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Electrochemical and Electroluminescent Properties of Random Copolymers of Fluorine- and Alkoxy-Substituted Poly(*p*-phenylene vinylene)s

R. Riehn,[†] J. Morgado,[†,‡] R. Iqbal,[§,||] S. C. Moratti,[§] A. B. Holmes,[§,||] S. Volta,[⊥] and F. Cacialli*,[†]

Cavendish Laboratory, University of Cambridge, Madingley Road, Cambridge CB3 0HE, U.K.; Departamento de Engenharia Quimica, Instituto Superior Técnico, Av. Rovisco Pais, P-1049-001 Lisboa, Portugal; Department of Chemistry, University Chemical Laboratory, University of Cambridge, Lensfield Road, Cambridge CB2 1EW, U.K; Melville Laboratory for Polymer Synthesis, New Museums site, Pembroke Street, Cambridge CB2 3RA, U.K.; and Institut für Organische Chemie und Makromolekulare Chemie, Friedrich-Schiller-Universität Jena, Humboldtstrasse 10, 07743 Jena, Germany

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ABSTRACT: We report the synthesis and electroluminescent properties of random poly(phenylenevinylene) copolymers, carrying fluorine substitutions. In particular, we used 2,3,5,6-tetrafluoro-1,4-phenylenevinylene and 2-methoxy-5-(2′-ethylhexyloxy)-1,4-phenylenevinylene moieties. The ratios of the two copolymer units were varied between 5:95 and 27:75. Electrochemical measurements showed an increase of the oxidation and a decrease of the reduction potentials of the copolymers upon increase of the fluorine content, as expected for the electron withdrawing effect of fluorine on the conjugated π-electron system. The copolymers showed emission in the red-orange region, with maximum photoluminescence efficiencies of 6.7%. We also observed lower turn-on voltages and higher electroluminescence efficiencies for lower fluorine contents.

Introduction

Since the discovery of electroluminescence in poly(*p*-phenylenevinylene) (PPV),[1] substantial research has been dedicated to improving light output, efficiency, and lifetime of polymer light-emitting diodes (PLEDs), as the latter are promising candidates for cheap, bright, and even flexible large area displays.

Among the various factors that control the efficiency, a central role is played by the imbalance of charge carriers with opposite signs. In fact, for the vast majority of electroluminescent polymers, hole mobilities are larger than electron mobilities by 1 order of magnitude or so.[2] Furthermore, charge injection is generally easier for holes than for electrons, as for most polymers the energy barrier for electron injection from common cathodes (Al, Ca) is larger than the barrier for hole injection from indium−tin oxide (ITO) anodes. In this regard, electron-withdrawing substituents have shown to increase both electron affinity and ionization potential (IP), allowing for easier electron injection from environmentally stable cathodes such as Al, and for very high efficiency devices.[3,4] For example, CN-substituted PPVs have allowed fabrication of extremely efficient two-layer structures with Al cathodes and a PPV layer between the ITO and the luminescent CN−PPV.[3] It is generally accepted that this efficiency increase ensues from both the enhancement of electron injection at the cathode and the reduction of the hole current, caused by the presence of an energy barrier for hole injection into CN−PPV at the interface with PPV. Importantly, this carrier confinement effect also increases the distance between the reflecting cathode and the recombination region, thereby reducing luminescence quenching by the metal film.

It has also been suggested that the presence of electron-withdrawing groups promotes electron mobility. If this is the case this should also be true for fluorine-substituted materials, since the electronegativity of fluorine is comparable to that of the CN group.[5] For example, F_{16}CuPc, a highly fluorinated copper−phthalocyanine, has been used successfully for fabrication of n-type organic field-effect transistors.[6]

Fluorine-substituted PPVs were first studied by McCoy[7] and Kang,[8] who found a slight blue-shift of the absorption, and a red-shift of the PL emission, for poly(2-fluoro-1,4-phenylenevinylene) (PFPV), with respect to PPV. They also fabricated single-layer devices with ITO and Al electrodes that displayed efficiencies much higher than PPV.[5,9] In this case, the high driving voltages required for EL (at least 7 V turn-on voltage) were attributed to a high ionization potential giving rise to a barrier of 0.29 eV for hole injection.[5]

A different approach to determine the influence of fluorine substitution has been explored by Benjamin et al.,[10] who studied copolymers of phenylenevinylene and 2,3,5,6-tetrafluoro-1,4-phenylenevinylene (TFPV) moieties and found evidence for conjugation breaking from blue-shifts of the absorption but only small shifts of emission.

