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Synthesis and Characterization of Highly Soluble Phenyl-Substituted Poly(*p*-phenylenevinylenes)

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ABSTRACT: We present the synthesis and characterization of soluble, high molecular weight phenyl-substituted poly(*p*-phenylenevinylenes). The studied polymers are poly(2-(2′,5′-bis(2″-ethylhexyloxy)-phenyl)-1,4-phenylenevinylene) (BEHP−PPV) and copolymers of BEHP−PPV and poly(2-methoxy-5-(2′-ethylhexyloxy)-1,4-phenylenevinylene) (MEH−PPV). Their photoluminescence and electroluminescence have been examined as well as their stability to air and light. The polymers emitted green and yellow light. Stability measurements showed increased stability compared to some previously known substituted PPVs.

Introduction

Since the discovery of electroluminescence (EL) in poly(*p*-phenylenevinylene) (PPV),[1] the interest in conjugated polymers has been focused on their use as the active substance in electronic devices. Today, PPV and its derivatives are still the most popular class of conjugated polymers used as the active material in light-emitting diodes (LEDs). Other important polymers with similar properties are poly(fluorene) (PF),[2,3] poly(*p*-phenylene) (PPP),[4,5] and poly(thiophene) (PT)[6] and their derivatives. Recently, several new applications of conjugated polymers such as their use in photovoltaic diodes,[7,8] in photodetectors and image sensors,[9] in light-emitting electrochemical cells,[10,11] and for laser devices[12−14] have initiated new research. Fully conjugated polymers without substituents are insoluble and not processable once formed. However, processability of aromatic conjugated polymers can be achieved by the introduction of flexible side chains on the polymer backbone. Flexible side chains also give rise to sterical hindrance along the polymer backbone. By the introduction of proper side chains, one can control the effective conjugation length and thus the color of the polymers.[15,16] A negative property of PPV derivatives with flexible side chains, such as poly(2-butyl-5-(2′-ethylhexyl)-1,4-phenylenevinylene) (BUEH−PPV)[17] and poly(2-methoxy-5-(2′-ethylhexyloxy)-1,4-phenylenevinylene) (MEH−PPV),[18] is their sensitivity to a combination of air and light. The low stability of some of the polymers requires careful handling during synthesis and of the device preparation and limits their use for commercial application. Some high molecular weight PPV derivatives are also difficult to dissolve in common solvents such as chloroform and THF.

Here we report the synthesis and characterization of a stable, highly soluble, and high molecular weight phenyl-substituted PPV derivative, BEHP−PPV (Figure 1).[19] The flexible side chain in BEHP−PPV was designed to give the polymer high solubility and high stability against photooxidation. It was also designed to look like the side chain in poly(3-(2′,5′-dioctylphenyl)-thiophene) (PDOPT),[20] the poly(thiophene) with the highest known photoluminescence efficiency (η_{PL}) in the solid state. The high η_{PL} is believed to be due to the well-separated conjugated polymer chains.[6] Recently, Spreitzer et al. reported that PPVs with side chains similar to BEHP−PPV were highly efficient as the active substance in LEDs.[21] Copolymers with BEHP−PPV and MEH−PPV were synthesized and studied to see whether energy transfer from the wider band-gap portion of the polymer (BEHP−PPV) to the more narrow band-gap portion of the polymer (MEH−PPV) can occur and further increase the η_{PL}, reducing the nonradiative decay from the excited states.

