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Annealing effects on conductivity and optical properties of the PANi layer in ITO/PAni/PPV+DBS/Al polymer light-emitting diodes

L C Poças¹, S A Travain², J L Duarte³, R A Silva¹, José A Giacometti⁴ and Alexandre Marletta^{1,5}

¹ Instituto de Física, Universidade Federal de Uberlândia, CP 593, 38400-902 Uberlândia—MG, Brazil

² Departamento de Física, Instituto de Ciências Exatas e Biológicas, Universidade de Ouro Preto, R Diogo de Vasconcelos 122, 35.400-000 Ouro Preto—MG, Brazil

³ Departamento de Física, Universidade Estadual de Londrina, CP 6001, 86051-990 Londrina—PR, Brazil

⁴ Faculdade de Ciências e Tecnologia, Universidade do Estado de São Paulo, CP 467, 19060-900, Presidente Prudente—SP, Brazil

E-mail: marletta@ufu.br

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Abstract

In this work we present the electrical and optical characterization of polymer light-emitting diodes (PLEDs) using indium–tin oxide (ITO), polyaniline (PAni), poly(*p*-phenylene vinylene) (PPV) + dodecylbenzenesulfonate (DBS) and aluminum (Al). To minimize the conversion temperature and reduce the structural defects of PPV layers, we introduced the counter-ion DBS in the PPV precursor polymer. The best PLED electrical properties were achieved at the PPV conversion temperature of 150 °C. Under this condition, the ITO/PAni/PPV+DBS/Al PLED operating voltage decreases to less than a third of the value obtained with the conventional structure ITO/PPV/Al. In addition, the electrical conductivity increases and the thermal degradation decreases in the PAni layer. The line shape of the PPV electroluminescence spectrum shows no influence of the PAni layer at relatively low electrical field (12 MV m⁻¹).

1. Introduction

Poly(*p*-phenylene vinylene) (PPV) conjugated polymer is a promising candidate for optoelectronic applications as an active layer in electroluminescent devices, such as polymer light-emitting diodes (PLEDs) [1]. This is possible due to significant progress in the synthesis route of PPV via a soluble non-conjugated precursor polymer, poly(xylylidene tetrahydrothiophenium)—PTHT [2]. After processing, the PTHT films are converted into PPV

⁵ Author to whom any correspondence should be addressed.

films using a thermal elimination step to eliminate the lateral group tetrahydrothiophenium, generally at elevated temperatures ($>200\text{ }^{\circ}\text{C}$). However, this procedure is not compatible with PLED fabrication, in which materials with different thermal properties could be employed. In addition, during the PTHT thermal treatment, chemical sub-products (HCl) react with the hole-injector and transparent electrode (indium–tin oxide, ITO) forming indium chloride (InCl_3). This sub-product degrades the ITO film and diffuses into the ITO/PPV interface forming an oxidized layer [3]. The final result is a non-intentional type of PLED with a Schottky potential barrier in the interface electrode/polymer that increases the operating voltage and decreases the electroluminescence efficiency of the diode [4]. In order to minimize these effects, the introduction of polymeric layers between the ITO and the polymeric active layer has been adopted for PPV and its derivative materials, in the device design. For poly[2-methoxy-5-(2'-ethylhexyloxy)-1,4-*p*-phenylene vinylene] (MEH-PPV), Yang and Heeger have utilized doped polyaniline (PAni) as a transparent anode in the ITO/PAni/MEH-PPV/Ca device [5]. This structure increases the performance of the PLED by about 30%–40% and decreases the operating voltage by about 30%–50% in comparison with the conventional device ITO/MEH-PPV/Ca. Another transparent and non-electronic polymer system, poly[(3,4-ethylenedioxythiophene):poly(styrene sulfonate)] (PEDOT:PSS), has been employed with similar results, i.e., the multi-layered device has shown enhanced electroluminescence efficiency [6]. However, these polymers usually present thermal and electrical degradation at $\sim 100\text{ }^{\circ}\text{C}$ and, therefore, they are not compatible with PPV processing at high conversion temperature ($\sim 300\text{ }^{\circ}\text{C}$) and PLED fabrication.

