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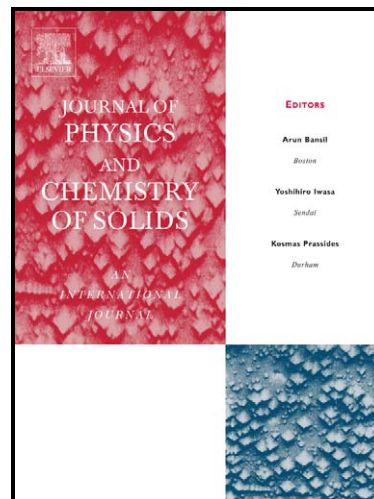
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Luminescence properties of poly(9,9-dioctylfluorene)/polyvinylcarbazole blends: Role of composition on the emission colour stability and electroluminescence efficiency

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ABSTRACT

Light-emitting diodes (LEDs), based on blue-emitting polyfluorenes are usually prone to the appearance of a contaminant green emission (centered around 520 nm), leading to an apparent whitish light emission. We find that, for LEDs based on poly(9,9-dioctylfluorene), PFO, the blending with the hole transporting polyvinylcarbazole, PVK, can suppress such green emission. LEDs based on a PFO/PVK blend with a 1:2 weight ratio and with aluminum cathodes show a quite stable blue emission. This result reveals the important role played by the interchain interactions on the observed contaminant green emission. In addition, we observe that in Al-based devices blending

causes a decrease in EL efficiency while in Mg-based devices we obtained higher efficiencies with the blend PFO:3PVK when comparing with neat PFO-based devices.

Keywords: polymers, luminescence, optical properties, transport properties

1. INTRODUCTION

Conjugated electroluminescent (EL) polymers have been explored for various optoelectronics applications, such as light-emitting diodes (LEDs)[1], photovoltaics [2] and light-emitting field-effect transistors [3]. The molecular nature of these materials makes their properties particularly sensitive to modifications of their chemical structure, either by design or as a result of degradation processes. For instance, significant modifications of their emission spectra, but also of their charge transport ability, are observed upon modifications of their molecular structure. In solid state, interchain excited states (e.g. excimers or charge transfer excited states) can be formed, competing with the intrinsic intrachain excited states for the capture of the excitons energy. Such interchain states can, in working LEDs, be directly populated through on site direct charge recombination, or act as energy traps for the initially formed intramolecular excited states. The combination of energy transfer plus on site recombination originates, in general, a significant amplification of such interchain states contribution to the total electroluminescence, when comparing with their relative importance for light-emission upon photoexcitation (photoluminescence). Aggregates and excimers or exciplexes are the most commonly invoked/observed interchain states in EL polymer films. While the first type of interchain states, possessing an attractive ground state, can be directly

probed/identified by UV/Vis absorption spectroscopy, excimers possess, instead, a repulsive ground state and are not detectable by UV/Vis absorption.

Interchain states have been explored to obtain, for instance, white LEDs. However, they usually represent a “degraded state” with respect to the intended emission from the pure system. Besides the formation of interchain states, the emission pattern can also change as a result of degradation processes induced by photooxidation or by secondary reactions following charge injection.

Polyfluorenes, PFs, are among the most studied EL conjugated polymers [4], the homopolymers being blue emitters. Emission colour can be tuned by copolymerisation with lower energy gap comonomers [5] or through blending with low energy gap polymers [6,7]. Stable pure blue emission is not easily obtained from LEDs based on neat PFs. Poly(9,9-dioctylfluorene), PFO, and other PF-based LEDs, exhibit a green emission component which grows during device driving, leading to a whitish emission. The origin of this green emission has been the subject of various reports and remains a matter of debate. First studies attributed this emission to excimer states [8]. Later, the presence of fluorenone defects, formed by photooxidation or electrically induced at the C9 position of PFs, was held responsible for such emission [9]. More recent studies proposed that excimer [10] or charge transfer states involving fluorenone units [11] are the true species responsible for such emission. Chemical strategies, such as modifications of the substituents on C9 of PFs [12], have been proposed to suppress or even eliminate such emission. Other approaches involve PFO chain isolation. In particular, blending of PFO with a photo- and electrically inert polymer (polystyrene) or with an electron-transporting polymer [13] or the PFO intercalation into nanocomposites [14,15], were shown to improve the blue emission stability.

