Flexible organic light-emitting diodes

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Abstract

Since the first report of electroluminescence observation in organic materials, both small molecules and polymers, the field of organic light-emitting diodes (OLEDs) has greatly expanded, covering various application areas such as displays and lighting. In view of the unique mechanical properties of the organic materials, flexible OLEDs are being developed for integration in buildings and furniture and for biomedical applications, namely sensors. OLEDs are key components of the Internet of Things paradigm. In this project, different materials were studied for the manufacture of OLEDs, to obtain the best performing ones in terms of luminance and efficiency, providing benchmarking systems to be replicated in flexible OLEDs. Within this project we achieved flexible OLEDs, fabricated on PET/ITO substrates, with a luminance of 663 cd/m^2, 0.45 cd/A luminance efficiency and 0.36% external quantum efficiency.

KEYWORDS: Light-emitting diodes (OLEDs), flexible OLEDs, electroluminescence, organic materials

1 Introduction

The first light-emitting diodes (LEDs) were based on inorganic semiconductors but, more recently, LEDs based on organic semiconductors, OLEDs, have become the center of a new S&T revolution, by the simplicity of fabrication and their mechanical properties, namely their flexibility. In search of a new field of research aimed at the foundation and deepening themes pertinent to the aspirations of society, the development of flexible organic light-emitting diodes emerges as a pillar supporting relevant research. The field of organic materials has greatly expanded, and OLEDs are a key for Internet of Things paradigm which consist on the combination of advanced data analytic capabilities and everyday objects, transforming how we work and live.

OLED technology has received great attention from industry and academia over the past three decades due to its obvious advantages such as easy preparation, low cost, safe application, light weight, low driving voltage and quick response. (Reineke *et all*, 2013). Organic light-emitting diodes (OLEDs) have drawn enormous attention due to their outstanding performance in high-quality full-color display and solid-state lighting applications.

An OLED typically consists of a stack of organic layers between an anode and a cathode, at least one of which is partially transparent. In organic electroluminescent (EL) devices, the generation of light is the consequence of recombination of injected holes and electrons of the electrodes. In a chemical sense, the reaction of radical cations (holes) and radical anions (electrons) provide excited molecules that emit light as one of the decay processes. (Kido *et al*, 1995) The final purpose of an OLED structure is to create an excited state (exciton) in a molecule (being it of low molecular weight or polymer). By similarity between the emission spectra resulting from photon excitation (fluorescence or photoluminescence) or from electrical excitation, it was concluded that the same emissive excited state was formed.

The structure of OLEDs was initiated with a single layer (described above) and gradually evolved to double layer and triple layer and to their current multilayer arrangement. Even though multilayer organic light-emitting devices (OLEDs) are known to be more efficient than single-layer OLEDs, these remain very interesting, in particular for materials characterization, due to their simple structure. (Kim *et al*, 2005).

2 Experimental

Square transparent substrates, 12x12 mm^2, made from different materials were used: glass coated with ITO and without ITO, for rigid OLEDs, and polyethylene terephtalate (PET) with ITO for flexible OLEDs, respectively. The ITO layer used has 100 nm of thickness. ITO is a conductive material and is transparent in the visible spectral region, but almost opaque in the UV and infrared regions (Gheidari *et al*, 2005), which makes this semiconductor a good option to be used as a transparent conductive oxide (TCO) (Farhan e*t al*, 2013). The ITO is etched from the sides, to avoid short circuits, leaving a central stripe, whose width (8 mm) determines the active area. The glass/ITO and PET/ITO substrates proceed for an oxygen plasma treatment.This treatment is very important when PEDOT: PSS is deposited on top (from an aqueous dispersion), as it allows a uniform film formation. Each treatment lasted approximately 3 min. To ensure an efficient treatment, this step was repeated once more under the same conditions.

The samples then went to the organic film deposition process using the method of spin coating. The concentration of the solution/dispersion and spinning speed directly influence the thickness of the deposited layer. The substrates were first coated with a hole-injection layer of PEDOT: PSS (CLEVIOS P VP AI 4083, PEDOT to PSS ratio 1:6, from Heraeus) with a thickness of 80 nm, which was cured at 125 °C for 10min on a hot plate in air. As emissive materials, we used some neat polymers and blends (poly(9,9-dioctylfluorene) – PFO; poly(9,9-dioctylfluorene-*alt*-benzothiadiazole) - F8BT; PFO: F8BT – F95; PFO: $Ir(dmpq)_2(acac)$ - PFOIr95), deposited by spin coating from toluene solution to obtain between 70 to 75 nm thick films. PFO, F8BT and the iridium complex ($Ir(dmpq)_2(acac)$) were obtained from Ossila and used as received.