We should emphasize that the polymers mentioned above are all from precursor routes, and that this may not be ideal, owing to the difficulties in achieving complete elimination of the labile groups, and also to the large number of parameters affecting the conversion of the precursor to the conjugated material (T, pressure, duration, rate of temperature increase/decrease). We opted instead for the synthesis of copolymers of fluorine

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* Corresponding author. E-mail: fc10004@cam.ac.uk.
[†] University of Cambridge.
[‡] Instituto Superior Técnico.
[§] University of Cambridge.
[||] Melville Laboratory for Polymer Synthesis.
[⊥] Friedrich-Schiller-Universität Jena.

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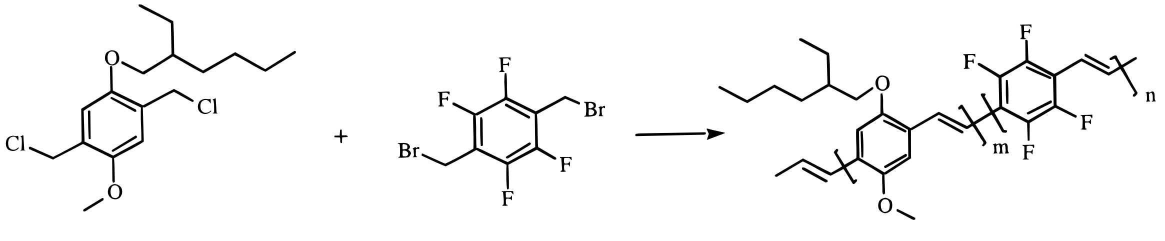
Scheme 1. Synthesis of the Random Copolymer Poly(2,3,5,6-tetrafluoro-1,4-phenylenevinylene)-*co*-(2-methoxy-5-(2′-ethylhexyloxy)-1,4-phenylene vinylene)



(1) **(2)**

containing TFPV and 2-methoxy-5-(2′-ethylhexyloxy)-1,4-phenylenevinylene (MEH−PV) moieties (Scheme 1). The latter provides good solubility and has proven an efficient emitter in PLEDs. The fluorinated copolymers in the present study were synthesized by the base-promoted Gilch-type polymerization[13] of bis(halomethyl)phenylenes in THF (Scheme 1). We used random copolymers with 7, 14, and 19 wt % of the TFPV unit labeled TF7, TF14, and TF19, respectively. We also compared these copolymers with the luminescent conjugated polymer poly(2-methoxy-5-(2′-ethylhexyloxy)-1,4-phenylenevinylene) (MEH−PPV).

In view of the studies on PFPV[5] and MEH−PPV[11] we expected the IP of the copolymers to be more than 0.2 eV lower than the work function of ITO, and we anticipated single-layer devices to suffer from difficult hole injection. For this reason, we fabricated double-layer devices featuring a hole injection layer of poly(3,4-ethylene dioxythiophene) (PEDOT) doped with poly(styrenesulfonate) (PSS), which has proven to lower the barrier for hole injection in devices with an ITO anode.[12]

Experimental Section

We measured oxidation and reduction levels using dynamic pulse polarography (DPP) with a standard three electrode cell. Oxidation data was obtained from thin solid films using a platinum working electrode, a platinum gauze counter electrode and a Ag wire reference electrode, externally referenced against Fc/Fc^+. Tetrabutylammonium hexafluorophosphate ($(TBA)PF_6$) dissolved in dry, degassed acetonitrile (0.1 M) was employed as the electrolytic solvent. We were not able to obtain reduction data for all polymers from films. For this reason, we conducted the reduction measurements in solution of 3 mg of the polymer and 300 mg of $(TBA)PF_6$ in 10 mL of dichloromethane, with data referenced against Ag/AgCl.