Here, we report on the luminescence properties of PFO/PVK blends and show that the use of the hole-transporting polymer PVK is successful at suppressing the green emission. The molecular structures of these polymers are shown in scheme 1. PVK was chosen due to its good hole-transporting properties, being widely used in multilayer LEDs. We note that previous studies on blends of PFO with an electron-transporting material and even with polystyrene were shown to improve colour stability and, for low amounts of the additional polymers, to increase device efficiency [16]. In this study we find that blending with PVK improves colour stability and, in some cases, leads to improved efficiency.

2. EXPERIMENTAL

Absorption spectra were recorded with a CECIL CE7200 spectrophotometer and emission photoluminescence (PL) spectra were obtained with a SPEX Fluorog fluorimeter. LEDs preparation closely followed a previously reported procedure [17]. Oxygen plasma treated indium-tin oxide (ITO)/glass substrates were spun coated with poly(3,4-ethylene dioxythiophene) doped with polystyrene sulphonic acid (PEDOT:PSS or PEDOT) (Baytron P, AI4083) and dried in air at 150 °C, for 2 minutes, yielding a film thickness of about 45 nm, as measured with a Dektak 6M profilometer. Films of PFO or PFO/PVK blends, with thicknesses of 70 nm and 100 nm, respectively, were spun coated on top of PEDOT from their chloroform solutions. Finally, aluminum or magnesium cathodes were thermally deposited at a base pressure of 2×10^{-5} mbar, defining pixel areas of 4 mm². The devices were tested under vacuum (ca. 10^{-2} mbar). Light output was measured with a calibrated silicon photodiode. The system was calibrated with a luminance meter. External quantum efficiencies (EQE) were estimated

taking into account the spectral response of the photodiode and assuming a Lambertian emission profile [18]. Electroluminescence, EL, spectra were recorded with an Oriel Instaspec IV spectrograph. PFO/PVK blends are identified according to the weight proportion of the two components (1:1, 1:2 and 1:3) as mixed in chloroform.

3. RESULTS

Fig. 1 shows absorption and emission spectra of PFO and PVK films deposited by spin coating on spectro-sil discs. The emission of PVK, of excimeric nature, shows a significant overlap with PFO's absorption spectrum, which favours energy transfer from PVK to PFO. Fig. 2 compares the effect of the annealing at 150 °C for 2 hours in air, under indoor daylight, on the emission spectra of films of neat PFO and films of PFO/PVK blends. Upon annealing, the PL spectrum of neat PFO shows the growth of the typical broad green emission centered at about 520 nm. However, no noticeable changes are found in the absorption spectra upon annealing of neat PFO films. As Fig. 2 clearly shows, the blending with PVK improves the colour stability: the intensity of the broad green band in the blends is much smaller compared to neat PFO and it decreases with increasing PVK content.

Fig. 3 shows the characteristics of the LEDs based on neat PFO and on their blends with PVK when either Al or Mg is used as cathode material. The devices were tested until maximum values of current and/or luminance were reached.

In the case of the devices with Al cathodes, we find that the blending of PFO with PVK up to 1:2 ratio leads to a reduction of the light-onset voltage (from ca. 4.5 V for neat PFO to 3.5 V) and an increase of the current flowing through the devices at lower

voltages. The devices based on neat PFO or on PFO:3PVK are those attaining highest maximum luminance values (ca. 150 cd/m²). Slightly lower values are obtained for the devices with the other compositions. In the device based on the PFO:3PVK blend, a notorious change of the current-voltage behaviour occurs since much higher voltages are required to attain current and luminance values similar to those of the other devices. Among these devices, the one based on neat PFO is the most efficient (reaching a maximum efficiency of 0.27 cd/A, corresponding to an estimated external quantum efficiency, EQE, of 0.12 %). Upon blending with PVK the maximum efficiency decreases, being the PFO:PVK blend the most efficient (reaching maximum values of 0.06 cd/A or 0.025% EQE).