Experimental Section

Instrumentation. NMR spectra were recorded on a Varian XL 400 MHz spectrometer, and mass spectra were recorded using a VG ZabSpec. Molecular weights were determined by size-exclusion chromatography (SEC) by a Waters 150 CV using SDVB columns at 25 °C. The calibration was made with a series of monodispersed polystyrene standards in THF. UV−vis spectra were recorded on a Varian, Cary 4 instrument. The photoluminescence (PL) spectra were measured by exciting the polymer samples with monochromatic light from a tungsten lamp, and the emission was measured with an Oriel Instaspec IV diode matrix spectrometer. An integrating sphere made by Labsphere was used to measure the PL efficiency. The electroluminescence (EL) test devices were made with ITO/glass substrate of 15 mm × 15 mm. The active area of each test device was ~0.1 cm^2. The film thicknesses were ~1000 Å for the EL layer and ~400 Å for the PVK layer. The EL intensity was measured with a silicon photodiode, and the quantum efficiency was determined by comparison with devices measured in an integrating sphere.

Materials. All starting materials are commercially available from Aldrich or Fluka. Diethyl ether was distilled over sodium and benzophenone, and dioxane was distilled over

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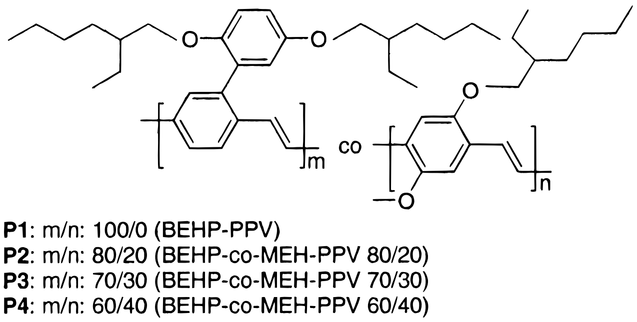

Figure 1. Chemical structure of the prepared polymers.

sodium. DMF was dried by azeotropic distillation with benzene.

Synthesis. 1,4-Bis(2′-ethylhexyloxy)benzene (1). To a solution of K_2CO_3 (93.0 g, 673 mmol) and hydroquinone (12.5 g, 114 mmol) in freshly distilled DMF (250 mL) was added dropwise 2-ethylhexyl bromide (43.9 g, 227 mmol). The mixture was heated to 100 °C for 18 h. The solids were filtered off, and the mother liquor was treated with 1 M HCl and pentane. The water phase was then extracted two more times with pentane, and the combined organic extracts were washed successively with 1 M HCl, three portions of 3 M NaOH, and water. The pentane solution was dried over Na_2SO_4 and evaporated to afford 18.9 g (50%) of 1,4-bis(2′-ethylhexyloxy)benzene. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.91 (m, 12 H), 1.3−1.5 (m, 16 H), 1.69 (m, 2 H), 3.78 (d, 4 H), 6.80 (s, 4 H). HRMS: Calcd for $C_{22}H_{38}O_2$: 334.287. Found: 334.292.

2-Bromo-1,4-bis(2′-ethylhexyloxy)benzene (2). A solution of NBS (9.12 g, 51.2 mmol) in freshly distilled DMF (100 mL) was added to a solution of **1** (17.5 g, 52.3 mmol) in freshly distilled DMF (150 mL) with stirring at room temperature. The solution was kept at room temperature with stirring for 8 h. The mixture was then added to 1 M HCl and pentane, and the two phases were separated. The water phase was extracted with three portions of pentane, and the resulting organic extracts were combined and washed with 1 M HCl, sodium metabisulfite (10%), and water. The organic extracts were dried over Na_2SO_4, evaporated, and purified by column chromatography (silica gel, pentane/dichloromethane (10/1) as eluent) to yield 13.1 g (62%) of 2-bromo-1,4-bis(2′-ethylhexyloxy)benzene. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.91 (m, 12 H), 1.3−1.5 (m, 16 H), 1.70 (m, 2 H), 3.76 (d, 2 H), 3.83 (d, 2 H), 6.79 (d, 1 H), 6.81 (s, 1 H), 7.11 (d, 1 H). HRMS: Calcd for $C_{22}H_{37}BrO_2$: 412.198. Found: 412.175.

