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# OLEDs fabricated by solution process based on a novel linear poly(arylene oxindole)

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## ABSTRACT

In this work, OLEDs based on a new modified polymer **PMC 300\*** (as emissive layer: EML) were manufactured with the structure ITO/PEDOT:PSS/Polymer (**PMC 300\***)/LiF or PFN/Al. This new polymer **PMC 300\***: Poly[(Benzo[c][1,2,5]thiadiazole-4,7-diylbis(9,9-dimethyl-9H-fluorene-7,2-diyl))-3,3-diyl(1-(3-(trifluoromethyl)phenyl)-2-oxindole)], is a modified version of PF-1 polymer that was synthesized and used previously in our group for non-linear optical properties and in OLED devices. The CF<sub>3</sub> additional group on **PMC 300\*** showed an improved electroluminescence and current efficiency on OLED devices. PF-1 and **PMC 300\*** polymers have a fluorescence quantum yield (FLQY) of approximately 1. Film formation of the hole injection layer (HIL) and the EML were made by spin coating and subsequently evaporating LiF (or PFN by spin coating) and Al as cathode. Polymer films show a very low roughness (~ 1-2 nm), as most of the polymers used in OLEDs. Due to **PMC 300\*** excellent properties like high solubility, very high QY, high conjugation and mechanical characteristics, OLEDs based on this new modified polymer (with emission in green-yellow wavelengths) showed luminances up to 1937 cd/m<sup>2</sup>, high current efficiencies of 35 cd/A and a maximum external quantum efficiency (EQE<sub>max</sub>) of 2.6 %. Additionally, preliminary tests of flexible OLEDs by using this polymer are currently carrying out, results are promising.

**Keywords:** OLEDs, Polymer, PMC 300\*, flexible OLEDs, solution process Times Roman, image area, acronyms, references

## 1. INTRODUCTION

The first OLED device, manufactured by Tang and Van Slyke<sup>1</sup>, was made with a small organic molecule and since then many other materials have been developed to be applied in those devices, not only small organic molecules but also polymers. Polymers can be deposited by solution processes because they generally have properties such as good solubility and film forming capacity, which lowers the manufacturing costs of OLED devices compared to the use of low molecular weight molecules that generally require processes of evaporation for the adequate deposit and formation films<sup>2</sup>.

The development of new polymers increases day by day, due to high demand of these compounds in various fields of application. Currently, worldwide researchers are working to improve the manufacturing process of OLED devices, both in the deposition methods, and the design of new materials with high fluorescence performance that improve the spectra emission, current-voltage characteristics and therefore the OLEDs efficiency<sup>3</sup>. Polymers of particular interest are fluorinated aromatic because of their unique properties and their performance at high temperatures<sup>4,5</sup>.

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The incorporation of elemental fluorine or fluorine-containing group in polymers has been studied because its inclusion modifies several physical properties. For example, the groups trifluoromethyl (-CF<sub>3</sub>) (3F) (used in our new polymer **PMC 300\***) and hexafluoroisopropylidene [-C(CF<sub>3</sub>)<sub>2</sub>] (6F) are the most used for structural modifications of different high-performance polymers, thus achieving various properties. The use of these fluorine groups in the polymer chains improves solubility, thermal stability (attributed to the strong CF bond), resistance to oxidation, glass transition temperature, environmental stability and optical transparency<sup>6</sup>. Likewise, their inclusion reduces crystallinity, the dielectric constant and the water absorption<sup>7</sup>. These fluorine groups have the so-called “fluorine effect” because they help to improve the solubility compared to their non-fluorinated counterparts, consequently improving their processability and making them optimal for use in a wide variety of applications, such as optical devices<sup>8,9</sup>. Therefore, the inclusion of groups -CF<sub>3</sub> and -C(CF<sub>3</sub>)<sub>2</sub> in high performance polymers, as the poly(arylene ether)s, polyimides, polyamides, polybenzazoles and related polymers, is of great interest<sup>4,5</sup>.