All optical and device investigations were carried out at room temperature with thin films (80−150 nm) of polymer prepared by spin-coating from a chloroform solution (approximately 2 wt %, filtered with 5 μm syringe filters). We determined the film thicknesses with a Dektak IIa stylus profiler. We used spectrosil substrates for absorption and PL experiments, and commercially available ITO-coated glass (Asahi, sheet resistance $\leq 10\ \Omega/\square$, transmittance at 550 nm $\geq$ 84%) for fabrication of LEDs. The ITO was patterned so that the metal electrodes on top could be contacted on the edge of the substrate without shorting. We first cleaned the ITO substrates in an ultrasonic bath with acetone and 2-propanol, each for 15 min, and then treated them for 10 min with oxygen plasma at approximately 300 W.[14,15]

Absorption spectra of copolymers were measured with an HP 8453 UV−vis spectrometer, whereas a Lambda 9 Perkin-Elmer double beam spectrometer was used for MEH−PPV. We performed PL measurements with an integrating sphere under nitrogen flow, using an Ar ion laser operating at 457.9 nm with incident power intensities of ~30 mW/cm^2. Spectra for PL and EL were taken with an Oriel Instaspec CCD-matrix spectrograph. Raman spectra were taken with a Renishaw Raman microscope.

Table 1. Summary of Microanalysis, GPC, and TGA Results for the Copolymers Synthesized

	MEH−PPV	TF7	TF14	T19
feed ratio (mol %)				
MEH[a]	100	93	85	80
TF[b]	0	7	15	20
ratio by microanalysis (mol %)[c]				
MEH		94.8	79.7	74.5
TF		5.2	20.3	26.5
microanalysis (%)				
found C	76.47	77.55	74.99	73.87
H	9.00	9.21	8.67	8.65
polymer yield %	93.9	49.5	72.6	51.6
GPC results				
$M_n \times 10^{-5}$ g mol^{-1}	1.1	1.3	1.0	0.96
$M_w \times 10^{-5}$ g mol^{-1}	6.9	9.4	7.0	9.1
PDI	6.28	7.41	6.84	9.42
TGA				
% decomp	31	15	19	18
temp (°C)	452	339	342	334

[a] MEH is 2,5-bis(chloromethyl)-1-methoxy-4-(2′-ethylhexyloxy-)benzene. [b] TF is 1,4-bis(bromomethyl)-2,3,5,6-tetrafluorobenzene. [c] Ratio of each monomer in resulting polymer from comparison of % Carbon ratios with theoretical values.

We prepared double-layer devices with a PEDOT:PSS (Bayer AG) layer (approximately 50 nm) that was spin-cast from an aqueous solution and afterward dried and linked by thermal treatment for 1 h at 100 °C under nitrogen. Spin-cast layers of luminescent polymer were dried for 10 min at 80 °C under nitrogen prior to the deposition of calcium and aluminum electrodes, using thermal evaporation through a shadow mask (pressures of some 10^{-6} mbar). The pixel size was determined by the overlap of the ITO and cathode patterns, yielding an area of $\sim 2 \times 10^{-2}$ cm^2. To prevent fast degradation we also capped the calcium electrodes with aluminum and stored all PLEDs in a dry nitrogen box with less than 20 ppm residual water and 10 ppm oxygen.

We carried out all EL measurements under vacuum using a large area calibrated silicon photodiode. We restricted driving voltages to 10 V and current densities to 180 mA/m^2 in order to prevent fast degradation and to allow repeatable results.

