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Electroluminescence from Substituted Poly(thiophenes): From Blue to Near-Infrared

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ABSTRACT: We report a systematic approach to the control of the conjugation length along the poly(thiophene) backbone. The planarity of the main chain can be permanently modified by altering the pattern of substitution and character of the substituents on the poly(thiophene) chain, and the conjugation length is thus modified. We obtain blue, green, orange, red, and near-infrared electroluminescence from four chemically distinct poly(thiophenes). The external quantum efficiencies are in the range of 0.01–0.6%.

Introduction

The use of conjugated polymers in electroluminescent polymer diodes has expanded greatly since the original report of electroluminescence in poly(*p*-phenylenevinylene).[1] Most current work is focused on poly(*p*-phenylenevinylenes), PPV,[2–6] materials which may be obtained from precursor polymers as well as in the form of soluble polymers with side chains. These have been reported to give blue,[7] green,[8] yellow,[9] and red[5] electroluminescence. In different poly(*p*-phenylene), PPP, polymers, blue[10] and yellow-green[11] electroluminescence has been reported.

We have approached the field of polymer light-emitting diodes, PLED's, using poly(thiophene) as our main system. Poly(thiophenes) of various kinds have been studied in PLED's.[12–16] This is a polymer system with great chemical variability through substitution at the 3- and 4-positions. The polymers can easily be obtained by 2,5-coupling of the substituted monomer via oxidative polymerization. Ease of synthesis through oxidative coupling however necessitates extensive purification of the resulting polymers. We have tried to tune the electroluminescence from different poly(thiophenes) through the whole visible spectrum. Partial coverage of the visible spectrum using poly(thiophenes) has been accomplished using block copolymers between thiophene oligomers of different lengths and nonconjugated blocks.[16] The color of the electroluminescence has also been tuned using different lengths of unsubstituted thiophene blocks between head-to-head coupled diads of 3-octylthiophenes.[15]

We have recently reported[17] the lowest energy for optical transitions in any 3-substituted poly(thiophene) films in the undoped state. The absorption maximum for films of poly(3-(4-octylphenyl)thiophene) occurs at 602 nm, and this is understood to be a consequence of the high regularity and order in the polymer.[17] An absorption maximum as low as 315 nm has been reported for poly(3,4-dialkylthiophene).[15,18] It is thus well established that changes of approximately 300 nm in absorption maximum can be obtained in the poly(thiophene) system by judicious design of the polymer geometry and choice of the polymerization method. Of course, if the conjugation length of the polymer were reduced to the monomer length, the absorption would be expected to be in the ultraviolet range, where the absorption maximum for the monomer is found at 231 nm. Taking into account that the Stokes' shift will move the emission closer to the visible region, we should thus be able to span the full visible range with electroluminescence from poly(thiophenes).

The dihedral angle and thus the π-orbital overlap between adjacent thiophene rings along the polymer backbone determine the conjugation along the polymer chain. Short conjugation gives a blue-shifted emission and long conjugation gives a red-shifted emission. We have chosen to modify the conjugation length along the polymer backbone by adding different substituents on the repeating unit. If the repeating unit is kept small, the ease of synthesis increases. Substituents in the 3- or both the 3- and 4-positions on the thiophene rings give steric interactions between the side groups and the adjacent thiophene ring and/or the side group attached to the adjacent thiophene ring. Large substituents give a large dihedral angle between the rings, and short conjugation along the polymer backbone is achieved. Using different amounts and sizes of the substituents on the repeating unit, it should be possible to modify the conjugation. The regioregularity is also important for the conjugation along the polymer backbone. When monosubstituted thiophenes are polymerized, head-to-head and head-to-tail coupling can occur. A large amount of head-to-head coupling gives shorter conjugation and blue-shifted emission compared to a regioregular head-to-tail coupled polymer.

A recent study of photoluminescence in poly(3-hexylthiophenes)[19] has indicated that the quantum yield for photoluminescence in the solid state is enhanced by the presence of head-to-head coupling in the polymer chain but that it decreases in solution. Given the correlation between photoluminescence and electroluminescence, this might be beneficial for good efficiency in blue-shifted PLED's.