2-(*p*-Xylene)boronic Acid. To a solution of absolute diethyl ether (50 mL) and *n*-BuLi (50 mL, 1.6 M in hexane, 80 mmol) under nitrogen was added dropwise 2-bromo-*p*-xylene (14.8 g, 80 mmol) with stirring at −70 °C. The solution was kept at −70 °C for 30 min and then allowed to warm to room temperature for 90 min. The yellowish solution was then cooled to −70 °C, and tributyl borate (27 mL, 100 mmol) was added. After 45 min at −70 °C and 2 h at room temperature the reaction was quenched by the addition of water. The two phases were separated, and the water phase was extracted twice with diethyl ether. The resulting organic phases were combined and extracted with three portions of 1 M NaOH, and the combined alkaline water extracts were treated with 37% HCl. The acidified water phase was extracted three times with ether, and the resulting organic extracts were combined and dried over Na_2SO_4 and evaporated to yield the boronic acid (8.6 g) as a white solid, which was used without further purification.

2-(1′,4′-Bis(2″-ethylhexyloxy)benzene)-*p*-xylene (3). To a stirred solution of compound **2** (12.5 g, 30 mmol) in DME (140 mL) was added $Pd[P(Ph)_3]_4$ (0.52 g, 0.45 mmol) at room temperature under nitrogen. After 10 min at room temperature 2-(*p*-xylene)boronic acid (7.0 g, 47 mmol) was added followed by 1 M $NaHCO_3$ (110 mL), and the mixture was then refluxed for 4 h. The catalyst was filtered off, and the phases were separated. The water phase was extracted twice with diethyl ether, and the resulting organic extracts were combined and washed twice with water, dried over Na_2SO_4, and evaporated. The resulting oil was purified by column chromatography (silica gel, pentane/dichloromethane (10/1) as eluent) to yield 11.7 g (88%) of 2-(1′,4′-bis(2″-ethylhexyloxy)benzene)-*p*-xylene. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.75 (t, 3 H), 0.81 (t, 3 H), 0.91 (m, 6 H), 1.1−1.2 (m, 7 H), 1.3−1.5 (m, 8 H), 1.70 (m, 1 H), 2.11 (s, 3 H), 2.32 (s, 3 H), 3.66 (br s, 2 H), 3.80 (d, 2 H), 6.73 (d, 1 H), 6.84 (d, 1 H), 6.86 (s, 1 H), 6.99 (s, 1 H), 7.06 (dd, 2 H). HRMS: Calcd for $C_{36}H_{46}O_2$: 438.350. Found: 438.348.

2-(1′,4′-Bis(2″-ethylhexyloxy)benzene)-1,4-bis(bromomethyl)benzene (4). A mixture of **3** (3.01 g, 6.86 mmol), NBS (2.44 g, 13.7 mmol), and benzoyl peroxide (50 mg, 0.21 mmol) in CCl_4 (75 mL) was refluxed and stirred until all white solid (succinimide) was on top of the solution (approximately 90 min). The succinimide was filtered off, and the mother liquor was washed with sodium metabisulfite (10%) and water, dried over Na_2SO_4, and then evaporated. The resulting oil was purified by column chromatography (silica gel, pentane/ethyl acetate (100/1) as eluent) to yield 3.2 g (78%) of 2-(1′,4′-bis(2″-ethylhexyloxy)benzene)-1,4-bis(bromomethyl)benzene. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.72 (t, 3 H), 0.80 (t, 3 H), 0.91 (m, 6 H), 1.1−0.1.2 (m, 7 H), 1.3−1.5 (m, 8 H), 1.71 (m, 1 H), 3.68 (dd, 2 H), 3.83 (d, 2 H), 4.39 (dd, 2 H), 4.48 (s, 2 H), 6.87 (s, 1 H), 6.89 (s, 2 H), 7.24 (d, 1 H), 7.36 (dd, 1 H), 7.49 (d, 1 H). HRMS: Calcd for $C_{30}H_{44}Br_2O_2$: 596.169. Found: 596.160.