Alternatively, the PPV can be obtained at a lower temperature using the dodecylbenzenesulfonate (DBS) ion as a counter-ion of the PTHT polymer, with the advantage of acting as a less aggressive leaving group [7]. The gain in this procedure is the short conversion time ($\sim 30\text{ min}$) and relatively low temperatures ($\sim 110\text{ }^{\circ}\text{C}$), under atmospheric environment, without compromising the PPV optical properties. The low conversion temperature opens the possibility of using the PPV as the active layer in multi-layer organic semiconductor devices. Recently, combining PPV thermal conversion at lower temperature and the use of poly(sodium 4-styrenesulfonate) as the counter-ion in a layer-by-layer procedure, Ogawa *et al* in [8] showed the possibility of producing photocells with higher conductivity and photovoltaic properties. Following this idea and using doped poly(*o*-methoxyaniline) (POMA—PAni derivative), we have shown, in [9], the enhancement of the electrical properties of PLEDs such as ITO/POMA/PPV+DBS/Al. In this case, the operating voltage decreases by about 30% in comparison with the conventional ITO/PPV/Al device. The new hole-injector electrode, ITO/POMA or ITO/PAni [5], reduces the hole potential barrier between ITO and the highest occupied molecular orbital (HOMO) state (for both PPV and its derivative MEH-PPV) from $\sim 180\text{ meV}$ to $\sim 75\text{ meV}$. Analogous data were found in a previous work for the ITO/PAni/PPV+DBS/Al device [10]. However, a study of the optimization of the processing and temperature effects on the PAni layer, during the PPV thermal conversion, was not performed in that work. In this work we have addressed the electrical and optical characterization of the PAni layer in an ITO/PAni/PPV+DBS/Al PLED and of the doped and non-doped PAni layer as a function of the PPV thermal treatment temperature (120, 150 and $200\text{ }^{\circ}\text{C}$). For PLEDs, the electroluminescence spectrum is shown, and the ITO/PPV/Al and ITO/PPV+DBS/Al structures were utilized as a reference.

2. Experimental details

The substrates, glass (BK7) covered with indium–tin oxide (ITO) and gold (Au), were washed using the RCA procedure and dried in N_2 flux [11]. Doped PAni films were deposited using

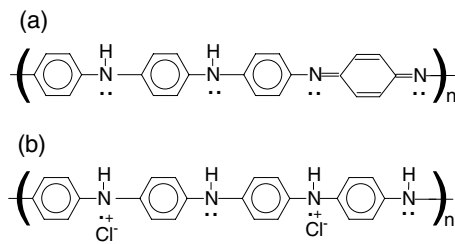


Figure 1. Chemical structure of PANi monomer in (a) emeraldine base form (insulator) and (b) emeraldine salt form (conductor).

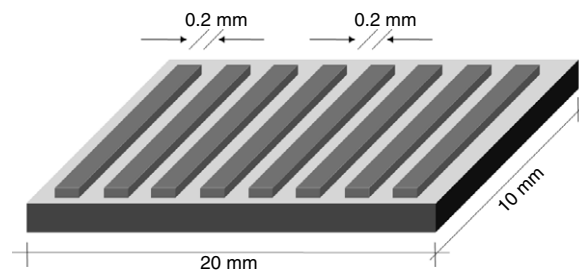


Figure 2. Layout of Au electrodes (hole-injector device) used to measure the conductivity of the PANi as a function of the temperature.

an *in situ* method during the polymerization process [12]. Film synthesis steps included: (1) aniline purification by distillation, (2) preparation of solutions at room temperature: solution A—5 ml of aniline and 300 ml of HCl (1 M) and solution B—2.88 g of ammonium peroxydisulfate ((NH₄)₂S₂O₈) dissolved in 200 ml of HCl (1 M), and (3) PANi deposition in the substrate. In a beaker containing solution A and the substrates, solution B was added for 40 min, under shaking at 0 °C. Finally, the films were washed with ultrapure water to remove the excess of material. The final thickness of the layer is 200 nm. In this work, conducting (PANi) and insulating (N-PAni) PANi samples were used; their chemical structures are presented in figures 1(a) and (b), respectively. The N-PAni films were obtained from *in situ* PANi film submitted to NH₄OH (0.1 M) flux for 15 min.