Another study was carried out using a different PEDOT: PSS formulation (PH1000, PEDOT to PSS ratio 1:2.5 from Heraeus, supplied by Ossila) which is one of the best choices for flexible electrodes owing to its high transmittance in the visible range, high and adjustable conductivity, intrinsically high work function, excellent thermal stability, and good film-forming capability as well as superior mechanical flexibility (Hu *et al*, 2020). It is important to have a transparent highly conductive polymer that may be used in the fabrication of flexible OLEDs, replacing the ITO.

The device structures were completed with thermal evaporation of the calcium cathode (20–30 nm) and a protection overlayer of aluminum (100 nm) under vacuum (base pressure of ca. 10^{-6} mbar) in a evaporator placed inside the glovebox. The OLEDs were measured in a home-made set up consisting on a power supply (1), a dark chamber (2), a support with 5 contacts (3) for the OLED analysis in the dark chamber, and a computer controlled Scansci spectrometer connected to the dark chamber (4) with an optical fiber to obtain the emission spectra.

The thickness of the films was measured with a profilometer (DEKTAK 6M Stylus Profiler), and the absorption was measured with a CECIL spectrophotometer. The photoluminescence spectra were obtained for films deposited on quartz substrates under illumination with light-emitting diodes and emission detection.

The conductivity of PEDOT:PSS-based films deposited by spin coating on glass substrates (as used during the manufacture of OLEDs) was determined by the 4-point probe technique with a Keithley 2400 Source Meter unit and a multimeter (Agilent 34401A 6½ Digital Multimeter). For the measurements, the films were coated with a 40 nm thick Au layer deposited under high vacuum ($<10^{-4}$ mbar) by thermal evaporation through a shadow mask to define four gold contacts equally spaced. At least 4 different films for each condition/material were characterised.

3 Results and Discussion

We have studied first OLEDs based on single emissive materials followed by the investigation of OLEDs based on blends. The use of blends has, in general, the aim of balancing the charge transport mobility (of electrons vs holes), confine the emissive states (excitons) in isolated chains to avoid

charge and exciton concentration quenching effects and/or tuning the emission color. The study also involved the application of electrode materials that would be more adequate for the fabrication of flexible OLEDs. Along the presentation of the results, the optical (absorption and emission) properties of the materials are also presented and discussed in connection with the properties of the OLEDs incorporating them.

3.1 LEDs based on neat polymers

Two well-known electroluminescent conjugated polymers, poly(9,9-dioctylfluorene) and poly(9,9-dioctylfluorene-*alt*-benzothiadiazole), were initially studied to gain insight on the OLEDs fabrication and characterization. They were later used in the fabrication of OLEDs based on blends.

3.1.1 Poly(9,9-dioctylfluorene), PFO

Figure 1 shows the performance of a PFO-based OLED with the structure ITO/PEDOT: PSS/PFO/Ca/Al.

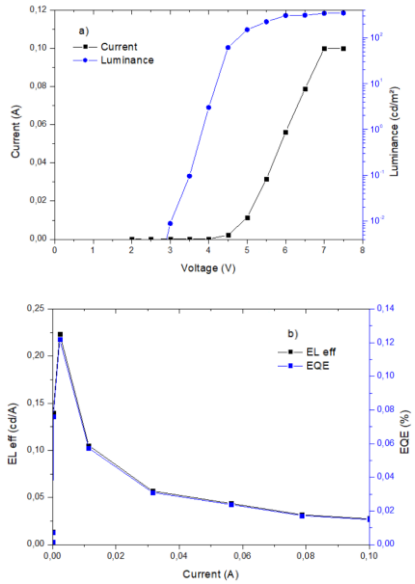

Figure 1. a) Current and luminance as a function of the applied voltage for an ITO/PEDOT: PSS/PFO/Ca/Al OLED, b) corresponding electroluminescence efficiency and EQE as a function of the current

The device shows a light-onset voltage (voltage at which a luminance of 10^{-2} cd/m^2 is detected) of 3.2V and reaches a maximum luminance of 347.86 cd/m^2 at 7V. At this voltage, the device reaches the current limiting value (100mA) of the voltage source. The efficiency of the device shows a peak value at low current (up to ca. 0.22 cd/A and ca. 0.12%), which decreases upon increase of the driving voltage. It is possible that at higher current values there is a charge-induced quenching of the excitons.