Based on the previous discussion, and motivated by the excellent properties of the already reported PF-1<sup>3,10</sup>, such as intense and broad emission band, solubility at high concentrations and easy processability into thin solid films of good quality, **PMC 300\*** has improved properties such as increased electroluminescence and processability when is used as EML in OLED devices. The modification consisted in the inclusion of a CF<sub>3</sub> group to improve properties such as solubility, thermal stability, resistance to oxidation, environmental stability and reduced water absorption. Then, with this new polymer, it was manufactured OLED devices with two different architectures (see Figure 1): ITO/PEDOT:PSS/**PMC 300\***/LiF/Al and ITO/PEDOT:PSS/**PMC 300\***/PFN/Al, depositing the polymers PEDOT:PSS, **PMC 300\*** and PFN by using the spin coating technique, while lithium fluoride (LiF) and aluminium (Al) were deposited by vacuum evaporation. Devices that showed the best efficiencies were those that had LiF, reaching an EQE<sub>max</sub> up to 2.6 %, a maximum luminance of 1937 cd/m<sup>2</sup> and a maximum current efficiency of 35 cd/A. On the other hand, those devices that were deposited with PFN instead of LiF showed a higher luminance: 2452 cd/m<sup>2</sup>, which is about 25 % more than those that had LiF, however, the current densities were much higher: up to 800 mA/cm<sup>2</sup>, and therefore, the maximum current efficiency: 17.9 cd/A and the EQE<sub>max</sub>: 1.3 %, decreased.

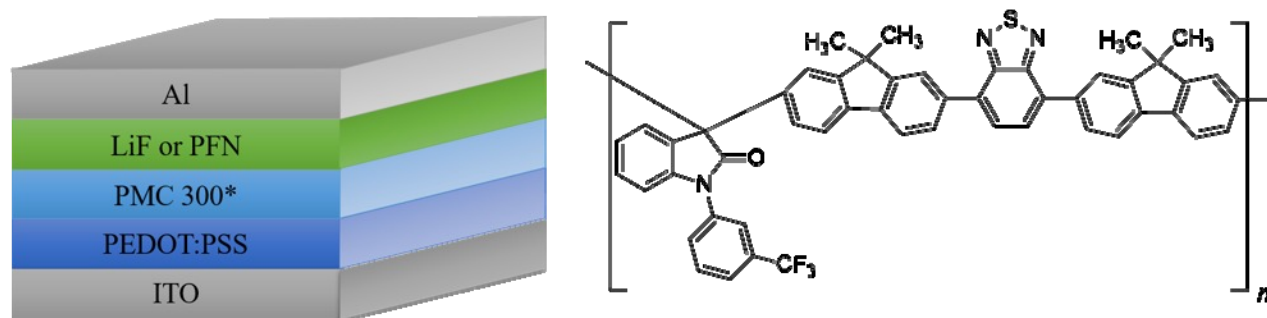


Figure 1. Used architecture for the manufacturing of our OLED devices and molecular structure of the new polymer **PMC 300\***.

## 2. EXPERIMENTAL

### 2.1 OLED devices

OLED devices were fabricated on glass substrates coated with indium-tin oxide ITO, which were cleaned with soap, acetone and ethanol in a sonicator. Plasma-oxygen treatment was given for 10 min. Poly(2,3-dihydrothieno-1,4-dioxin)-poly(styrenesulfonate) (PEDOT:PSS, CLEVIOS P VP Al 4083) was deposited by spin-coating on the substrate, with a thickness of 30-40 nm, a thermal annealing at 120 °C was provided for 20 min to remove the residual solvent. Emissive layers (EML) were deposited by spin-coating, inside of a controlled nitrogen atmosphere, at different speed to reach the best performance for each EML (60-70 nm and 80-90 nm of thickness). **PMC 300\*** was dissolved in chlorobenzene at a concentration of 6 mg/mL. The emissive layers received thermal annealing at 80 °C for 20 min. Evaporation of 1 nm thickness of LiF was at the rate of 0.01-0.03 Å/s and the other electron injector film poly[(9,9-bis(3'-(N,N-dimethylamino)propyl)-2,7-fluorene)-alt-2,7-(9,9-dioctylfluorene)] (PFN) was deposited by spin coating (10 nm of thickness). Finally, 150 nm of Al were evaporated at a rate of 0.05 Å/s for the first 30 nm, 0.08 Å/s for the next 70 nm and at 1-1.5 Å/s for the last nm.