The use of magnesium instead of aluminum, as cathode material, leads to similar effects of blending on the device characteristics, as shown in Fig. 3b). It is worth noting, the reduction of light-onset voltage when PFO:PVK is used instead of neat PFO, and its significant increase when PFO:3PVK is used. Devices with this last blend (having the highest PVK content) exhibit a quite distinctive current- and luminance-voltage behaviour. While the efficiency of the PFO-based device reaches a maximum value of 0.23 cd/A or 0.095% EQE, similar to the values obtained with Al cathodes, the devices based on the blends are more efficient when magnesium is used as cathode material. For instance, the device based on PFO:3PVK reaches a maximum efficiency of 0.31 cd/A or 0.22% EQE, which is nearly five times higher than the efficiency of the Al-based devices. Furthermore, comparing figures 3a) and 3b), we can also conclude that the current values obtained for the devices with magnesium cathodes are, in general, lower than for those with aluminum cathodes, at similar voltages.

Fig. 4 compares the EL spectra of the various devices, at various driving voltages. Devices based on neat-PFO (Fig. 4a) show quite extensive degradation of the blue emission. The relative intensity of the green band at ca. 520 nm is significant, increasing with the driving voltage. When PFO:PVK is used (Fig. 4b), the green component is still present but, up to the maximum driving voltage, its relative intensity (with respect to the blue peak at about 420 nm) never gets as high as observed in the neat PFO devices. Further increase of the PVK content (1:2 blend) leads to a significant stabilization of the blue emission. Up to the maximum driving voltage (maximum luminance), the green emission remains with very small intensity (Fig. 4c). Finally, the use of the PFO:3PVK blend does not significantly modify the colour stability of the Al-based devices. The use of Mg cathodes leads to a significant amplification of the green component. Electroluminescence spectra were also recorded as a function of the driving time. Contrary to what is observed for LEDs based on neat PFO, with the green component growing in time, we find that the devices based on PFO/PVK blends show much higher spectral stability. For instance, Fig. 5 shows the EL spectra of devices based on the PFO:3PVK blend with Al cathodes as a function of time under a bias voltage of 12V. The spectral variation is very small. It is worth noting, however, the appearance of an “extra” low intensity band at ca. 620 nm. This is not observed in neat PFO-based devices but is observed in devices based on neat PVK. Its appearance is therefore attributed to a degradation of PVK. When Mg is used as cathode we find a higher contribution of the green band. The time dependence of the EL spectra of a ITO/PEDOT/PFO:3PVK/Mg device is shown in Fig 5b). It can be observed that, compared with results for the Al-based LEDs, there is a higher contribution of “impurity” emissions at ca. 520 nm and 620 nm.

4. DISCUSSION

The dilution of PFO with PVK increases the thermal stability with respect to the appearance of the contaminant green emission, as shown in Figure 2. This effect is in agreement with previous studies that show that isolation of PFO chains decreases the importance of the green emission. Two factors can contribute to this observation: i) the mechanism leading to the green-emitting sites may require interchain interactions, as mentioned above, and the blending reduces the probability for such interchain interactions to occur; and/or, ii) the green-emitting sites are of intrachain nature, and isolation simply reduces the probability of the energy transfer from pure PFO sites to these lower energy sites. Therefore, these results, while evidencing the better colour stability against annealing, do not allow us to draw a conclusion on the mechanism behind the green colour. This remains, therefore, a matter of debate and further studies are currently in progress.

PFO and PVK are both good hole-transporting polymers. Hole mobility in PFO has been reported to be around 10^{-4} $\text{cm}^2/\text{V}\cdot\text{s}$ for electric fields around 10^6 V/cm, while electron mobility is found to be around 3 orders of magnitude smaller [19]. Similar values, in the range $4 \times 10^{-5} - 5 \times 10^{-4}$ $\text{cm}^2/\text{V}\cdot\text{s}$, for the hole mobility of PFO were obtained from time of flight studies at a field of 5×10^5 V/cm [20]. Using this same technique, the hole mobility of PVK at 10^6 V/cm was reported to be much lower, around 10^{-7} $\text{cm}^2/\text{V}\cdot\text{s}$ [21]. Therefore, besides the increased disorder associated to the use of blends, the presence of PVK should lead to a decrease of hole mobility.