1,4-Bis(bromomethyl)-2-(2′-ethylhexyloxy)-5-methoxybenzene (5). The synthesis of this compound has been described earlier.[17,18]

Polymer Synthesis. A representative procedure for the preparation and isolation of polymers **P1**−**P4** follows.

Polymer P3 from Monomers 4 and 5. All solutions were kept under nitrogen. A stirred solution of **4** (0.537 g, 0.900 mmol) and **5** (0.163 g, 0.386 mmol) in xylene (30 mL) was heated to reflux. Then potassium *tert*-butoxide (4.1 mL, 0.8 M in dioxane, 3.3 mmol) was added under 2 min, and the resulting mixture was refluxed for another 4 h. Methanol was added dropwise, and the precipitate was collected, washed with methanol, and then stirred with methanol for 30 min. The crude polymer was filtered off, washed again with methanol, and stirred with a mixture of methanol and water (1/1) for 1 h. The polymer was again filtered off, washed with methanol, and stirred with a mixture of methanol and water (1/1) overnight. The polymer was collected by filtration, washed with methanol, and dissolved in xylene with stirring for 2.5 h. The resulting solution was filtered through a 325 mesh filter, and methanol was added dropwise to precipitate the polymer. The precipitated polymer was collected, washed with methanol, and dried under high vacuum to yield 270 mg (55%) of polymer **P3**.

Results and Discussion

Synthesis. The BEHP−PPV monomer, 2-(1′,4′-bis(2″-ethylhexyloxy)benzene)-1,4-bis(bromomethyl)benzene (**4**), was prepared in a four-step reaction according to Scheme 1. The reaction of hydroquinone and 2-ethylhexyl bromide in the presence of K_2CO_3 gave **1**, which was further reacted with NBS to afford bromide **2**. The Suzuki coupling[22] of **2** and 2-(*p*-xylene)boronic acid in DME and in the presence of tetrakis(triphenylphosphine)palladium(0) as catalyst yielded **3**. Radical-initiated benzylic bromination of **3** finally gave monomer **4** in an overall yield of 21%. The MEH−PPV monomer **5** was prepared as described earlier.[17,18] Polymerization of **4** as well as copolymerization of **4** and **5** with potassium *tert*-butoxide (2.6 equiv) in refluxing xylene[23,24] yielded polymers **P1**−**P4** (Table 1). The molecular weight of these polymers was measured by SEC. All polymers **P1**−**P4** were completely soluble in chloroform, THF, and xylene at room temperature. Insoluble

Scheme 1. Synthesis of BEHP−PPV

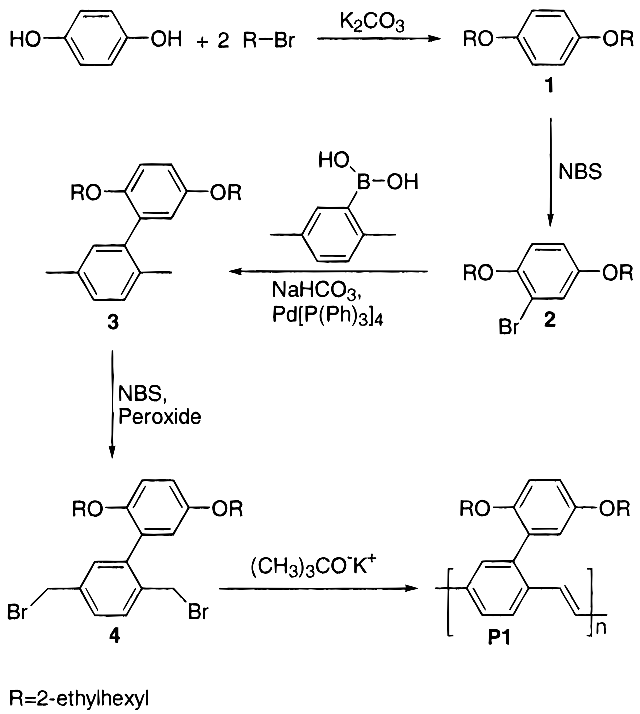

Table 1. Polymerization Yield, Number-Average ($\bar{M}_n$), Weight-Average ($\bar{M}_w$), and Polydispersity ($\bar{M}_w/\bar{M}_n$)