Results

(A) Synthesis. The maximum level of tetrafluorophenyl incorporation attempted was 20% (mol/mol)—higher levels lead to completely insoluble material (as the molecular weights of the two monomers are coincidentally almost identical, the weight and mol % feed ratios are to practical purposes identical). Microanalysis and NMR suggested that the amount of fluorine incorporation in the copolymer was only slightly higher than the feed levels (Table 1), which suggests that the copolymerization may have been largely random given less than quantitative yields of polymer recovery. However, not too much should be read into

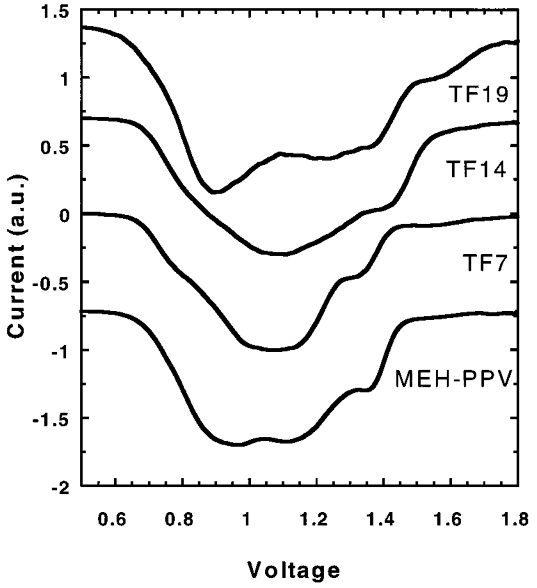


Figure 1. Electrochemistry data showing positive DPP voltage sweeps referenced against Fc/Fc$^+$ for determination of oxidation potentials.

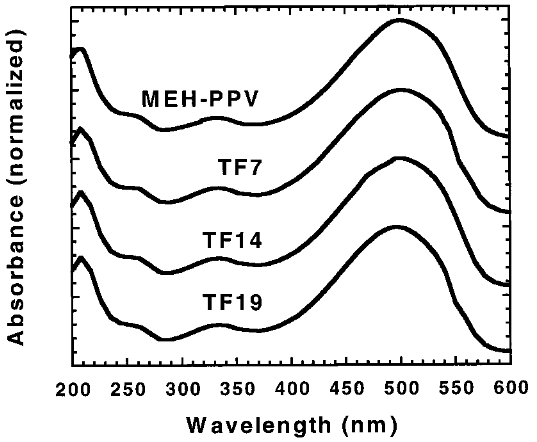


Figure 2. UV–vis absorption spectra of thin films on spectrosil substrates show peaks at 500, 499, 499, and 496 nm for MEH–PPV, TF7, TF14, and TF19, respectively.

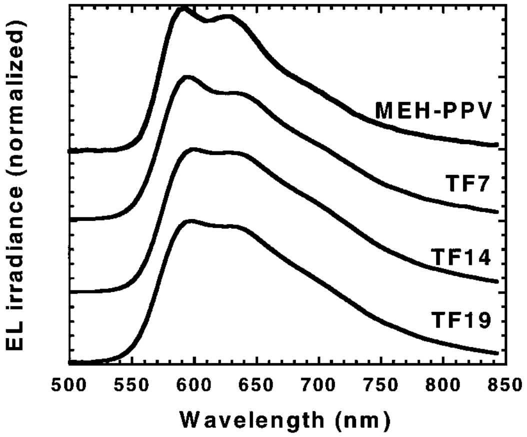


Figure 3. EL spectra measured in devices with ITO anodes, PEDOT:PSS hole injection layer, approximately 100 nm thick luminescent polymer layer, and Ca cathodes. The active area is ~2 mm^2.

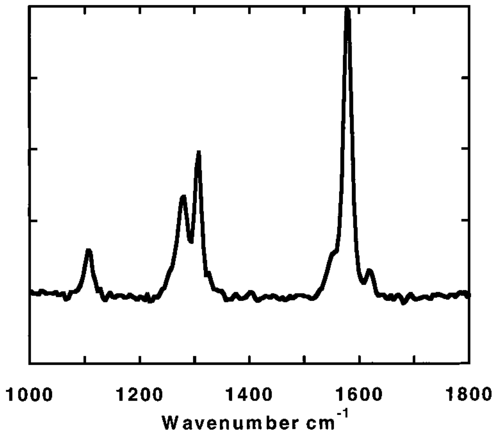


Figure 4. MEH–PPV Raman spectrum. Energies of peaks in spectra of fluorine-containing copolymers were identical within the resolution of the Raman microscope.