3.1.2. Poly(9,9-dioctylfluorene-*alt*-benzothiadiazole), F8BT

Figure 2 shows the result of a F8BT-based LED. It shows a light-onset voltage of ca. 2V, a maximum luminance of 7326 cd/m^2 reached at 5.5 V, and a maximum efficiency of 1.75 cd/A and EQE of 5%. The EL spectrum is shown in Figure 3. The emission maximum occurs at 547 nm. F8BT's absorption spectrum shows two peaks at 325 and 475 nm. At variance with the behavior of PFO, these LEDs show a stable EL spectrum.

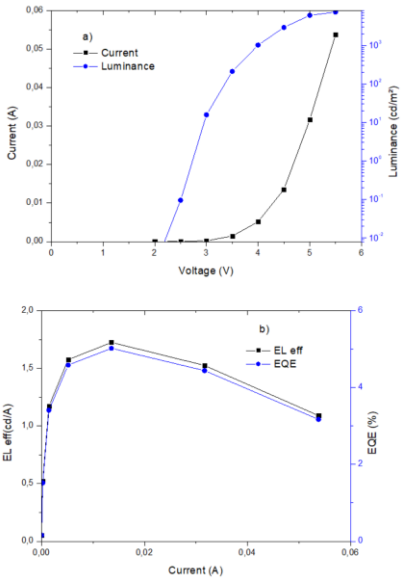

Figure 2. a) Current and luminance as a function of the applied voltage of an ITO/PEDOT: PSS/F8BT/Ca/Al LED.

3.2.LEDs based on blends

3.2.1. PFO: F8BT (F95)

PFO has been reported to show higher hole than electron mobilities while these mobilities are similar for F8BT (Lee *et al*, 2015); electron mobilities of about 10^{-3} cm^2 /Vs and hole mobilities of about 10^{-2} cm^2 /Vs. As shown in Figure 3a, the emission of PFO has a significant spectral overlap with F8BT absorption, thereby fulfilling a condition for efficient energy transfer. In addition, as shown in Figure 3 b) the energy level

alignment forms a type I heterojunction, which fulfills a second condition for efficient excited state energy transfer. Several studies have shown that blends of these two polymers have led to LEDs with better performance, as compared to LEDs based on neat F8BT (Chen *et al*, 2006). PFO and F8BT have similar HOMO energies, though the LUMO energy of F8BT is lower than that of PFO (Bernardo et al, 2010); for PFO we have LUMO at an energy of -2.1 eV and HOMO at -5.8 eV and for F8BT we have LUMO at -2.4 eV and HOMO at -5.9 eV. Therefore, electron injection from calcium into the LUMO of F8BT is easier than the electron injection into PFO, while hole injection from PEDOT:PSS has similar barriers for both polymers. A blend that was shown to be quite successful for LEDs applications is F95, which is a mixture of 95% of PFO and 5% of F8BT (by weight). Despite the low F8BT content, the LEDs emit in the green, evidencing the efficient energy transfer from PFO to F8BT. The fact that electrons may be trapped in the LUMO of F8BT (as this has lower energy than the LUMO of PFO) can also promote the exciton formation within F8BT chains.

Figure 4 shows the result of a typical F95-based LED (T12). A light-onset voltage of 2.5 V is observed, reaching a maximum luminance of 18920 cd/m^2 at 7 V. The emission efficiency has a maximum value at 4V (4cd/A and EQE of 1.25%), decreasing upon increase of the flowing current, indicating an increase of the luminescence quenching (a phenomenon known a roll-off). In the calculation of the LEDs luminance a conversion factor of 2765 cd/m^2/V was used. At the maximum luminance, the efficiency reaches 1.8 cd/A. It is worth noting that, by comparison with the LEDs based on F8BT, a much higher luminance and EL efficiency are obtained, evidencing the benefits of the addition of PFO. It is also remarkable that the presence of only 5% F8BT is enough to obtain an almost pure F8BT emission. No significant PFO residual emission is observed. Despite this small effect, the major conclusion is that the efficiency of the combined effects of on-F8BT-sites recombination and the exciton transfer from PFO to F8BT are not noticeably affected by the flowing current.