Based on these facts, we have chosen to prepare and use the polymers shown in Figure 1 to achieve electroluminescence over the whole visible spectrum. PLED's prepared from polymers I–IV show electroluminescence

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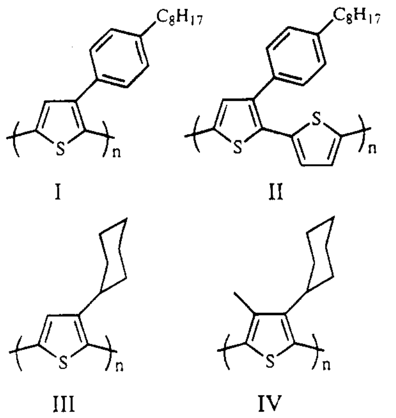

Figure 1. Structures of polymers **I**–**IV**.

ranging from the blue into the near-infrared, with green, orange, and red as intermediate steps. In some of these PLED's, we have incorporated a so-called electron injection layer to enhance the efficiency. We here report the blue diode, others are presented elsewhere.[20–22] We also discuss the structure-related properties of the polymers such as melting point, conjugation length, oxidation potentials, turn-on voltage, and color of the electroluminescence.

Experimental Section

The synthesis and characterization of polymers **I** and **III** are reported elsewhere.[17,22] NMR spectra were recorded using a Varian XL-400 spectrometer, and mass spectra were recorded by a VG ZabSpec. The molecular weights were determined by a Waters 150-CV using SDVB columns at 25 °C with THF as a solvent and polystyrene standards for calibration.

3-Cyclohexyl-4-methylthiophene was prepared by reacting 3-bromo-4-methylthiophene[23] and the Grignard reagent from cyclohexyl bromide.[24] After flash chromatography in pentane, the yield was 65%.

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.88 (s, 2H), 2.49 (m, 1H), 2.20 (s, 3H), 1.90 (m, 2H), 1.82 (m, 2H), 1.75 (m, 1H), 1.33 (m, 5H).

MS (EI) m/z 181.102 (M^+), calculated for $C_{11}H_{16}S$, 180.097.

3-(4-Octylphenyl)-2,2′-bithiophene was synthesized from 2-bromo-3-(4-octylphenyl)thiophene and the Grignard reagent from 2-bromothiophene as described.[24] 2-Bromo-3-(4-octylphenyl)thiophene was prepared by reacting NBS in DMF with 3-(4-octylphenyl)thiophene[17] as described in ref 25. The yield of 3-(4-octylphenyl)-2,2′-bithiophene was 75% after flash chromatography in pentane.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24 (m, 3H), 7.15 (m, 3H), 7.06 (d, 1H, J = 5.2 Hz), 6.97 (dd, 1H, J = 1.2, 4.0 Hz), 6.92 (dd, 1H, J = 3.6, 5.2 Hz), 2.61 (t, 2H, J = 7.8 Hz), 1.65 (m, 2H), 1.27 (m, 10H), 0.88 (t, 3H, J = 6.8 Hz).

MS (EI) m/z 354.1480 (M^+); calculated for $C_{22}H_{26}S_2$, 354.1476.

Poly(3-cyclohexyl-4-methylthiophene) (**IV**) was prepared by adding a slurry of $FeCl_3$ and chloroform to 3-cyclohexyl-4-methylthiophene dissolved in chloroform over a period of 6 h as described.[17] The mixture was then stirred for a further 18 h. The polymer was then carefully dedoped with ammonia and EDTA.[17] The yield of soluble polymer was 65%, and the molecular weight was determined to be $\bar{M}_n$ = 26 000 and $\bar{M}_w$ = 72 000.

Poly(3-(4-octylphenyl)-2,2′-bithiophene) (**II**) was polymerised and dedoped as described for poly(3-cyclohexyl-4-methylthiophene). The yield was 84% and the molecular weight was M_n = 9000 and M_w = 82000.