Using cyclic voltammetry we estimated [22] the ionization potential (I_p) and electron affinity (EA) of PFO to be 5.8 and 2.1 eV, respectively, in agreement with previous reports [23]. For PVK the reported values are 5.8 and 2.3 eV [24]. Based on these values, we do not expect significant variation of the hole-injection barrier from PEDOT (workfunction of ca. 5.2 eV [25]) into the electroluminescent layer, being this either neat PFO or the PFO/PVK blends ($\Delta h \approx 0.6$ eV). Furthermore, the presence of PVK should only slightly decrease the electron injection barrier. These values of ionization potentials and electron affinities for PFO and PVK, combined with the spectral overlap between PVK emission and PFO absorption, support the existence of energy transfer of the excited states from PVK to PFO. This is also consistent with the absence of PVK contribution to the emission of the blends (neat PVK emission is centered at ca. 400 nm).

As mentioned above, PFO and PVK have the same I_p values. In these devices the electron injection barriers (Δe) are much higher than the hole-injection ones (in case of PFO, $\Delta e \approx 2.1$ eV, for Al, and $\Delta e \approx 1.6$ eV, for Mg, considering a workfunction of 4.2 eV for Al and 3.7 eV for Mg). For PVK the expected electron injection barrier should be slightly decreased, by 0.2 eV. The reduction of the light-onset voltage and the higher currents observed above light-onset voltage for the devices where PFO:PVK and PFO:2PVK blends are used instead of neat PFO, is somewhat unexpected, in view also of the lower hole-mobility of PVK. The small difference in the electron-injection barrier, which controls light-onset and should become more important in the bipolar regime, appears to have a rather relevant effect.

For the blend with the highest PVK content, PFO:3PVK, a quite extensive detrimental effect on the current flowing through the devices occurs, which seems to indicate that

the benefits from improved electron injection are overcompensated by a reduction of charge mobility. We note that both hole and electron mobilities are expected to be reduced in the blends with respect to neat PFO (due to disorder).

In terms of the external quantum efficiency, we find that the addition of the PVK significantly reduces the values. In particular, the much lower value found for the PFO:2PVK blend-based LED, when comparing with the devices based on the two other blends, is unexpected and still not explained!

In general we also found that the use of Mg as cathode material, although having a lower workfunction than Al, does not lead to an increase of the current flow through the devices. This is an unexpected result, which we cannot yet explain, but interfacial contamination may be involved. Surprisingly, the EL efficiency of the neat PFO based device is similar for both cathode materials, but increases for the blends-based devices using Mg cathodes as expected for an improved electron injection ability of this material. The observed increase of the light-onset voltage when Mg is used instead of Al is also unexpected, on the same grounds, but is likely related to the decrease of the current when comparing with Al-based devices.

As these are mainly hole-transporting devices, it appears as if the improvement of the cathode electron injection ability is not enough to improve efficiency, which contradicts a priori expectations and usually reported results. A possible explanation is that the improved exciton formation efficiency resulting from a better charge carrier balance leads to an improvement of exciton formation mainly close to the cathode, where metal plasmons are efficient at quenching luminescence. However, we need further studies to confirm if this explanation is correct.

As observed in the PL studies, the dilution of PFO with PVK has a profound effect on the colour stability of the LEDs. In fact, the results of Fig. 4 and 5 show that devices with Al cathodes and using PFO:2PVK or PFO:3PVK exhibit negligible contaminant green emission. The drawback of the use of these blends is the reduction of the EL quantum efficiency. Among the blends used in the Al-based LEDs, the one with composition 1:2 is the one that combines a low light-onset voltage, and a high colour stability. However, this is the one giving the lowest EL efficiency: a maximum EQE of about 0.01% compared to about 0.1% for the neat PFO. The use of a 1:3 composition leads to a significant recovery of the efficiency, while maintaining colour stability (see Fig. 4d). However, the voltage required to attain similar current and luminance values almost doubles.