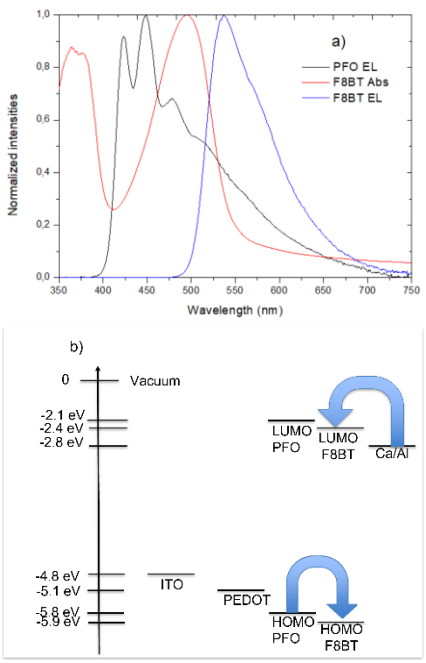

Figure 3 a) PFO emission spectrum and F8BT absorption and emission spectra and b) Energy levels diagram for the ITO/PEDOT:PS/F95/Ca/Al LED. The frontier levels energy values are taken from Bernardo *et al* (2010).

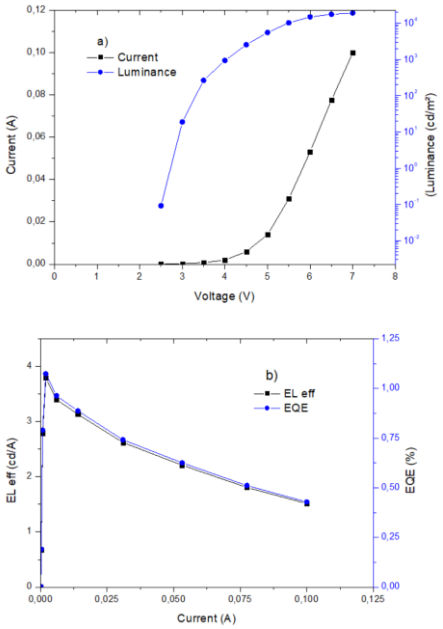

Figure 4 a) Current and luminance as a function of the applied voltage of an ITO/PEDOT: PSS/F95/Ca/Al OLED, b) corresponding electroluminescence efficiency and EQE as a function of the current.

3.2.2 PFO: F8BT:Ir (1) /66:4:30

A composition with iridium complex content was prepared, consisting of 64% of PFO; 4%

of F8BT and 30% of $Ir(dmpq)_2(acac)$, by weight.

$Ir(dmpq)_2(acac)$ is a phosphorescent red emitter. The absorption shows an onset at 375nm, with a significant spectral overlap with PFO's emission (as shown in Figure 5a), fulfilling one of the conditions for excited state energy transfer. The energy level alignment (Figure 5b) shows that a second condition, where the energy gap energy of $Ir(dmpq)_2(acac)$ is smaller and this will make it easier for charge carriers. This diagram also shows that the iridium complex can act as efficient charge trapping site, increasing exciton formation and subsequent emission.

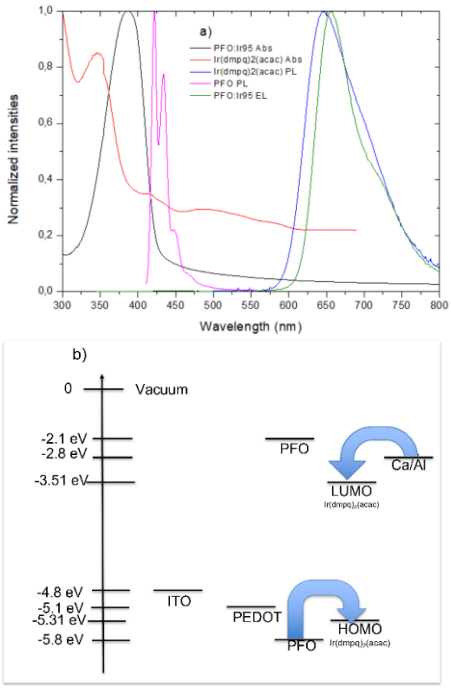

Figure 5 a) Absorption and photoluminescence spectra of $Ir(dmpq)_2(acac)$ and the photoluminescence spectrum of PFO compared with blend absorption and emission; b) shows the energy levels diagram for the ITO/PEDOT:PS/PFO:Ir95/Ca/Al LED, the energy values are from Lee *et al* (2013) and Bruno *et al* (2012).