Results and Discussion

Solubility and thermal stability are ideal properties for conjugated polymers used in PLED's. The solubility is needed for the preparation of the diodes. All the polymers dissolve in hot chloroform. Good thermal stability of the polymer is needed because of the heat generated when current is passed through the diode. Melting of the polymer film in the diode is clearly deleterious. The melting intervals of the polymers measured by a melting point microscope on powders of the polymers are listed in Table 1. The melting intervals are unusually high for the polymers compared to those of normal poly(3-alkylthiophenes).[26] The higher melting interval depends on the use of more rigid side groups than normal straight alkyl chains.

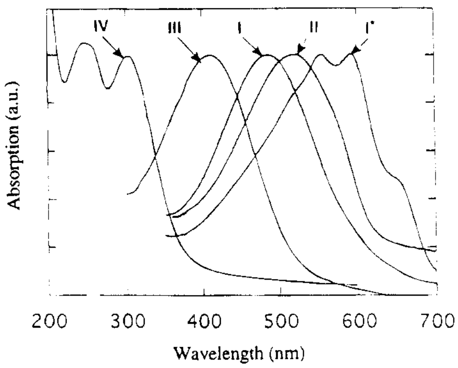

Figure 2. Optical absorption spectra of polymers **I**–**IV**. Absorption spectra were taken on spin-coated thin films on glass slides or quartz (for **IV**) with a Perkin-Elmer 19 spectrophotometer.

Table 1. Melting Interval (MI), Molecular Weight, and Absorption Maximum (E_{max}) for Polymers I–IV

polymer	MI, °C	$\bar{M}_n$	$\bar{M}_w$	E_{max}, nm (eV)
I*	>300			594 (2.09)
I		23 000	52 000	485 (2.56)
II	280–300	9000	82 000	518 (2.39)
III	195–215	6000	56 000	413 (3.00)
IV	>300	26 000	72 000	305 (4.06)

Thin films of polymers **IV** to **I** show optical absorption maxima spanning over a range of 289 nm, from 305 to 594 nm. This is demonstrated by optical absorption data given in Figure 2 and Table 1. The color of thin spin-coated films of the polymers are blue-red (**I***), brown-red (**I**), red (**II**), yellow (**III**), and colorless (**IV**). The two forms of **I** are obtained by spin-coating **I** from chloroform solution. The pristine film is labeled **I**. Upon thermal treatment of this film, an irreversible change to a new structure, labeled **I***, is obtained. This can also be achieved by treating the spin-coated film with saturated chloroform vapor at room temperature, giving a slightly better order in the film.[17] The spin-coated film is amorphous and, when the film is heated or treated with chloroform vapor, the crystallinity is increased, as observed in X-ray studies of spin-coated films.[27] This is a result of the high regioregularity in polymer **I**; the head-to-tail content is 94 ± 2%, as determined with ^{1}H NMR.[17,20] We can clearly interpret the blue shift in the optical absorption as being due to a decreasing conjugation length of the polymers along the sequence **I**–**IV**. This is in itself sufficient to claim success in modifying the conjugation length and optical properties of poly(thiophene) chains.

FT Raman Spectroscopy. Further verification of the control of conjugation length is obtained from Raman spectroscopy. The experimentally obtained Raman spectra of polymer powder (Figure 3) show the dominating peak assigned to the symmetrical C=C stretching vibration. The shift in position (1442–1506 cm^{-1}) from **I** to **IV** represents the decrease in conjuga-

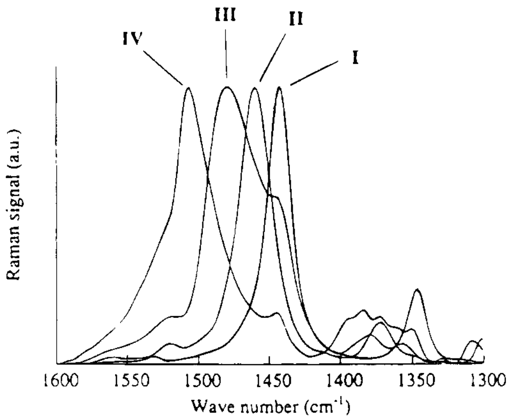

Figure 3. Raman spectra of polymers **I**–**IV** in powder form. The spectra were taken with a Bruker IFS 66 FT-IR spectrometer in the Raman mode and have been normalized; the relative intensities of the spectra are **I**:**II**:**III**:**IV** = 18:43:12:1. The radiation power of the laser used as exciting light source was 100 mW, the exciting wavelength was 1064 nm, and the resolution of the equipment was 4 cm^{-1}. All measurements were carried out at room temperature.