Table 2. Summary of PL Quantum Yields, Maximum EL Efficiency, and Turn-on Voltage for Double-Layer PLEDs, with a PEDOT:PSS Hole Injection/Transport Film between ITO and the Luminescent Polymer

active layer	PL (%)	EL efficiency (cd/A)		turn-on voltage (V)		ratio of EL efficiencies using Ca and Al cathodes
		Al	Ca	Al	Ca	
MEH–PPV	14		0.52		2.5	
TF7	4.2	0.0045	0.077	2.2	2.2	17.1
TF14	6.7	0.0038	0.069	2.8	2.4	18.2
TF19	4.5	0.0017	0.033	2.8	2.5	19.8

the microanalytical data in view of the probable margin for error. As the amount of fluorine in the monomer feed was increased, it was noticeable that the solubility of the polymers decreased markedly, though the molecular weights were not affected.

(B) Electrochemical and Optical Properties. The positive DPP voltage sweeps for oxidation are shown in Figure 1. Note that MEH–PPV shows minima at 0.96 and 1.11 V as well as a shoulder at 1.35 V. The main feature of the TF7 sweep is a minimum at 1.10 V. We also see a shoulder at 1.32 V (similar to that in MEH–PPV, at ~1.35 V) and an additional feature at 0.8 V. Moving to TF14, we notice that the main feature at 1.10 V is broader than that in TF7. There are shoulders at 0.85 and 1.41 V. The TF19 sweep shows a distinctively different structure with a sharp deep minimum at 0.90 V. A plateau centered at ~1.24 V, and a shoulder at 1.51 V can be seen as well. Negative DPP voltage sweeps for reduction show first minima at −1.29, −1.23, and −1.19 V for TF7, TF14, and TF19, respectively.

The absorption spectra (Figure 2) of the copolymers are very similar to that of MEH–PPV, but we find a blue-shift with increasing fluorine content. We report photoluminescence quantum efficiencies in Table 1 and EL spectra in Figure 3. All spectra peak at very similar wavelengths in the range between 592 and 599 nm. Raman spectra were identical for all the materials in this study (Figure 4).

(C) PLEDs. A summary of the EL characteristics for double-layer PLEDs made with a PEDOT hole injection layer is given in Table 2. The reported turn-on voltage is the voltage at which the photodiode showed a current above the background noise (~10^{-3} cd m^{-2}).

The EL properties of MEH–PPV diodes are consistent with those reported in the literature,[17] with a peak EL efficiency of 0.52 cd/A, for Ca cathodes. Peak EL efficiencies (cd/A) using TF7, TF14 and TF19 were about 1 order of magnitude lower using the same cathode metal (Ca) (Figure 5). In general we found that the EL

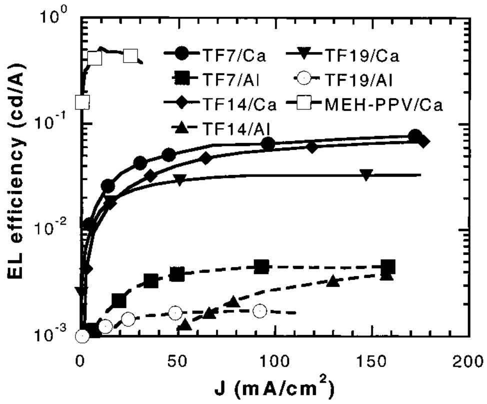


Figure 5. EL efficiency (cd/A) for LEDs with PEDOT:PSS layer vs of current. We found that devices featuring Ca cathodes are 1 order of magnitude more efficient than those featuring Al cathodes. The thickness of the active layer is given in the Figure 6 and the active area is ~2 mm^2.

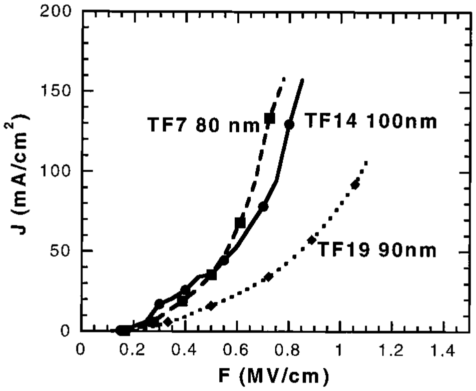


Figure 6. Current density vs electric field characteristics for devices using Al cathodes. Resistivity rises as the fraction of TFPV units is increased.

efficiency decreases with increasing fluorine content. However, this is not always the case at low current levels (see for example TF19 and TF14, Figure 5). Interestingly, the ratio of EL efficiencies between LEDs fabricated with Al and Ca was higher for lower fluorine content suggesting that the charge population imbalance, and in particular the electron injections are poorer if the fluorine content is increased.