The effects of PPV thermal conversion on the PANi and N-PAni films were studied using the hole-injector electrodes shown in figure 2; this procedure was the same as that used in [9]. The 20 pairs of Au electrodes of 100 nm thickness were deposited on BK7 substrate with the channel (separation between the electrodes) equal to 0.2 mm. PANi films were deposited *in situ* on Au electrodes as described above. The PTHT or PTHT+DBS spin-coating films were added on the Au/PAni and Au/N-PAni films. The samples were heated at a 1 °C min⁻¹ rate in a homemade heating camera coupled with a Gefram 3000 temperature controller. The PANi and N-PAni conductivities were obtained from current–voltage (*I*–*V*) curves as a function of temperature, using Ohm’s law and the hole-injector device geometrical parameters [9]. The *I*–*V* curves (not shown in this paper) were obtained using a Keithley 238-Voltage Source Unit *I*–*V* tracer contacted by two probes. The electrical contact between parallel Au electrodes was made using Au wires and silver paint.

PPV films were obtained by a thermal conversion process of its precursor polymer (PTHT) according to the chemical synthesis route described in [2]. PTHT and PTHT+DBS films were deposited on the substrates by the spin-coating technique. In the case of the PTHT+DBS films,

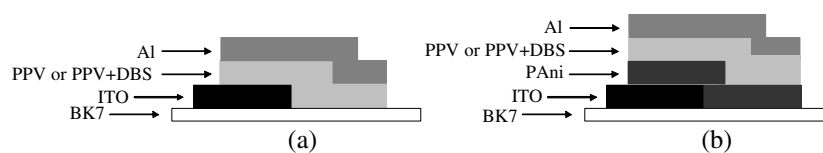


Figure 3. Layout of polymer light-emission diodes: (a) ITO/PPV(or PPV+DBS)/Al and (b) ITO/PAni/PPV(or PPV+DBS)/Al.

Table 1. Thermal conversion temperature (T_{conv}) and thermal conversion time (t_{conv}) for polymer light-emission diodes (ITO/PPV/Al, ITO/PPV+DBS/Al, ITO/PAni/PPV/Al and ITO/PAni/PPV+DBS/Al).

Device	PAni	PPV	DBS	T_{conv} ($^{\circ}\text{C}$)	t_{conv} (min)
PPV200	—	X	—	200	120
PAniPPV200	X	X	—	200	120
DBS120	—	X	X	120	30
DBS150	—	X	X	150	30
DBS200	—	X	X	200	30
PLED120	X	X	X	120	30
PLED150	X	X	X	150	30
PLED200	X	X	X	200	30

we have adopted the concentration ratio 1:1 (mol:mol) of PTHT:DBS, following reference [7]. In order to obtain homogeneous films, we have utilized a solution of PTHT with concentration equal to 2 mg ml^{-1} .

Figure 3 shows the layout of the devices processed and studied in this work. The I - V curves were obtained using a Keithley 238-Voltage Source Unit I - V tracer and the electrical contacts on ITO and Al electrodes were made using Au wires and silver paint. The PLEDs were kept in a closed-circuit helium cryostat under a vacuum of 10^{-4} Torr.

Table 1 presents the composition, the thermal conversion temperature (T_{conv}) and the thermal conversion time (t_{conv}) used in the PLEDs. The ITO/PAni/PPV+DBS/Al diodes were processed at 120, 150 and 200°C for 30 min. For comparison purposes, PPV200 (ITO/PPV/Al) and PAniPPV200 (ITO/PAni/PPV/Al) conventional devices, thermally treated at 200°C for 120 min were made, and the ITO/PPV+DBS/Al diodes were processed at 120, 150 and 200°C , using the fast thermal conversion process for 30 min. The PPV and PPV+DBS films thicknesses were about 200 nm. Thermal treatment of samples was carried out under vacuum (10^{-2} atm).

The ultraviolet-visible (UV-vis) absorption measurements were performed using a Hitachi U-2001 spectrophotometer. The electroluminescence (EL) and photoluminescence (PL) spectra were studied at room temperature and the corresponding signals were measured in the 480–700 nm range using a photomultiplier (S1) mounted on a 0.5 m Spex monochromator, through a standard lock-in technique. The electroluminescence measurement was carried out using direct polarization, continuous current (CC) mode with intensity equal to 1.0 mA and electric field intensity equal to 12 MV m^{-1} . The samples were kept in a closed-circuit helium cryostat under a vacuum of 10^{-4} Torr.