Figure 6 shows the performance of a PFO: F8BT: Ir (1)-based LED. A minimum luminance of 10^{-2} cd/m^2 is detected at 2V reaching a maximum value of 1341 cd/m^2. EQE reaches a maximum value of 0.164% corresponding to a maximum luminance efficiency of 0.17 cd/A. In the calculation of the LEDs luminance a conversion factor of 765 cd / m^2 / V was used.

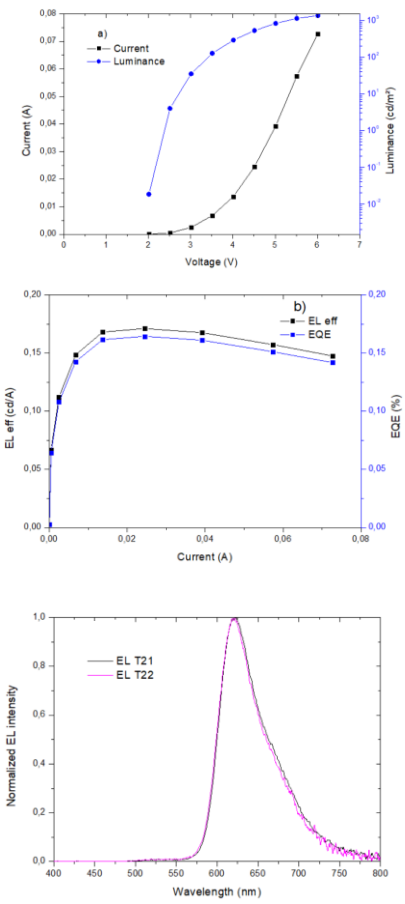

Figure 6 a) Current and luminance as a function of the applied voltage of a PFO: F8BT: Ir (1)-based LED, b) corresponding electroluminescence efficiency and EQE as a function of the current and c) normalized EL spectra of PFO:F8BT:Ir (1)-based LEDs

Figure 6c shows the contribution of only one component, the iridium complex, which proves that the singlet excitons transferred to the iridium complex, along with those formed directly at the iridium complex sites, will then undergo an intersystem crossing to the triplet excite state (T_1), from which the red-light emission takes place. It also important to mention that the triplet excitons are the majority of the electrically formed excited states within the OLED, those formed at the PFO sites (which would undergo an almost complete non-radiative decay) can be transferred to nearby iridium complex sites (considering that the T_1 state of the iridium complex has lower energy (2.0 eV) (Lee *et al*,2013) than the T_1 of PFO (at 2.3 eV) (Monkman *et al*, 2001). Therefore, the use of the iridium complex should allow an increase of the EQE in comparison with the OLEDs based on neat PFO.

3.2.3 Modification of PEDOT: PSS

PH1000 was investigated aiming to replace PEDOT: PSS (AI 4083), because PH1000 has been reported to show higher conductivity and it is important to have a transparent highly conductive polymer that may be used in the fabrication of flexible OLEDs, replacing the ITO.
It was reported that the conductivity of PEDOT: PSS can be increased by addition of dimethyl sulfoxide (DMSO) (Hwang *et al*, 2018). We investigated this process, preparing samples with different DMSO contents (6% and 10%, by weight) and measuring the resistance of thin films prepared on glass by spin coating. It is observed that DMSO promotes an increase of conductivity.

3.2.3.1 Glass/PH1000 + 6%DMSO/ PFO: F8BT: Ir (66:4:30) /Ca/Al

Figure 7 shows the performance of this blend LED. A light-onset voltage of 2.85 V is observed (in this case we consider the minimum luminance of 10^{-2} cd/m^2), reaching a maximum value of 234 cd/m^2. The efficiency reaches a maximum value of 0.025% at the highest driving voltage. The luminance efficiency in 0.029 cd/A.