When the blends are used as emissive layer in the Mg-based LEDs, we now find a much higher efficiency than for the similar devices with Al-cathodes, as mentioned above. In fact, LEDs with the 1:3 blend show a quantum efficiency that is twice that of the LEDs of neat PFO. We find, however, that the purity of the blue emission is lower than that of the Al-based devices (see Fig. 4 and 5). The EL spectra of Fig 4 and 5 were recorded at higher voltages for the Mg devices, but the current at such voltages is mostly smaller than that of the corresponding Al-based devices. Therefore, we consider that there is no direct correlation between the increased weight of the green component and the current value, when comparing Al and Mg cathodes. In fact, at similar currents, we find that the Al-based devices show much lower contaminant green emission. It appears as if Mg induces itself a faster degradation. Furthermore, the second contaminant emission at about 620 nm (ascribed to PVK) is more important. A possible explanation may be that the higher electron injection from Mg may favour the formation of the species (such as

fluorenone in the case of PFO) responsible for that emission. It appears that driving the devices at higher voltages, leading to increasing green emission, mainly leads to higher electron currents into the bulk of the device and contaminant species formation.

Therefore, the use of Mg may, in this case, facilitate the formation of those species.

It is worth noting that, in the EL spectra, the contaminant green emission occurs at the same wavelength range irrespective of the cathode material used (Al or Mg). In addition, the EL green emission peaks at 522 nm with shoulders at 490 and at 580 nm. In the PL spectrum of annealed PFO (Fig. 2) we find a peak and shoulders at similar wavelengths, although with lower resolution. This observation contrasts with previous reports on additional peaks observed in the EL spectra of polyfluorene LEDs and which were attributed to “interface defects” originating from chemical reactions between the polymer and the cathode metals [26,27].

5. CONCLUSIONS

The blending of PFO with PVK suppresses the contaminant green emission, centered around 520 nm, observed in the PL spectrum of neat PFO samples annealed in air. This suppression effect increases with the amount of PVK present. This is in agreement with previous observations that have shown that isolation of PFO chains decreases the importance of the green emission. This beneficial effect of blending of PFO with PVK on the PL colour stability of the annealed samples is also observed in the EL emission spectra of the LEDs with the general structure: ITO/PEDOT/(PFO : x PVK)/(Al or Mg), where $x=0,1,2$ or 3 . For devices based on neat PFO, the intensity of the green emission increases considerably upon increase of the driving voltage. However, for the devices based on PFO:2PVK and PFO:3PVK the emission colour remains stable upon increase

of the driving voltage, in particular if Al cathodes are used. A comparison of Al and Mg-based devices shows that, at similar currents, the colour stability is better for the devices with Al cathodes.

In terms of current-luminance –voltage behaviour we find that the blending of PFO with PVK up to 1:2 ratio, by weight, leads to a reduction of the light-onset voltage and an increase of the current flowing through the devices.

These results show that blending is a good strategy to improve the blue colour stability of PFO-based devices. The results obtained for the PFO:/PVK blends are not yet ideal to achieve this purpose, but provide a background for studies along this strategy.

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FIGURE CAPTIONS

Scheme 1. Molecular structure of poly (N-vinylcarbazole), PVK, and of poly(9,9-dioctylfluorene), PFO.

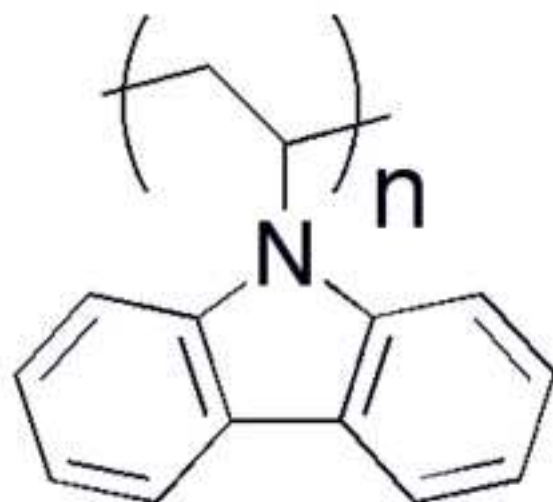
Fig. 1. Normalized absorption and emission (PL) spectra of neat films of PFO and PVK. PL spectra were recorded upon excitation at 380 nm for PFO and at 296 nm for neat PVK. Changing PVK excitation to 346 nm leads to the same PL spectrum.