For flexible OLED devices, the highly conducting poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) PEDOT:PSS (with a brand name PH1000, Clevios Heraeus Co.) was deposited by spin coating technique on flexible substrates (common acetate) after a plasma-oxygen treatment. A thermal annealing at 100 °C was provided for 20 min to remove the residual solvent. PEDOT:PSS, **PMC 300\***, LiF and Al were deposited in the same way as for devices on rigid substrates.

## 2.2 OLEDs characterization

The current density versus voltage (J-V) plots and luminance efficiency versus voltage (L-V) curves were measured simultaneously using a power supply (Keithley 2400, Cleveland, Ohio) with an in-house-designed and calibrated detection system. The J-V curve was recorded by direct processing of data acquired from the used Keithley 2400 power supply. Luminous density was estimated through the voltage delivered by a photodiode located at fixed distance from the OLED. Photodiode calibration was performed by using the luminance of commercial LEDs, at different wavelengths and considering all geometrical parameters involved in the detection system. Signal was previously quantified by a highly sensitive lux meter and correlated with the photodiode voltage response. All data acquisition routines were automated by using LabVIEW software specially designed for this purpose<sup>3</sup>.

## 3. RESULTS

The absorption spectra in solid state film for **PMC 300\*** polymer are shown in Figure 2, with a maximum absorption wavelength  $\lambda^{abs}$  at 318 nm. This absorption peak centered at 318 nm is attributed to the  $\pi$ - $\pi^*$  transition of the conjugated chain. Likewise the spectra of fluorescence in solution (Figure 2) show its maximum peak  $\lambda^{em}$  at 533 nm for **PMC 300\***. Fluorescence quantum yield in solution (chloroform) and in solid state were determined to be close to one (0.95 and 0.9 respectively).

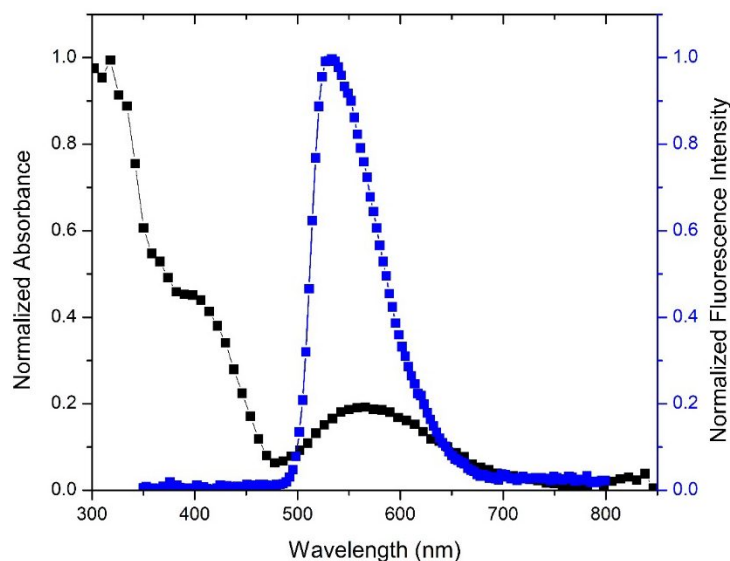


Figure 2. Absorbance in film and fluorescence in solution for **PMC 300\*** polymer.

In Figure 3 (left) the photoluminescence spectra of two films of PF-1 and **PMC 300\*** are shown when are excited by a lamp emitting in the UV. As can be seen, the solid state fluorescence of **PMC 300\*** films is slightly higher than that of PF-1. However, spectra are almost identical (Figure 3 (left)), as if it were the same polymer, therefore, the inclusion of the CF<sub>3</sub> group does not influence the photoluminescence spectrum in any remarkable way. Electroluminescence spectrum of OLED based on **PMC 300\*** polymer as emissive layer is shown in Figure 3 (right) and it has its maximum peak at 533 nm.