3.2.3.2 Glass/PH1000 + 10%DMSO/PFO: F8BT: Ir (66:4:30) /Ca/Al:

Figure 8 shows the performance of this blend LED. A light-onset voltage of 2.5 V is observed (in this case we consider the minimum luminance of 10^{-2} cd/m^2) reaching a maximum value of 314 cd/m^2. The efficiency reaches a maximum value of 0.036% at the highest driving voltage. The luminance efficiency in 0.041 cd/A. In the calculation of the LEDs luminance a conversion factor of 842 cd / m^2 / V was used.

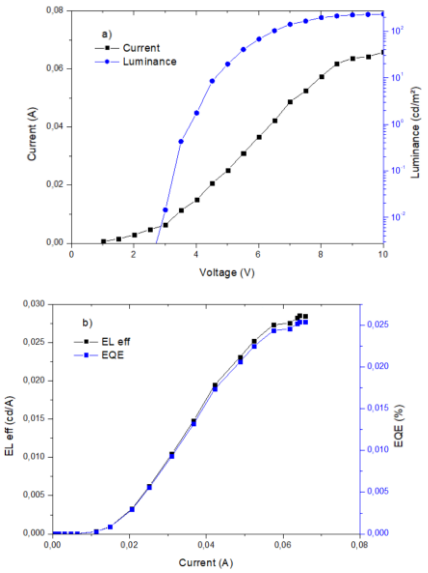

Figure 7. a) Current and luminance as a function of the applied voltage for Glass/PH1000 + 6%DMSO/ PFO: F8BT: Ir(1)/Ca/Al. b) corresponding electroluminescence efficiency and EQE as a function of the current.

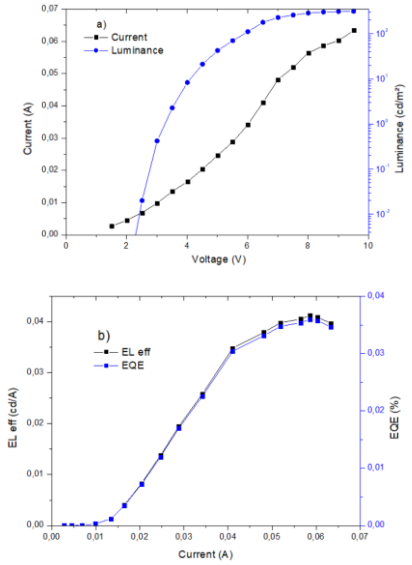

Figure 8. a) Current and luminance as a function of the applied voltage for Glass/PH1000 + 10%DMSO/ PFO: F8BT: Ir(1)/Ca/Al. b) corresponding electroluminescence efficiency and EQE as a function of the current.

Comparing the values obtained in LEDs with 6% of DMSO and the ones with 10% of DMSO, it is possible to conclude that the luminance, EQE and luminance efficiency improved when the concentration of DMSO is higher.

It is concluded that the use of PH1000 modified with DMSO could be a replacement for ITO.

3.4 LEDs on flexible material (PET/ITO)

3.4.1 Poly(9,9-dioctylfluorene), PFO

Figure 9 shows the performance of a PFO-based LED on PET/ITO/PEDOT: PSS/PFO/Ca/Al. The device shows a light-onset voltage (voltage at which a luminance of 10^{-2} cd/m^2 is detected) of 3.5 V and reaches a maximum luminance of 202 cd/m^2 at 6V. Above this voltage we find a performance degradation. The efficiency of the device shows a high value at low current (up to ca. 0.18 cd/A and 0.15%) which decreases and stabilizes at about 0.125 cd/A and 0.08% at the highest luminance values.

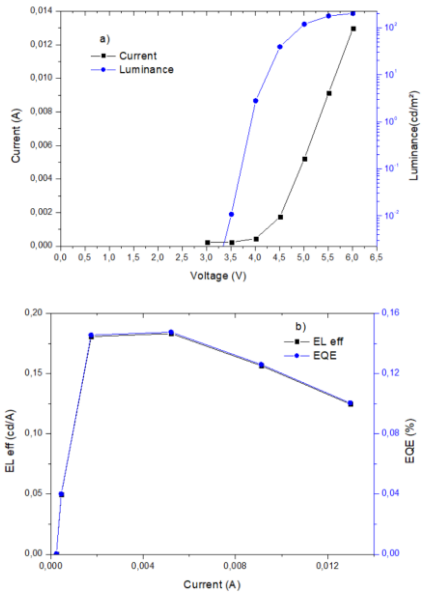

Figure 9 a) Current and luminance as a function of the applied voltage for PET/ITO/PEDOT: PSS/PFO/Ca/Al. b) corresponding electroluminescence efficiency and EQE as a function of the current.