We also found that turn-on voltages tend to increase with fluorine content, with the lowest value registered for TF7 and Ca cathodes (2.2 V). However, we found the highest turn-on voltage for MEH−PPV (2.5 V) (Ca cathode). The electrical characteristics for LEDs with Al electrodes are shown in Figure 6. From these we observe that lower fractions of TFPV result in higher currents. Current densities in MEH−PPV are lower than in the fluorinated copolymers. Similar results were obtained using Ca cathodes, with higher current densities for the fluorine-substituted polymers with respect to MEH−PPV.

Introduction of a 20 nm 2-(4′-biphenylyl)-5-(4″-*tert*-butylphenyl)-1,3,4-oxadiazole (PBD) 75% in PMMA hole-blocking layer between TF19 and the Ca cathode resulted in lower currents and an increase of efficiency of the device of ~40%.

Discussion

Electronic Structure. We notice first of all that the trends for reduction potentials are as expected on the basis of the fluorine electron-withdrawing and the alkoxy chain's electron-donating nature. It follows that electron injection should become easier upon fluorine addition.

We consider the oxidation behavior to be a result of both steric and electronic effects. Of special interest is the prominent feature at 0.90 V in TF19. This feature can be interpreted either as being the feature (a) appearing at 0.96 V in the MEH−PPV spectrum, after a ~0.06 V shift to lower voltages, or (b) corresponding to the shoulders at 0.8 and 0.85 V in TF7 and TF14 voltammograms, respectively, and therefore could be associated with the presence of fluorine.

However, we notice that the electron-withdrawing character of fluorine would suggest that shifts of any feature should be toward higher potentials. Indeed, we observe that other features, and in particular the shoulder at ~1.35 V in MEH−PPV, seems to follow this trend. In the same way the minima at 0.96, 1.06, 1.10, and 1.24 V in MEH−PPV, TF7, TF14, and TF19, respectively, appear to be corresponding and of the same physical nature. We therefore consider that hypothesis b above is more likely, although we cannot exclude that the different reactivity of the monomers may lead to formation of oligomeric blocks of the same unit (though there is little evidence from TF7 and TF14). Alternatively there could be a drift in polymer composition as the polymerization proceeds as one monomer is preferentially used up. This by itself would not explain the strong change from TF19 to TF14 and TF7, but high fluorine material (monomer and polymer) may then tend to phase separate from solution during the polymerization, leading to more MEH−PV rich chains later on.

Following hypothesis b, we notice that the intensity of the feature at around 0.85 V does not grow proportionally with the fraction of TFPV units. We suppose that the latter are sterically screened from intimate contact with the electrolyte by the bulky alkoxy chains of the neighboring MEH−PV units. In this picture only clusters of adjacent TFPV units can be efficiently oxidized electrochemically. Corroborating evidence is provided by the observation that the feature under discussion lies at voltages lower than the oxidation potential of MEH−PPV (implying a lower binding energy, hence a higher HOMO), whereas one would expect the electron-withdrawing character of fluorine to increase the oxidation potential. This may be an indication that orbitals partially extended on the alkoxy groups are involved, or that the coupling of electrons on the benzyl ring to the ones on the vinyl group is different because of a different molecular symmetry upon F substitution. However, we cannot exclude phase segregation phenomena, owing to preferential interaction of TFPV clusters with the Pt electrode. We consider that full quantum-chemical modeling is required in order to get further insight into the nature and spatial distribution of the orbitals involved in these electrochemical processes.