3. Results and discussion

The thermal treatment of PAni may considerably reduce its electrical conductivity and its properties as a hole-injector electrode. This may introduce an insulating layer between the

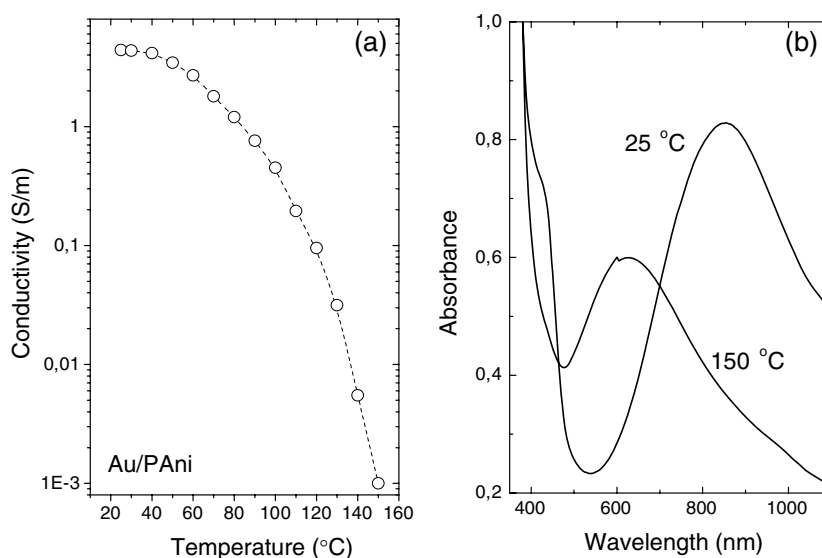


Figure 4. (a) Electrical conductivity of the PAni layer on Au electrodes as a function of the sample temperature. (b) Absorbance spectra in the UV-vis-NIR region before (25 °C) and after (150 °C) the thermal treatment.

cathode and the polymeric active layer in the PLEDs [9]. In the case of PPV, the devices are, usually, processed at temperatures above 200 °C, in vacuum, for a long time (~6 h). To show the influence of the temperature on the electrical properties of the PAni layer deposited *in situ* on Au electrodes (Au/PAni), we present figure 4(a). For absorption characterization in the UV-vis-NIR (NIR: near-infrared) region, the spectra were obtained at room temperature for the sample in two different conditions: before (25 °C) and after (150 °C) the thermal treatment. These spectra are displayed in figure 4(b). As the temperature increases from 25 to 150 °C, the electrical conductivity of the PAni layer (figure 4(a)) shows a substantial decrease from 4.4 to 10⁻³ S m⁻¹ and the polaronic band, initially centered at 825 nm in the absorbance spectra (figure 4(b)), undergoes a blue-shift of 200 nm. These results are related to the changes on the PAni structure, mainly above 100 °C, where the secondary doping [13] and the cross-linking [14] are simultaneous and competitive processes, and they result in N-PAni (insulating) at the end of the heating process. In addition, the PAni degradation is not favorable to the PPV emission due to the substantial increase in the absorbance coefficient in the spectral range between 500 and 650 nm (figure 4(b)).

In order to verify the temperature effects on the electrical conductivity of PAni spin-coating films covered with PTHT or PTHT+DBS, we present in figure 5(a) a graph of conductivity as a function of temperature.

For the Au/PAni(PTHT) and Au/PAni(PTHT+DBS) samples, the curve profiles are not significantly dependent on temperature, in comparison with the experiment described in figure 4(a), and one observes that the initial conductivity values do not change when the PTHT or PTHT+DBS films are added on Au/PAni hole-injector devices. As the temperature increases, the conductivity of the PAni films increases gradually and, above 80 °C, this parameter decreases slowly; the final values are higher than that observed for Au/PAni (figure 4(a)). This dependence can be correlated with three simultaneous processes: (a) PAni secondary doping (~100 °C) [13], (b) PAni dedoping (>110 °C) and/or cross-linking [14], and (c) PAni co-doping via a PPV thermal conversion route (>100 °C), which will be described