polymer	yield (%)	$\bar{M}_n \times 10^{-3}$	$\bar{M}_w \times 10^{-3}$	$\bar{M}_w/\bar{M}_n$
P1	66	50	280	5.6
P2	34	68	420	6.2
P3	55	115	430	3.7
P4	47	66	350	5.3

polymers were obtained when polymerizations were carried out with potassium *tert*-butoxide (2.6 equiv) in refluxing THF. The reason for this solubility effect might be that polymerization at higher temperature gives polymers with higher disorder and thereby higher solubility.

The ^{1}H NMR of polymers **P1**−**P4** showed seven signals at 0.6−1.8 ppm for the flexible side chains, two signals at 3.65 and 3.76 ppm for the methylenes next to oxygen in the side chains, and signals at 6.6−7.7 ppm due to the aromatic part of the polymers. MEH−PPV-containing copolymers **P2**−**P4** showed an additional signal at 3.90 ppm for the methoxy group in these polymers. Comparing the areas for the peaks at 3.65 and 3.76 ppm with the 3.90 ppm peak showed that the copolymers **P2**−**P4** had approximately the same MEH−PPV content as their monomer composition.

Stability Measurements and UV−vis Spectroscopy. Stability measurements were carried out on polymer films spin-coated onto glass slides (10 mg of polymer/mL of $CHCl_3$, 800 rpm). The films were irradiated with an ordinary light bulb (100 W) in air, and the UV−vis spectra were recorded. Absorption spectra from BEHP−PPV and BUEH−PPV (included in this measurement as a reference of a well-known polymer having absorption maximum in the same region as polymers **P1**−**P4**) after 0 and 24 h are shown in Figure 2. The photodegradation of the polymer was observed as a decrease and blue shift of the absorption. BUEH−PPV was totally degraded after 24 h while polymers **P1**−**P4** sustained the conditions for a much longer time. The BEHP−PPV film was irradiated for more than 3 weeks before it was completely degraded. For the copolymers **P2**−**P4** we found that the rate of degradation increased with the MEH−PPV content.

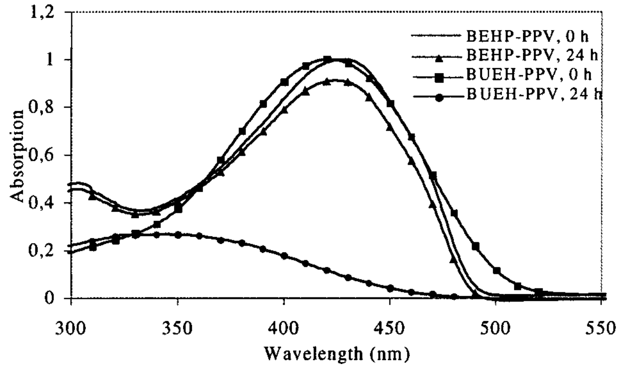

Figure 2. Absorption spectra of films of BEHP−PPV and BUEH−PPV after 0 and 24 h irradiation in air.

The low stability of BUEH−PPV is probably due to the presence of benzylic hydrogens. They react fast with radicals or singlet oxygen to form a relatively stable radical which can then react in a variety of ways giving rise to chain scission.[25−27] The more stable BEHP−PPV has been designed not to contain such reactive hydrogens.

From the UV−vis measurements on polymers **P1**−**P4** in film and solution it is clear that these polymers show only one broad single absorption maximum which indicates that the copolymers are randomly formed. The absorption maxima are shown in Table 2.