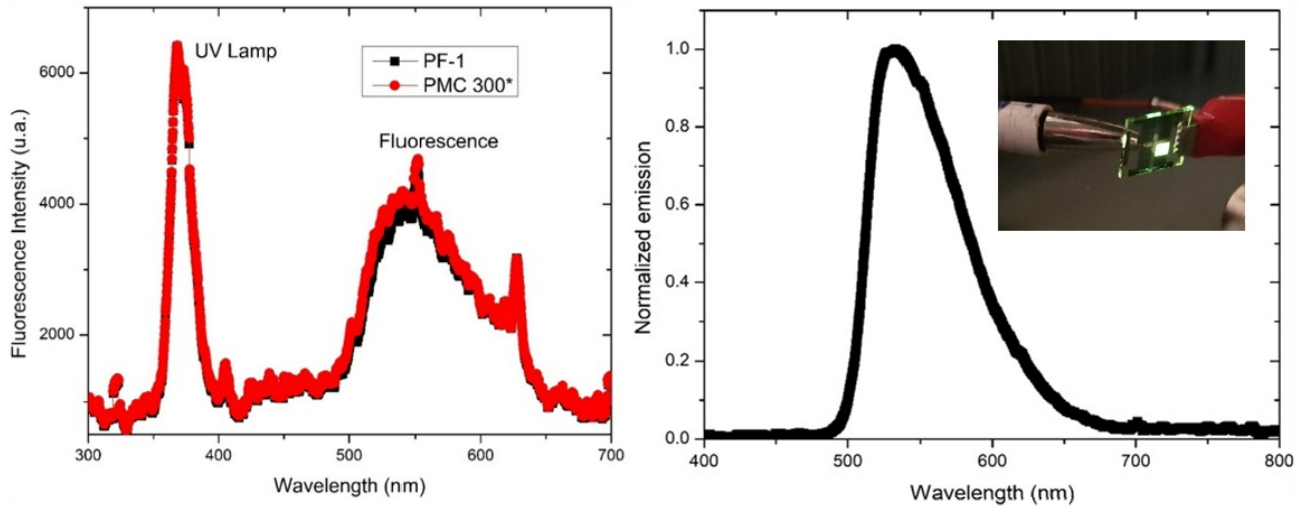


Figure 3. Relative comparison of fluorescence spectra for PF-1 and **PMC 300\*** film deposited by spin coating technique and being excited by an UV lamp (left), electroluminescence spectrum and photograph of one OLED based on **PMC 300\*** polymer as EML (right).

Current-voltage (J-V) curves and luminances (L-V) of OLEDs based on **PMC 300\*** polymer with the architecture ITO/PEDOT:PSS/**PMC 300\***/LiF/Al are shown in Figure 4 (left) while current and luminous efficiencies in Figure 4 (right). Devices based on polymer **PMC 300\*** with film thickness in the range 60-70 nm presents a turn on voltage of 3.6 V, as well a maximum luminance of 1927  $\text{cd/m}^2$  (@6.2 V) at a current efficiency of 1.8  $\text{cd/A}$ , while having a maximum efficiency of 14.8  $\text{cd/A}$  at 1688  $\text{cd/m}^2$  (at a maximum luminous efficiency of 12  $\text{lm/W}$ ) Furthermore, OLEDs with exactly the same architecture but different thickness for **PMC 300\*** (80-90 nm) presents a turn on voltage of 3.8 V, a maximum luminance of 1937  $\text{cd/m}^2$  at 7.8 V (@3.2  $\text{cd/A}$ ) and, a maximum current efficiency of 35.3  $\text{cd/A}$  at 1638  $\text{cd/m}^2$  (with a maximum luminous efficiency of 27  $\text{lm/W}$ ).

Also, in Figure 5 (left) the J-V curves and luminances are displayed for OLEDs devices based on polymer **PMC 300\*** with the architecture ITO/PEDOT:PSS/**PMC 300\***/PFN/Al while current efficiencies and luminous efficiencies are shown in Figure 5 (right). Devices based on polymer **PMC 300\*** with film thickness in the range 60-70 nm presents a turn on voltage of 3.8 V, as well a maximum luminance of 2842  $\text{cd/m}^2$  (@5.8 V) at a current efficiency of 0.7  $\text{cd/A}$ , while having a maximum efficiency of 10.1  $\text{cd/A}$  (maximum luminous efficiency of 8  $\text{lm/W}$ ) of at 2038  $\text{cd/m}^2$ .