Optical Properties. The relative independence of the absorption spectra from fluorine content is remarkable. For similar copolymers, but without alkoxy substitution, Benjamin and colleagues found a blue shift of the absorption maximum of more than 50 nm, when going from 1 to 30% TFPV content,[10] whereas we only see a 4 nm blue shift when passing from MEH−PPV to TF19. Note that the molar concentration of TFPV in TF19 is 26.5%, and therefore a similar effect might be expected for our material. Benjamin's results also indicate that the absorption of a pure TFPV polymer lies close to 360 nm, which is 140 nm lower than the peaks observed by us. However, Gurge et al. found that substitution of another benzylic hydrogen in PFPV with an additional fluorine (poly(2,5-difluoro-1,4-phenylenevinylene)) resulted in a marginal alteration of absorption, but also in a more significant (~30 nm) red shift of the emission in both PL and EL.[9] Notably, Gurge et al.[9] also report a red shift of the absorption and emission spectra for the homopolymer poly(2[5]-*n*-hexyloxy)-5[2]-fluoro-1,4-phenylenevinylene) (EL peak at 610 nm) with respect to PFPV (EL peak at 560 nm).

The origin of these discrepancies may be due to a variety of factors such as selective coupling of similar units during the polymerization, competition of the electron-withdrawing and -donating effects of the different substituents, the influence of the conversion parameters for the precursor-route polymers used by other groups, or even to competing effects of steric (TF−PPV) and electronic (donor−acceptor) interactions.

First, we notice that different polymerization kinetics may result in a block copolymer with long subchains of TFPV and MEH−PV units, and in this case the MEH−PV units are still expected to dominate both the low-energy absorption features and the emission spectra. However, we do not see any shoulder around 360 nm, as would be expected in view of Benjamin's work.[10] Interestingly, Raman spectra of MEH−PPV and of the fluorinated copolymers also display identical spectral features, suggesting a negligible influence of the F substitutions. Corroborating evidence about the presence of relatively long segments of MEH−PPV in the fluorine copolymers is also provided by the EL and PL spectra that show essentially the same maxima as MEH−PPV. However, although this observation does not imply lack of polymeric segments with shorter conjugation, as it can be explained easily by exciton migration (*via* Förster transfer) to the MEH−PPV segments, the lack of TFPV signature in the absorption spectra suggests a different origin.

Second, it may be possible that the electron-donating effect of the alkoxy chains is strong enough to realize an "electronic buffer", compensating or neutralizing the effects of fluorine. As mentioned above, Gurge et al.[9] have indeed shown that addition of an alkoxy chain onto the phenyl ring of PFPV can effectively red shift the spectrum, in agreement with general trends and previous studies on the effects of alkoxy chains in 2,5-positions.[18] We note in this regard that steric effects related to the difference between H and F hindrance have been considered to play a minor role,[9,10] in view of the similar radii of the two elements. We consider again that quantum chemical calculations can provide significant insight into these effects.

Third, we wish to emphasize that for precursor polymers the optical and electronic properties of the conjugated polymer segments are heavily influenced by the conversion process, as also observed in PPV.[19,20] In particular the high-temperature treatment is likely to influence the degree of order and crystallinity of the thin films and, through constraints on the molecular geometry, the electronic structure.

PLED Characteristics. From DPP measurements, we expect the energy barrier for hole injection into TFPV containing copolymers to be within 0.2 V of the level in MEH−PPV and, therefore, no barrier to hole injection at PEDOT/luminescent polymer interface. Current densities were indeed very similar for both Al and Ca cathodes, although the work functions of these metals differ by more than 1 eV. We conclude that the current−voltage characteristics are dominated by the mobilities of the charge carriers rather than by injection processes.

We also observe that PL and EL efficiencies do not correlate (Table 2). Whereas the PL efficiency of the copolymers are between one-half and one-third of that of MEH−PPV, the maximum EL efficiencies only are between 14% and 6% of that of MEH−PPV. From analysis of devices prepared with a PBD hole-blocking layer, we can also conclude that electron and hole currents are not balanced, with holes as majority carriers and the recombination zone closer to the metallic reflecting cathode. This is not ideal, because both chemical and physical interactions of the cathode with the polymer quench the luminescence in proximity of the electrode.

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