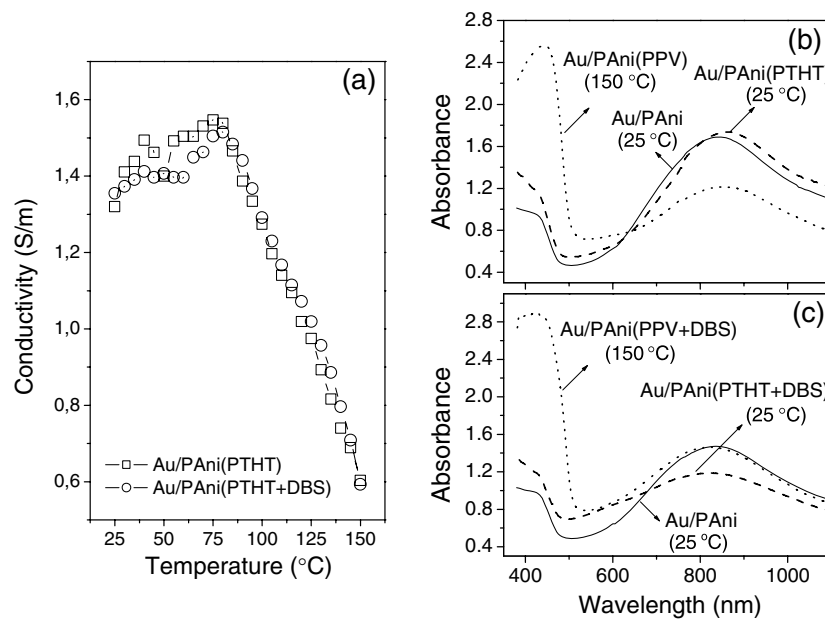


Figure 5. (a) Electrical conductivity of a doped PANi layer on Au electrodes and covered with PTHT and PTHT+DBS films as a function of the sample temperature. (b) Absorbance spectra in the UV-vis-NIR region before (25 °C) and after (150 °C) the thermal treatment of the Au/PAni(PTHT) device. (c) Absorbance spectra in the UV-vis-NIR region before (25 °C) and after (150 °C) the thermal treatment of Au/PAni(PTHT+DBS) device.

later. It is important to observe that the introduction of DBS does not change these properties much, which is very different from our previous results, obtained from hole-injector devices using POMA [9]. This can be explained by considering the difference between the PPV processing techniques: spin-coating in this case, and layer-by-layer in [9], both using PTHT and DBS. For spin-coating films, the DBS molecule is randomly bound on the PTHT matrix. This bond makes the DBS diffusion into the PPV matrix and in the interface ITO/PPV difficult. Besides, the PANi is less sensitive to the co-doping by a functional acid [15]. Figures 5(b) and (c) show the absorbance spectra obtained at room temperature for Au/PAni(PTHT) and Au/PAni(PTHT+DBS) hole-injector devices in two different conditions: before (25 °C) and after (150 °C) the thermal treatment. As was expected, the polaronic absorption bands of PANi do not change considerably with the thermal treatment. Below 500 nm, the non-localized transitions $\pi \rightarrow \pi^*$ from PPV, which appear at the end of the thermal process (150 °C), are perceptible, for both cases. The PANi polaronic band is, initially, centered at 841 nm, and it undergoes a red-shift of 20 nm for the Au/PAni(PTHT) structure. This can be correlated with the HCl diffusion from PTHT solution (pH = 5.0) during the PTHT film processing. However, this effect is not important for the PANi doping status. Using a PTHT+DBS layer, the PANi polaronic band does not change, and it is centered in 834 nm (25 °C). In both cases, PANi/PTHT and PANi/PTHT+DBS, the absorption bands undergo a blue-shift of approximately 10 nm after the thermal treatment of the film (150 °C). From these results, we can conclude that the PPV or PPV+DBS layers protect the PANi layer from electrical degradation, i.e., oxidative process, dedoping and cross-linking, at high temperatures. These effects can contribute to the breaking of the PANi effective conjugation length, substantially reducing the PANi electrical conductivity, as shown for the Au/PAni device in figure 4.