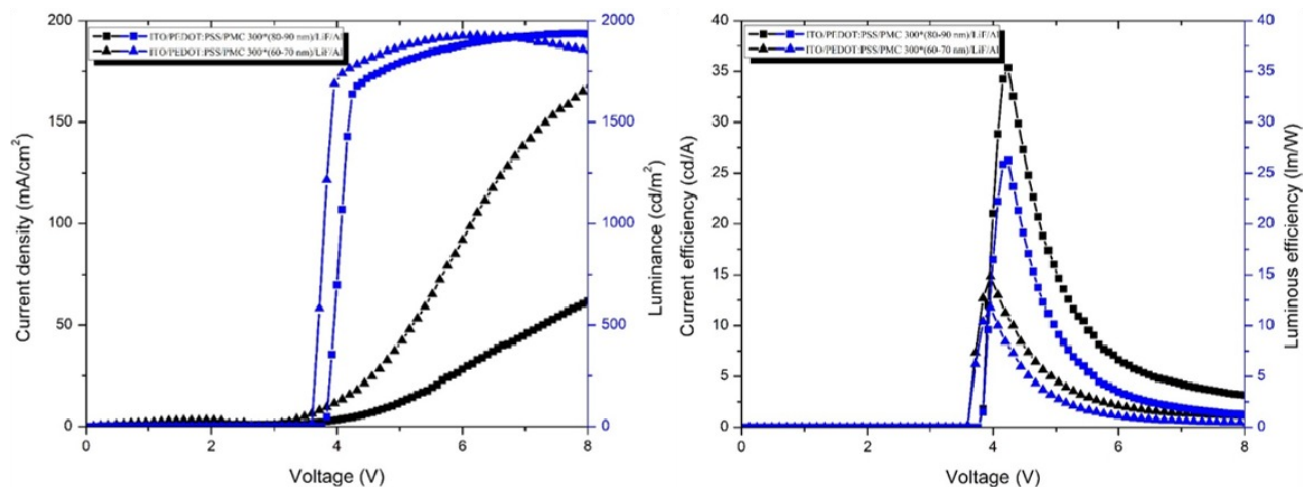


Figure 4. J-V curves, luminances (left), current efficiency and luminous efficiency (right) of OLEDs based on polymer **PMC 300\*** with the architecture ITO/PEDOT:PSS/**PMC 300\*** (60-70 nm or 80-90 nm)/LiF/Al.

Furthermore, OLEDs with exactly the same architecture but different thickness for **PMC 300\*** (80-90 nm) presents a turn on voltage of 4 V, a maximum luminance of 2452 cd/m<sup>2</sup> at 6.1 V (@3.8 cd/A) and, a maximum current efficiency of 17.9 cd/A at 1706 cd/m<sup>2</sup> (with a maximum luminous efficiency of 12 lm/W).