tion length[28] caused by the side groups attached to the backbone. The broadening of the peak and the growth of the shoulder on the high-energy side are also related to the decrease in conjugation length. These vibrations have also been interpreted using the so-called "effective conjugation coordinate", which is a linear combination of the C—C and C=C skeletal bonds involved in the delocalization path[29–31]and can be understood as the dispersion of the Raman line with the conjugation length in this interpretation.[28] The shoulder on the low-energy side of the central peak observed for **III** and **IV** can be assigned to the cyclohexyl groups in these materials. The powder used of polymer **I** can be regarded as being a mixture of ordered and nonordered polymer.

Electrochemical Spectroscopy. The energy level of the electronic states into which holes are injected can be studied by electrochemical spectroscopy. Electrochemical studies of the polymer were carried out in $LiClO_4$ in acetonitrile or propylene carbonate. Both these electrolytes could be used for studies of the oxidation (hole injection) of the polymers as reported in Figure 4. We were not able to obtain any reduction processes (electron injection) of the polymers down to −2.0 V vs Ag/AgCl in propylene carbonate.

The polymers were adsorbed onto a Pt wire used as the working electrode. The peak currents were linearly dependent on the sweep rate, consistent with the electrochemical behavior of a surface-bound redox species. The first sweep is in most cases different from that of consecutive sweeps; we have systematically used the first sweep for data extraction, as the decay of the material observed is not relevant when characterizing the band edges of the materials in their undoped state. In the case of **IV**, the cyclic voltammogram was highly irreversible.

The presence of more than one peak in the poly(thiophene) voltammograms is quite general and has been interpreted as being due to differences in conjugation length, different conformers, or polarons followed by bipolaron formation.[32] These voltammograms were evaluated using the potential of the first oxidation current peak (Table 2). We clearly observe that a lower wavelength of the absorption maximum also usually leads to a higher oxidation potential. In Figure 5, the potential for the oxidation peak current is plotted, i.e., the energy position of the HOMO band/orbitals in combination with the HOMO−LUMO gap from optical absorption. We have chosen to plot this gap as the energy of maximum optical absorption (E_{max} in Table 1) as expected from an ideal 1-D electronic system,[33] since the convolution of the 1-D dispersed density of states of the valence and conduction energy bands in an optical absorption event gives a maximum at the gap energy. The conversion from the electrochemical potential axis to the vacuum scale has been done assuming the normal hydrogen electrode (NHE) to be at 4.5 eV versus vacuum[34] and including the potential difference of +0.22 V between Ag/AgCl and NHE. The shift of the oxidation potential from **I** to **IV** is ≈0.4 V. At the same

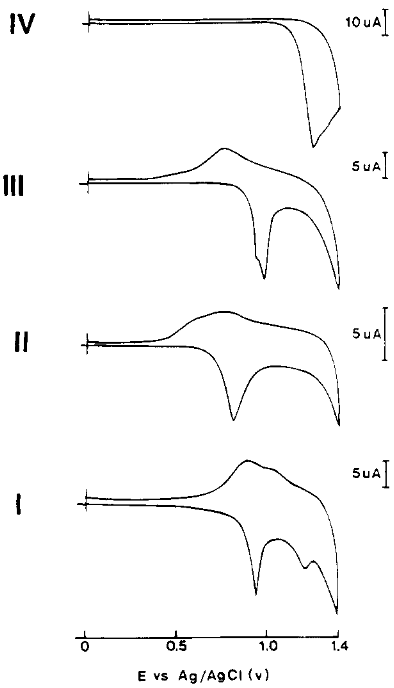

Figure 4. Electrochemical spectroscopy of polymers in a 0.5 M $LiClO_4$ electrolyte in acetonitrile. The cyclic voltammograms were all taken at 100 mV/s, and samples were prepared by adsorbing the polymers onto a Pt rod. This was used as the working electrode in a three-electrode, single-compartment electrochemical cell with a Pt counter electrode and a Ag/AgCl reference electrode. The cell was purged with argon.