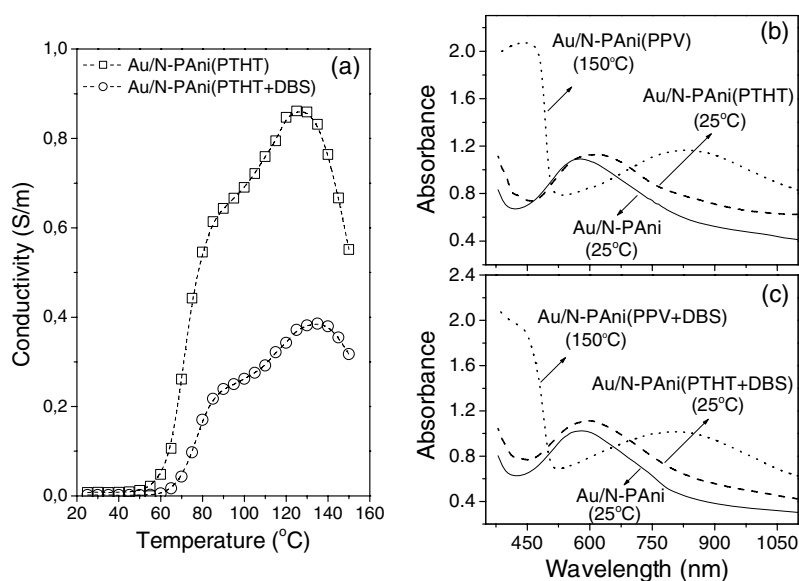


Figure 6. (a) Electrical conductivity of an N-PAni layer on a Au electrode covered with PTHT and PTHT+DBS films as a function of the sample temperature. (b) Absorbance spectra in the UV–vis–NIR region before (25 °C) and after (150 °C) the thermal treatment of the Au/PAni(PTHT) device. (c) Absorbance spectra in the UV–vis–NIR region before (25 °C) and after (150 °C) the thermal treatment of the Au/PAni(PTHT+DBS) device.

To confirm the importance of the PAni co-doping and the thermal protection effects, figure 6(a) displays the electrical conductivity curves as a function of the temperature for two PAni undoped (N-PAni) films, deposited on Au electrodes (figure 2). The films were recovered with PTHT (Au/N-PAni(PTHT)) or PTHT+DBS (Au/N-PAni(PTHT+DBS)) layers.

For both cases, the N-PAni electrical conductivity increases abruptly after 60 °C. This is indicative of the beginning of the PTHT thermal elimination process and the HCl and/or DBS diffusion in the interface with an N-PAni layer (co-doping). This observation is corroborated by the fact that the PAni secondary doping process and the thermal degradation only begins at about 100 °C. Above 80 °C, the conductivity starts increasing more slowly due to the onset of the PAni thermal degradation. The lower conductivity for the Au/N-PAni(PTHT+DBS) device demonstrates the difficulty of the occurrence of DBS diffusion and the PAni co-doping effect and corroborates the results presented in figure 5. Comparing this device with the POMA system, in [9], where the thermal degradation is not observed up to 225 °C, the PAni layer covered with PTHT (or PTHT+DBS) possesses a lower thermal stability, at approximately 150 °C. This is an important observation for a PLED layout using material with distinct thermal properties. Figure 6(b) shows the absorbance spectra for Au/N-PAni, Au/N-PAni(PTHT) and Au/N-PAni(PPV) films, and figure 6(c) presents the absorbance spectra for Au/N-PAni, Au/N-PAni(PTHT+DBS) and Au/N-PAni(PPV+DBS). In both cases, the PAni polaronic band is centered, initially, at 576 nm and undergoes a red-shift of 40 nm for the PTHT layer and 20 nm for the PTHT+DBS layer. This is in agreement with the higher facility for the HCl diffusion process to occur in PTHT solution in comparison with DBS. After the thermal treatment at 150 °C, there is observed, in both curves, an increase in the absorbance intensity at about 450 nm, characteristic of the PPV non-localized electronic transitions. In addition, the N-PAni polaronic band shifts to 825 nm for Au/N-PAni(PTHT)

