.16

^a J_{sc} : the short-circuit current; V_{oc} : the open-circuit voltage; FF: fill factor.

circuit currents were also enhanced. Among these photovoltaic devices, an ITO/PNB/PTCDI- C_{13} /Al device showed a good performance, which was estimated to have external quantum efficiency (EQE) around 1% at 330 nm.

Conclusion

Aiming to prepare novel optoelectronic materials, three series of conjugated polymers containing carbazole, phenothiazine and triphenylamine units in the main chain were designed and synthesized via Wittig, Knoevenagel and Heck condensations. These polymers have good solubility in common organic solvents such as THF, chloroform, etc. The polymers also have high thermal stability and good hole-injection ability. All of the polymers showed an absorption maximum in the range of 294~470 nm, the onset absorption wavelength in the range of 451~652 nm and the band gaps in the range of 1.90~2.75 eV. When the polymer solutions in THF were excited by 289~477 nm light, they emitted light from purple to yellow with the emission maximum in the range of 347~597 nm. Using these polymers as light-emitting materials or photovoltaic materials, single-, double- and multi-layer light-emitting or photovoltaic devices were fabricated. Among these PLEDs devices, the performance of the device composed of ITO/PEDOT:PSS/PQTN/Mg:Ag is the best, in which the maximum brightness is 2434.0 cd/m² under an 11.0 V forward bias voltage. As for photovoltaic devices, an ITO/PNB/PTCDI- C_{13} /Al device showed the best performance, which was estimated to have external quantum efficiency (EQE) around 1% at 330 nm. In a word, the preliminary experimental results indicate that these conjugated polymers may be good light-emitting materials for PLEDs; while for photovoltaic application, their

absorption spectra need to be improved to match the solar illumination.

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