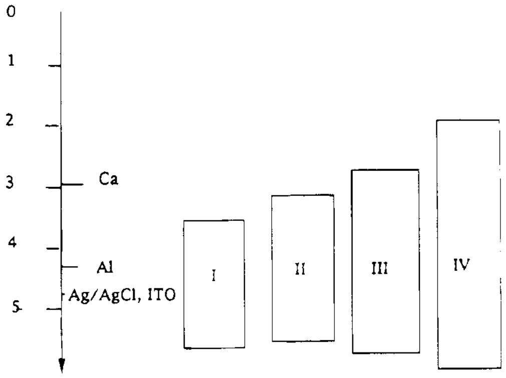

Figure 5. Band edges and energy gaps as deduced from optical and electrochemical spectroscopy.

Table 2. Oxidation Potential (E_{peak}) and the HOMO Position for Polymers I–IV

polymer	E_{peak}, (V vs Ag/AgCl)	HOMO, eV vs vacuum
I*	0.94	5.66
II	0.80	5.52
III	1.0	5.72
IV	1.27	5.99

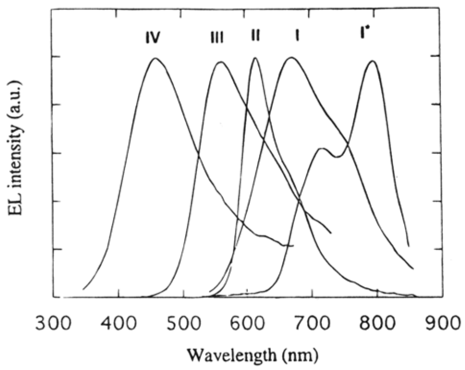

Figure 6. Electroluminescence spectra of polymers **I**–**IV**. Luminescence spectra were taken with an Oriel Instaspec 1B diode array spectrometer. All these diodes were built using ITO as the hole-injecting electrode and Ca/Al as the electron-injecting contact. The **IV** diode also contains a layer of PBD, but this does not contribute to the electroluminescence.

Table 3. Turn-On Voltage, External Quantum Efficiency, and Maximum of the Electroluminescence (EL_{max}) of Polymers I–IV in PLED's[a]

polymer and PLED structure	turn-on voltage,[b] V	external quantum eff, %	EL_{max}, nm (eV)
I*	1.4	0.3 (6 V)	800 (1.55)
I	1.4	0.3 (6 V)	670 (1.85)
II	1.6	0.1 (8 V)	610 (2.03)
III	2.4	0.01 (7 V)	555 (2.25)
IV		<0.0001 (4 V)	
IV/PBD	7	0.6 (25 V)	460 (2.69)

[a] All diodes were prepared with ITO and Ca/Al as the hole-injecting and electron-injecting contacts, respectively. [b] The detector signal rises above the noise level of $\approx 10^7$ photons/s.

time, the energy gap increases by 2 eV. We thus observe that the LUMO level—or the electron affinity—decreases more than the HOMO level—or the work function—increases. This is in marked disagreement with theoretical predictions from quantum chemical calculations on the different oligomers of thiophene.[35] The evaluation of band edges from electrochemical data is still being debated[32] and the absolute position of the energy levels is open to discussion. We may however conclude that the barrier for electron injection should increase in the polymer sequence **I**–**IV**, resulting in higher operating voltages and, possibly, lower quantum efficiencies in the PLED's. The diodes prepared from the polymers have an increasing turn-on voltage with decreasing wavelength of the absorption maximum of the polymers; see Tables 3 and 1. Polymer **I** has been considered to be in the ordered form (**I***).

In addition to the PLED's from polymers **I**–**III** giving near-infrared and red (**I***, **I**),[20] orange (**II**),[21] and green (**III**)[22] colors, we can now add the blue color from polymer **IV** (Figure 6). This is the first time this wide electroluminescence has been described using modified conjugated polymers with the same polymer backbone or using different types of conjugated polymers. To our knowledge, the shortest wavelength of the electroluminescence maximum from PLED's reported so far is 415 nm[16] and the longest wavelength of the maximum is 710 nm.[5] Our PLED's have electroluminescence maxima spanning from 460 to 800 nm.