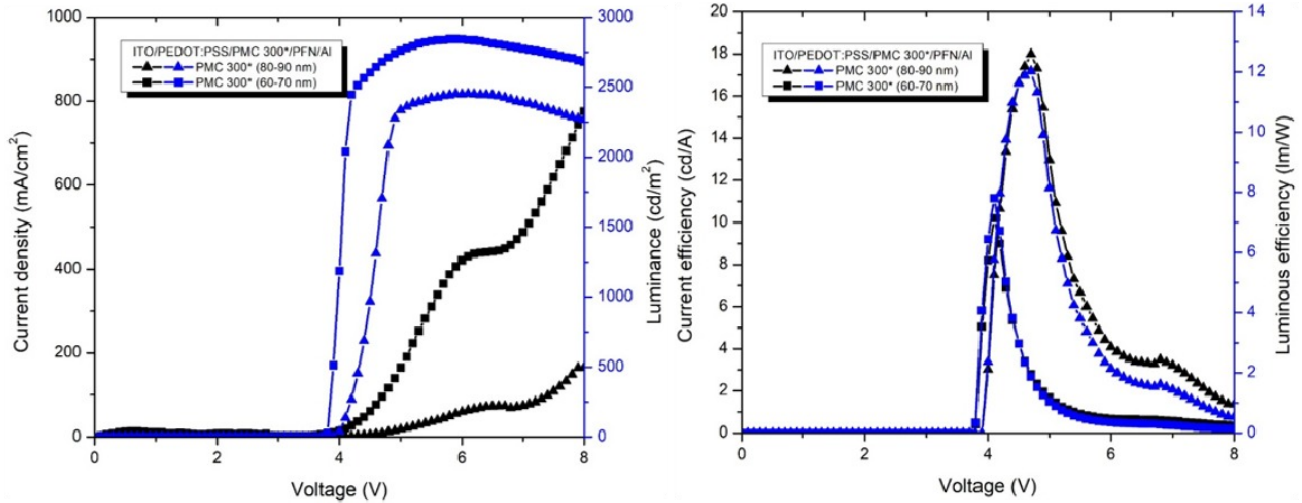


Figure 5. J-V curves, luminances (left), current efficiency and luminous efficiency (right) of OLEDs based on polymer **PMC 300\*** with the architecture ITO/PEDOT:PSS/**PMC 300\*** (60-70 nm or 80-90 nm)/PFN/Al.

Figure 6 shows the best EQE (%) values and photographs of OLEDs based on polymer **PMC 300\***, using LiF (upper photograph) and PFN (lower photograph). As can be seen, OLEDs having PFN have a higher luminance, but also a much higher current density than OLEDs that use LiF as the electron injection layer, practically, the EQE<sub>max</sub> of the latter doubles the value reached by the OLED that uses PFN: 2.6 % vs 1.3 %. It is very likely that there is a greater injection of electrons to the emitter layer in OLEDs that uses PFN, but the efficiency is lower, this may be a consequence of the electrons (and holes) that are not being combined in the EML and therefore they are slipping away to the next layers (electrons to the injector of holes and/or holes to the injector of electrons) and therefore the amount of charge carriers that are injected into the device is much greater than when compared to the number of charge carriers injected in OLEDs using LiF, that is why OLEDs with PFN could have a 25% higher luminance but current densities up to 800 mA/cm<sup>2</sup>.

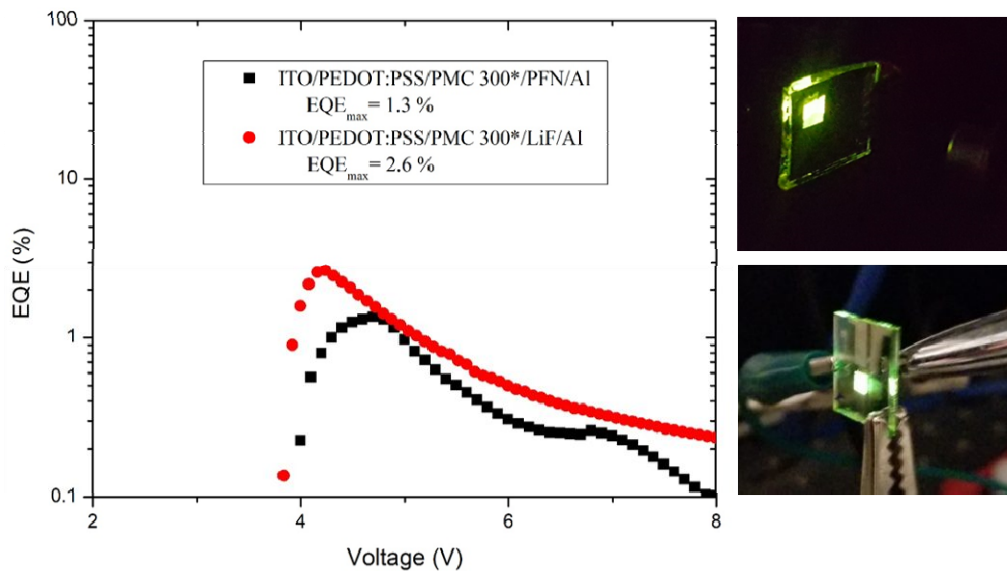


Figure 6. EQE (%) (left) and photographs (right) of OLEDs based on polymer **PMC 300\***, using LiF (upper photograph) and PFN (lower photograph).

Table 1 shows the best values obtained by our OLED devices using the ITO/PEDOT:PSS/PMC 300\*/LiF/Al and ITO/PEDOT:PSS/PMC 300\*/PFN/Al architectures compared with values obtained for other devices with emitter layers based on non-doped emitters (direct emitting layer). For example, for polymers P1 and P2<sup>11</sup>, turn on voltages of 3.7 and 4.2 V respectively have been reported, with low maximum luminances (167 and 274 cd/m<sup>2</sup> respectively, which is an order of magnitude lower than our devices) but with an excellent EQE<sub>max</sub> of 3.3 and 3.9 %, higher than in our devices but using multilayer architecture ITO/PEDOT:PSS/(P1 or P2)/TPBi/LiF/Al.