3.4.2. Poly(9,9-dioctylfluorene-*alt*-benzothiadiazole), F8BT

Figure 10 shows the performance of a F8BT-based LED. It shows a light-onset voltage is 2.5V. a maximum luminance of 20 cd/m^2 reached at 8 V. The maximum efficiency reached 0.05 cd/A and an EQE of 0.04%. It is surprising the poor performance of this device when comparing with the regular F8BT OLED prepared on glass/ITO.

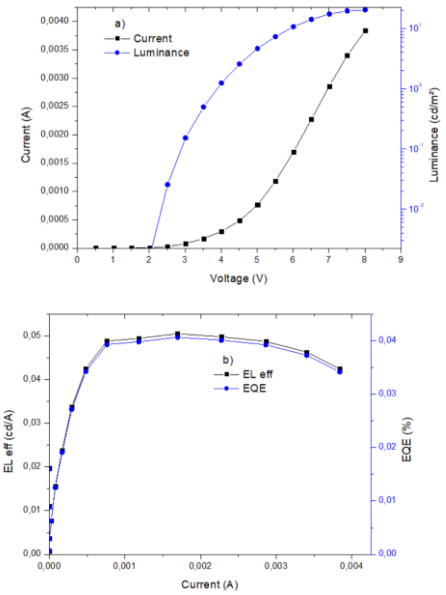

Figure 10 a) Current and luminance as a function of the applied voltage for PET/ITO/PEDOT: PSS/F8BT/Ca/Al b) corresponding electroluminescence efficiency and EQE as a function of the current.

3.4.3. PFO: F8BT (F95)

Figure 11 shows the result of a typical F95-based LED. A light-onset voltage of 2.5V is observed, reaching a maximum luminance of 663 cd/m^2 at 8 V. The emission efficiency has a maximum value at 0.45 cd/A and EQE of 0.36%.

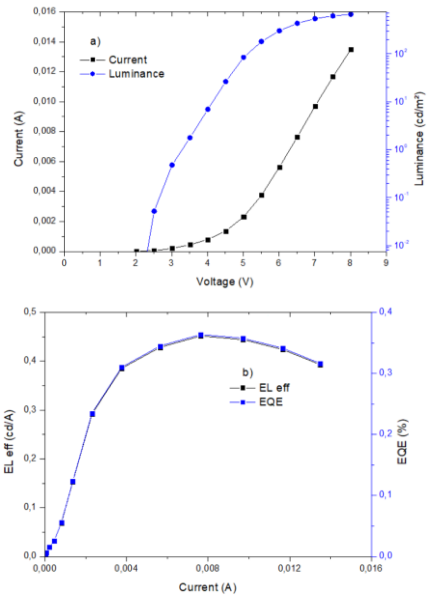

Figure 11 a) Current and luminance as a function of the applied voltage for PET/ITO/PEDOT: PSS/F95/Ca/Al b) corresponding electroluminescence efficiency and EQE as a function of the current.

3.4.4. PFO: F8BT: Ir (1) (66:4:30)

Figure 12 shows the performance of a PFO:F8BT:Ir (1) - based LED. A light-onset voltage of 2V is observed (in this case we consider the minimum luminance of 10^{-2} cd/m^2) reaching a maximum value of 154 cd/m^2. The efficiency reaches a maximum value of 0.08% at the highest driving voltage. The luminance efficiency in 0.1 cd/A.