Fig. 2. Effect of the annealing in air, under indoor light at 150 °C for 2 hours, on the PL spectra of neat PFO and two blends with PVK. Excitation wavelength: 380 nm.

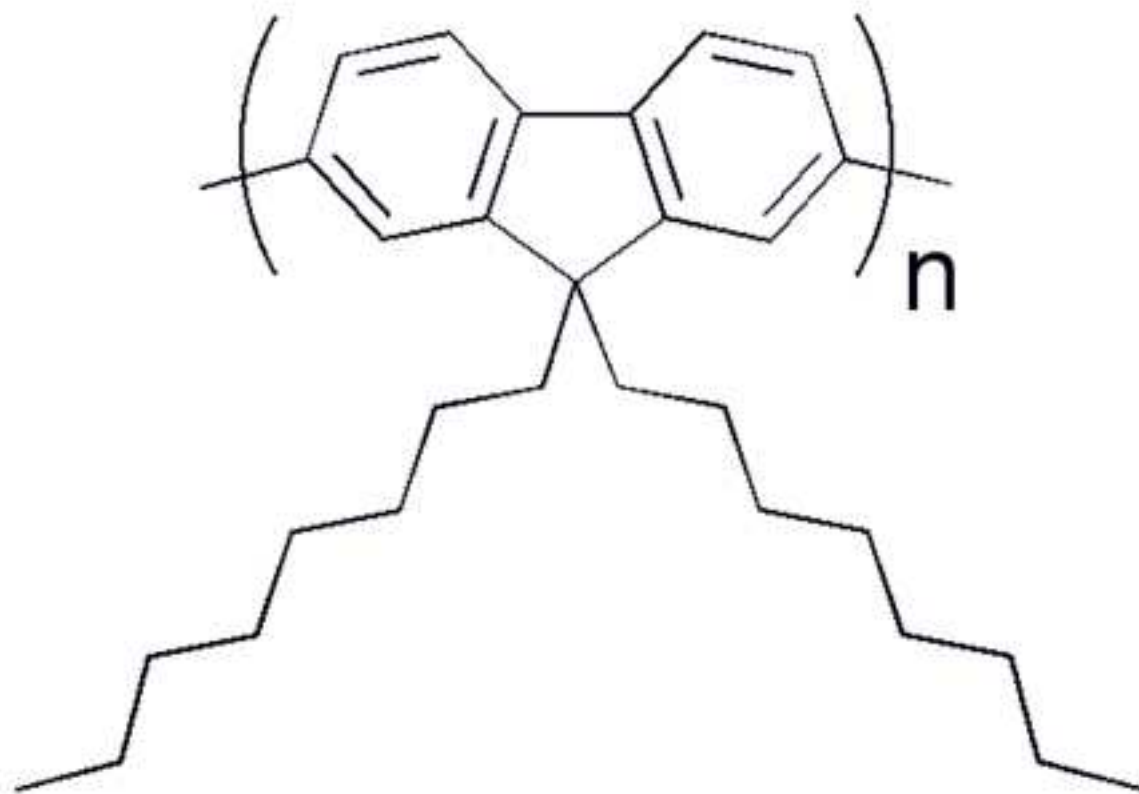
Fig. 3. Current density (J, filled symbols) and luminance (L, open symbols) of the LEDs based on neat PFO (circles) and its blends with PVK (1:1-squares; 1:2- triangles; 1:3-diamonds), with a) Al cathodes and b) Mg cathodes, as a function of the applied voltage.

Fig. 4. Normalised EL spectra at various applied voltages for devices with Al and Mg cathodes (M) and using either (a) neat PFO, (b) PFO:PVK; (c) PFO:2PVK or (d) PFO:3PVK blends.

Fig. 5. EL spectra of devices based on PFO:3PVK as a function of time, for devices with (a) Al cathodes under an applied voltage of 12V; and (b) Mg cathodes at 18 V.



PVK



PFO