Table 1. Device performance of the two kinds (with LiF or PFN) of OLEDs based on PMC 300\* polymer as EML. Several other devices from the literature are also mentioned.

EML	V <sub>on</sub> (V)	L <sub>max</sub> (cd/m <sup>2</sup> )	V <sub>Lmax</sub> (V)	η <sub>max</sub> (cd/A)	EQE <sub>max</sub> (%)	λ <sub>max</sub> (nm)	Reference
PMC 300* <sup>a</sup>	3.8	1937	7.8	35.3	2.6	533	This work.
PMC 300* <sup>a,b</sup>	4	2452	6.1	17.9	1.3	533	This work
PF-1 <sup>b</sup>	4.5	878	--	40	--	551	<sup>3</sup>
P1 <sup>c</sup>	3.7	167	--	--	3.3	422	<sup>11</sup>
P2 <sup>c</sup>	4.2	274	--	--	3.9	401	<sup>11</sup>
Alq <sub>3</sub> <sup>c</sup>	3.6	56704	--	8.9	2.6	513	<sup>12</sup>
2-DIPO <sup>a,c</sup>	1.5	2636	7.5	1.5	--	540	<sup>13</sup>
3-DIPO <sup>a,c</sup>	2.5	2228	8	0.2	--	578	<sup>13</sup>
P5 <sup>c</sup>	3.4	2500	--	4.4	3.2	450	<sup>14</sup>
F8BT <sup>a,c</sup>	2.6	--	--	10.3	3.1	~560	<sup>15</sup>
F8BT <sup>c</sup>	2.5	--	--	12.1	3.7	~560	<sup>15</sup>
BP <sup>c</sup>	4.5	7544	--	4.2	--	436	<sup>16</sup>
Super Yellow <sup>a</sup>	2.0	77100	11	10	3.5	~540	<sup>17</sup>

EML= Emissive Layer, <sup>a</sup> LiF/Al cathode, <sup>b</sup> PFN/Al cathode, <sup>c</sup> multilayered device, V<sub>on</sub>= turn on voltage, L<sub>max</sub>= maximum luminance, V<sub>Lmax</sub>= voltage for L<sub>max</sub>, η<sub>max</sub>= maximum current efficiency, EQE<sub>max</sub>= maximum external quantum efficiency.

The well-known Alq<sub>3</sub> is reported<sup>12</sup> in a multilayer architecture (ITO/PTC-U-1hr/NPB/Alq<sub>3</sub>/LiF/Al) with an EQE<sub>max</sub> of 2.6 % and a turn on voltage of 3.6 V, values that are very similar to the those were obtained here with our OLEDs using LiF/Al cathode (EQE<sub>max</sub>= 2.6 % and V<sub>on</sub>= 3.6 V). In 2014 a series of fluorescent polymers were reported, among them, P5<sup>14</sup> was the one that showed the best performance when was used in an OLED in a multilayer architecture (ITO/PEDOT:PSS/P5/TPBi/LiF/Al), the turn on voltage was 3.4 V, a luminance of 2500 cd/m<sup>2</sup> comparable with that for our devices and an EQE<sub>max</sub> of 3.2 %. Also in 2016, an EQE<sub>max</sub> of 3.1 %, a turn on voltage of 2.6 V and a maximum current efficiency of 10.3 cd/A were achieved for an OLED device based on F8BT<sup>15</sup> emitter using a multilayer architecture (ITO/PEDOT:PSS/TFB/F8BT/LiF/Al) and a cathode of the LiF/Al type. Devices have also been reported using a relatively simple architecture (ITO/PEDOT:PSS/polymer/LiF/Al) by using the commercial material and highly reported “Super Yellow”<sup>17</sup> by using a PEDOT:PSS/GO composite layer to increase the efficiency and device performance, reaching an EQE<sub>max</sub> of 3.5 %, a very low turn on voltage of 2 V and a maximum huge luminance of 77100 cd/m<sup>2</sup>. Finally, when comparing devices based on PMC 300\* with devices based on PF-1<sup>3</sup>, it is observed maximum current efficiencies of the same order, maximum current efficiencies of 35.3 cd/A and 40 cd/A, but higher luminances 2452 cd/m<sup>2</sup> against 878 cd/m<sup>2</sup>, respectively, which can be a consequence of the inclusion of the CF<sub>3</sub> group in the new PMC 300\* polymer.