The structure and design of the PLED prepared from polymer **IV** are shown in Figure 7. Without the electron transport layer, we observed only weak electroluminescence ($\approx 10^{-4}\%$). Addition of the 2-(4-biphenylyl)-5-(4-*tert*-butylphenyl)-1,3,4-oxadiazole,[36] PBD, layer enhanced the quantum yield by a factor of $\approx 10^3$. The electroluminescence spectrum of the (**IV**/PBD) PLED is given in Figure 6. We observe blue emission centered on the peak at 460 nm, almost identical to that of the photoluminescence of **IV**. There is no contribution from the PBD layer nor from indirect transitions at the interface as observed in **II**/PBD PLED's.[21] The $I(V)$ curve of the blue PLED can be seen in Figure 8. External quantum efficiencies of 0.6% were obtained, which is an extraordinarily high efficiency for a blue PLED compared to reported data for other blue PLED's.[7,11,37–42]

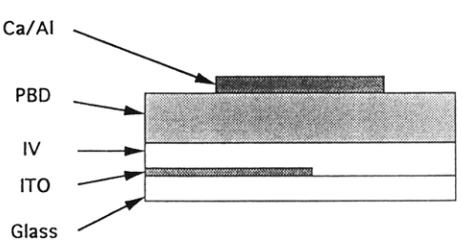

Figure 7. Construction of the blue PLED. The blue PLED was constructed by spin-coating **IV** from a chloroform solution (8 mg/mL) in a 40–60 nm thick layer on patterned ITO electrodes, followed by thermal evaporation of PBD (thickness 100–130 nm) on top of the polymer. A thin layer of calcium overcoated with a thick layer of aluminum was used as the electron-injecting contact and evaporated onto the film at a pressure less than 4×10^{-7} Torr. The aluminum was used to protect the calcium from the ambient atmosphere. Electrical and radiometric characterizations were done with a Keithley 617 electrometer and a Hamamatsu silicon photodiode (1010BR) together with a Keithley 485 picoampere meter. The quantum efficiency, emitted electrons per injected electrons, was estimated using this photodiode. We have verified by experiment that Lambert emission of light is an almost correct assumption and have made this assumption when evaluating the quantum efficiency. The spectral response of the photodiode was taken into account.

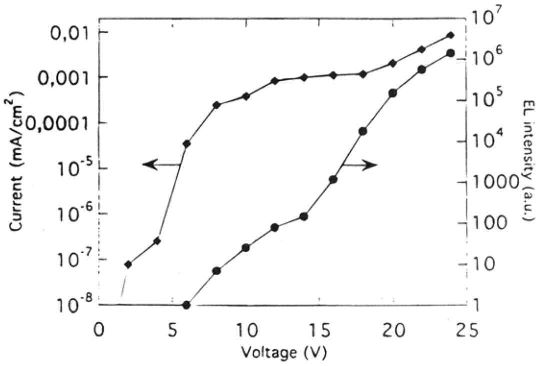

Figure 8. Current–voltage characteristics of the blue PLED.

We have reported elsewhere that diodes prepared from blends of polymers **I**/**III** and **II**/**IV** show voltage-controlled color changes.[43] This is interpreted as being due to the fact that polymer blends phase separate, and the diode can then best be described as consisting of many small parallel diodes. The small diodes containing different polymers have different turn-on voltages as seen in Table 3. At a low voltage applied over the diode, only one polymer emit light, and at a high voltage, both polymers emits light. Using a suitable ratio of the polymers, different colors can be achieved. This principle of self-organization in the polymer blends, giving a new technical function, heralds many more developments in the field of polymer electronics.

Conclusion

We have prepared different substituted poly(thiophenes) with optical absorption maxima spanning from 594 to 305 nm, using steric hindrance of side-group substituents to modify the dihedral angle between the thiophene rings in the main chain. Raman spectroscopy verifies the change in conjugation in the main chain, and electrochemical spectroscopy verifies a change in the energy position of the HOMO. PLED's prepared from the polymers prepared have blue, green, orange, red, and near-infrared electroluminescence. In conclusion, steric hindrance offers a viable route for the design of poly(thiophenes) with widely different emission.

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