Electrochemical Spectroscopy. The cyclic voltammograms of polymers **P1**−**P4** adsorbed on a Pt wire from chloroform solution were measured in a single-compartment electrochemical cell with a Pt counter electrode and a Ag/AgCl reference electrode. The electrolyte (0.1 M tetrabutylammonium perchlorate in acetonitrile) was purged with nitrogen. In Figure 3 we present the first sweep only. (The consecutive sweeps of degraded materials are not relevant for the characterizing of the oxidation processes of the polymers.) We were not able to observe any reduction processes of the polymers above −2.0 V. It is evident from the cyclic voltammograms that there is only a moderate change in oxidation peak potential going from **P1** to **P4**. In addition to this, there are tails seen in the voltammograms of copolymers which has an onset at approximately 0.8 eV. The magnitude corresponds roughly to the percentage of the MEH component. These tails observed for the copolymers certainly help to improve hole injection from the ITO contact, as evidenced by the increase in electroluminescence efficiency (η_{EL}) of the single-layer devices (Table 2) and reduction of operation voltage in single-layer devices.

Photoluminescence and Electroluminescence Properties. Photoluminescence (PL) and electroluminescence (EL) spectra of BEHP−PPV are shown in Figure 4. Both PL and EL spectra show distinct vibronic fine structures with two maxima which indicates relative ordered local structures. Although there is only one emission maximum for the copolymers (Table 2), they still show clear vibronic fine structures (Figure 5). While there is almost no difference in absorption maxima between films and solutions, the emission in solutions is blue-shifted compared to that in films. This effect is caused by the interchain interactions in the films. The

Table 2. PL, EL and Absorption Data for Polymers P1−P4

polymer	Abs_{max} (nm), THF	PL_{max} (nm), THF	η_{PL} (%), THF	Abs_{max} (nm), film	PL_{max} (nm), film	η_{PL} (%), film	η_{EL} (%), SL[a]	η_{EL} (%), DL[b]
P1	429	482, 516	84	429	489, 524	28	0.21	0.63
P2	438	516	71	438	542	36	0.58	0.71
P3	447	527	61	446	551	32	0.68	0.71
P4	449	527	59	449	551	30	0.67	1.15

[a] SL = ITO/polymer/Ca diodes. [b] DL = ITO/PVK/polymer/Ca diodes.

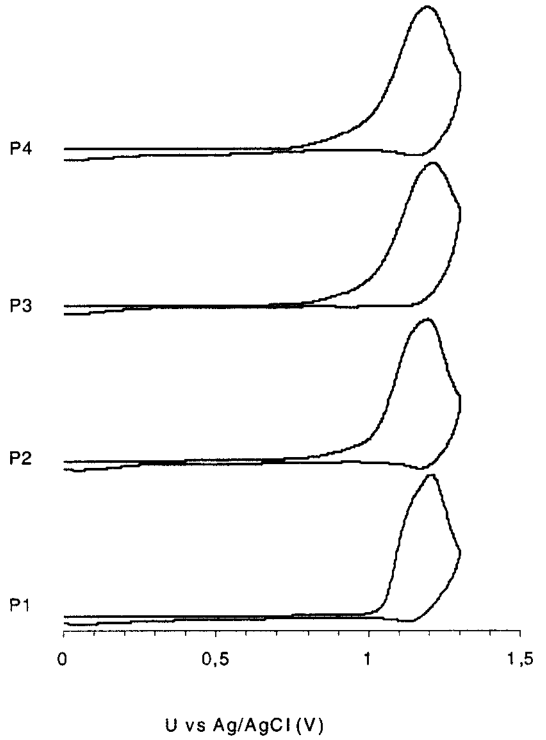

Figure 3. Electrochemical spectroscopy of polymer **P1**−**P4**. The cyclic voltammograms were taken at 100 mV/s.