Additionally, we are using the polymer PMC 300\* for the development of flexible devices, by employing an anode based on the polymer PH1000 and 5% of DMSO as additive like is reported<sup>18</sup>, but fabricated in plastic substrates (common acetate). Preliminary tests have proved that this polymer is functional even in such simple architectures for this kind of devices (PH1000 (5 % DMSO)/PEDOT:PSS/PMC 300\*/LiF/Al) with low turn on voltage 3-4 V, and current densities lowers than 50 mA/cm<sup>2</sup> (Figure 7).

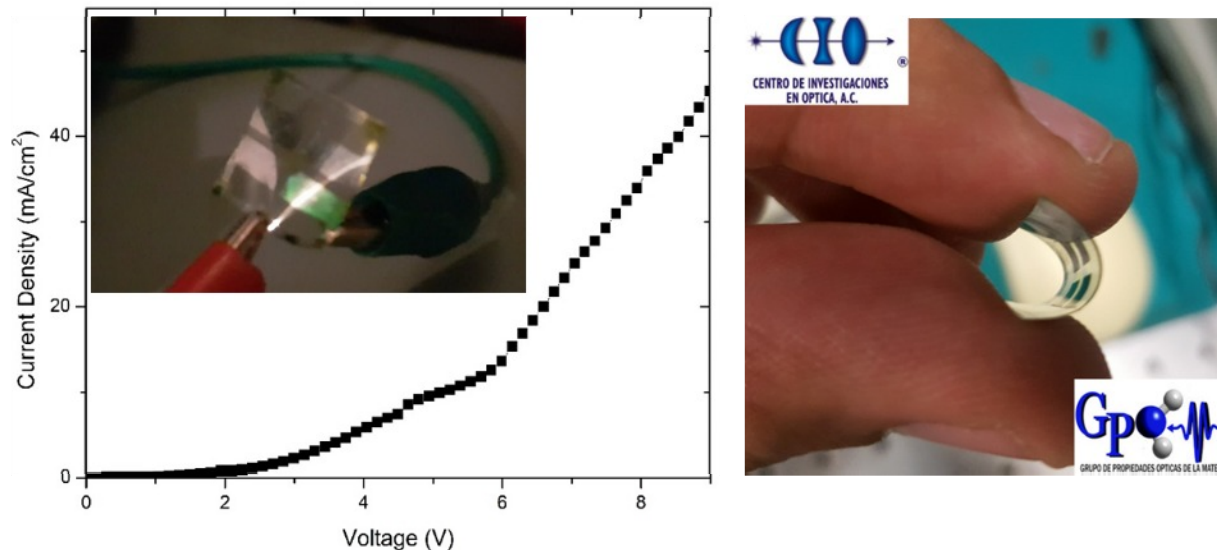


Figure 7. Flexible OLED device based on **PMC 300\*** with the architecture PH1000 (5% DMSO)/PEDOT:PSS/PMC 300\*/LiF/Al made on an simple acetate.

#### 4. CONCLUSIONS

This new polymer **PMC 300\*** showed  $EQE_{max}$  of up to 2.6 %, maximum current efficiency of 35.3 cd/A and maximum luminances of 2452  $cd/m^2$  (this value with PFN as EIL) for OLED devices, even with architectures relatively simple: Anode/HIL/EML/EIL/Cathode. The inclusion of the  $CF_3$  group had a positive influence on the devices when compared with those based on the PF-1 polymer (maximum current efficiency of 40 cd/A and maximum luminance of 878  $cd/m^2$ ), since an improvement in the luminance of the devices and an external quantum efficiency was achieved. In addition, it is worth to mention the high solubility that the material presents, the high quantum yield (in solid and solution), the mechanical resistance noticed in flexible OLED devices and the easy deposit through the spin coating technique.

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