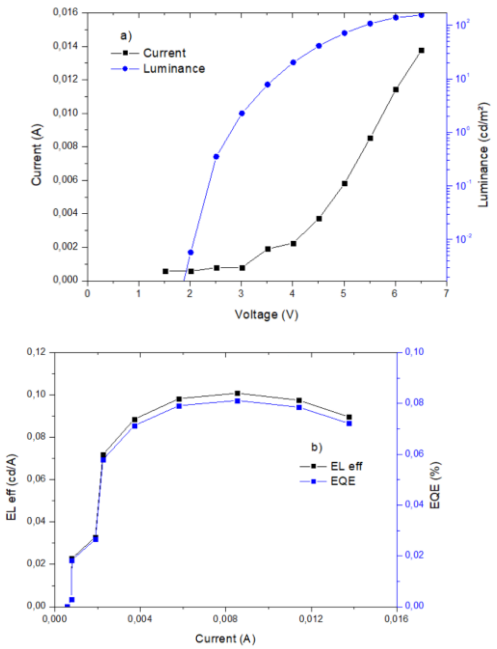

Figure 12 a) Current and luminance as a function of the applied voltage for PET/ITO/PEDOT: PSS/PFO: F8BT:Ir95(1)/Ca/Al b) corresponding electroluminescence efficiency and EQE as a function of the current.

Table 1 compares the performance of OLEDs produced on PET and on glass.

It is possible to conclude that OLEDs produced on a flexible substrate (PET) have lower luminance, EQE and luminance efficiency. When comparing only PFO, it is noticed that the values are very close.

When comparing PFO: F8BT: Ir(1) it is possible to verify a closely luminance efficiency between them but on flexible material the luminance is 10 times lower.

It is concluded that it is possible to manufacture flexible LEDs with the use of PET/PH1000: DMSO but with lower luminance and efficiencies values than on Glass with ITO.

Table 1. Comparison between different OLEDs produced.

Material	Type	Luminance (cd/m^2)	EQE (%)	Luminance efficiency (cd/A)
PFO	PET/ITO	202	0.15	0.18
PFO	GLASS/ITO	348	0.12	0.22
F8BT	PET/ITO	20	0.04	0.05
F8BT	GLASS/ITO	7326	0.5	1.75
F95	PET/ITO	663	0.36	0.45
F95	GLASS/ITO	18920	1.25	4
PFO: F8BT:Ir (66:4:30)	PET/ITO	154	0.08	0.1
PFO: F8BT:Ir (66:4:30)	GLASS/ITO	1341	0.164	0.17

4 Conclusions

This research was based on the study of the fabrication and characterization of Organic Light Emitting Diodes on ITO-coated glass substrates already found in the literature, for comparison of results for the different compositions;it was also based in the optimization of these same OLEDs for the replacement of ITO as an electrode in order to be possible to manufacture OLEDs completely flexible and the final step was the fabrication and characterization of flexible OLEDs on PET/ITO substrates. OLEDs with different types of polymers (neat and blends) were prepared and tested. All solutions were prepared and deposited in a glove box filled with nitrogen in order to avoid oxygen and humidity. For each composition films were prepared various layer thicknesses, methods of deposition, temperature and annealing times. It was necessary the preparation of the differentiated formulation of PEDOT with DMSO to study the conductivity in order to understand which concentration of DMSO would bring better results when applied to OLEDs.

A maximum luminance of 18920 cd/m^2 was possible, luminance efficiency of 4 cd/A and EQE of 1.25% for the blend of PFO and F8BT known as F95, while in flexible OLEDs, this same blend achieved 663 cd/m^2 luminance, luminance efficiency of 0.45 cd/A and EQE of 0.36%.

With this study, it was possible to carry out the fabrication of flexible OLEDs based on PET / ITO, with results related to the

literature studied. It is possible to verify on Figure 13 examples of the flexibles OLEDs prepared in this research.The world is evolving and developing technologies more and more, OLEDs become more and more the key to this growth. Its applications continue to grow and no longer just meet the basic needs that drove its development. Therefore, this study does not end in itself, but it opens up to other possibilities and continuity of investigations, as the theme finds an echo in current needs.

Figure 13 Flexible OLED with a) PFO and b) F8BT.

For future developments, the fabrication of flexible OLEDs on elastomeric substrates, which have the ability to avoid the use of metallic electrodes, which has an advantage in relation to avoiding possible short circuits.

This development can be based on the data obtained in this research, which demonstrates the conductivity of the differentiated formulation of PEDOT with DMSO, in order to avoid ITO and make the OLED completely flexible, and can also be based on the polymers (neat and blends) used here in order to optimize their manufacture and characterization.

The world is always developing, which we didn't even think could exist before, today we don't even imagine how to live without.

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