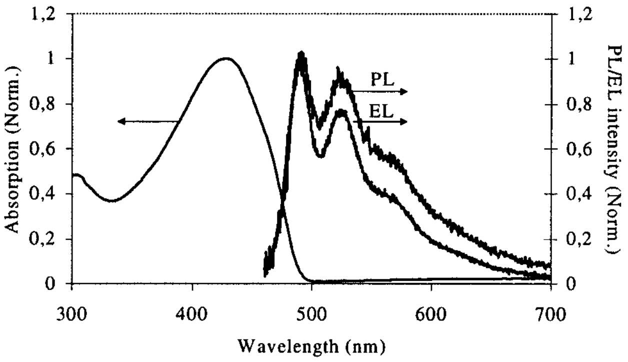

Figure 4. Absorption, PL and EL spectra of BEHP−PPV.

presence of a continuous red shift in absorption and PL maximum on increasing the MEH−PPV content of polymers **P2**−**P4** indicates that they are randomly copolymerized. From Table 2 it can also be seen that BEHP−PPV has the highest photoluminescence efficiency (η_{PL}) in solution (84%) and that an increased amount of the MEH−PPV content decreases the η_{PL}. This effect of MEH−PPV content is not seen on the η_{PL} in films though, where BEHP−PPV has the lowest efficiency and **P2** has the highest efficiency. The lower η_{PL} in pure BEHP−PPV films may indicate strong interchain coupling in its solid state. The higher η_{PL} in the copolymers can be explained by quantum confinement. The presence of segments with narrow band gap in the MEH−PPV part of the copolymer in which the excitons are localized decreases the rate for the excitons to be transported to a defect along the polymer backbone where nonradiative relaxation to the ground state can occur. There must be an energy difference between the two components in the copolymer large enough to localize the excited state. One MEH−PPV segment is probably not enough to achieve this energy difference. Since pure MEH−PPV has a lower η_{PL} than BEHP−PPV, there should be a maximum in η_{PL}, and that we see for **P2**. The emission from BEHP−PPV was green while the investigated copolymers emitted yellow light.

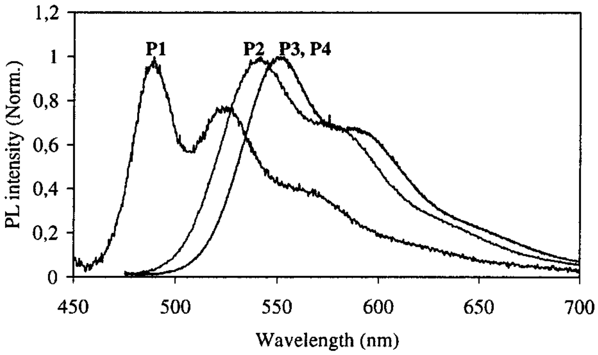

Figure 5. PL spectra in film for **P1**−**P4**.

It is important to get a good balance between electrons and holes injected into the polymer film in LEDs in order to form diodes with high efficiencies. This can be seen in Table 2 from our single-layer devices (ITO/Polymer/Ca) and double-layer devices (ITO/PVK/polymer/Ca). In the double-layer devices poly(vinylcarbazole) (PVK) was used as an electron blocking layer. For all polymers the electroluminescence efficiency (η_{EL}) increased because of the improved electron and hole balance. The ITO used in the single-layer devices was modified (cleaned with acetone and 2-propanol and treated with oxygen plasma) to increase the work function, thereby making it easier to inject holes into the polymers.

Conclusions

This study has shown that it is possible to prepare highly soluble, high molecular weight PPVs. We have also shown that it is possible to design PPV derivatives that are more stable to photooxidation than some of the previously known soluble PPVs. The copolymers showed increased η_{PL} with green and yellow emission. These interesting properties would make them useful as the active substance in devices such as LEDs and photovoltaic diodes.[28]

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