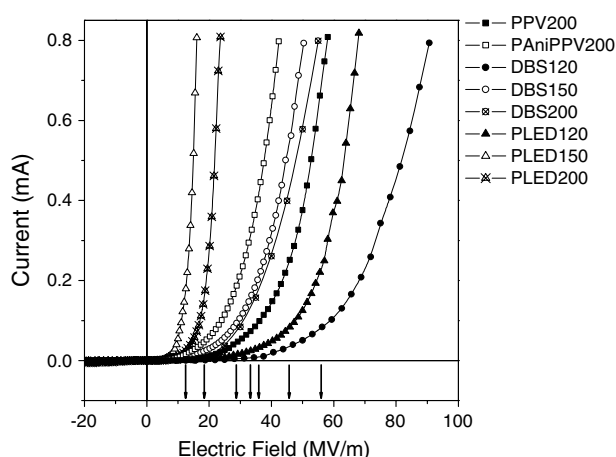


Figure 7. Characteristic I - V curves of the PLEDs: ITO/PPV/Al and ITO/PAni/PPV/Al ($T_{\text{conv}} = 200$ °C); ITO/PAni/PPV+DBS/Al ($T_{\text{conv}} = 120, 150$ and 200 °C) and ITO/PAni/PPV+DBS/Al ($T_{\text{conv}} = 120, 150$ and 200 °C).

and 805 nm for Au/N-PAni(PTHT+DBS) devices. These data are in agreement with the conductivity experiment (figure 6(a)), showing that the chemical sub-products originated from PTHT thermal elimination act as a co-doping of the PAni layer.

The characteristic curves (I versus V) of the PLEDs (table 1) are presented in figure 7. For all diodes, we observe the typical Schottky curve. Comparing the conventional devices, PPV200, with the simple PAniPPV200 PLED, the inclusion of the PAni film between the hole-injector electrode (ITO) and the active layer (PPV) lowers the operating voltage from 45.80 to 28.81 MV m^{-1} (63%). Two effects can be considered in this case: (i) the lower hole injection barrier (~ 100 meV) and (ii) the decrease of the thermal and chemical electrode degradation. Similar results were obtained for ITO/PAni/MEH-PPV/Ca [5] and ITO/POMA/PPV/Al [9] diodes, as previously mentioned. The thermal protection effect, observed in this work, is an important result, which allows the reduction of the PPV oxidative process in the ITO/PPV interface due to the HCl diffusion during the PTHT thermal treatment. Using PPV+DBS as an active layer (DBS120, DBS150 and DBS200), the operating voltage decreases by about 53% as the T_{conv} increases from 120 to 200 °C. This enhancement occurs with this process because the conjugation degree [7] and the electrical conductivity increase substantially for PPV, i.e., the transition from insulator to semiconductor is observed. It is important to remark that the increase in the thermal conversion temperature up to 200 °C does not improve the device's performance. Above 150 °C, a substantial increase in the number of structural defects is observed along the PPV main chain, such as carbonyl groups [7]. Therefore, the operating voltage for DBS150 and DBS200 diodes is similar to that for the conventional PPV200 PLED (figure 7).

Finally, we analyze the results obtained from the PLEDs which were processed making use of the properties observed for PAni and PPV+DBS layers: hole-injection barrier reduction and low-temperature conversion of PPV, respectively. The operating voltage of the PLED150 shows a 72% decrease from the conventional PPV200 PLED (from ~ 45.80 MV m^{-1} to ~ 13 MV m^{-1}). In addition, even for diodes obtained at high temperature (200 °C), this reduction is also obtained. From these results, we can state that the PAni layer acts as a protection layer for the ITO electrode during the PPV thermal conversion besides reducing

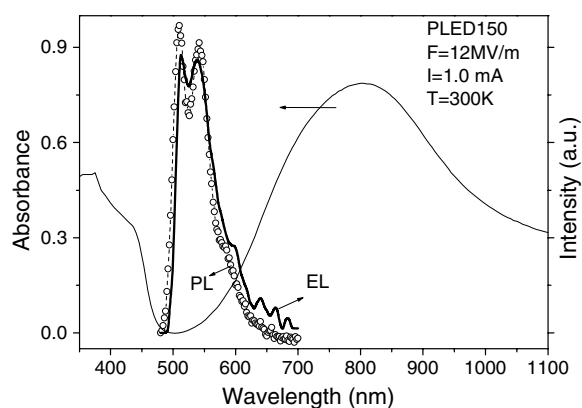


Figure 8. Spectra of absorbance of the PAni layer and of photoluminescence and electroluminescence of PLED150.

the hole energy barrier. Allied to this result, the DBS act as a less aggressive counter-ion for PTHT, decreasing the PPV thermal conversion temperature and, consequently, the number of structural defects. All these facts are responsible for the enhancement in the performance of the diode.

Figure 8 shows the photoluminescence (PL) and electroluminescence (EL) spectra for the PLED150 device at room temperature, which confirms the emission properties of the PPV active layer.

We have observed in this plot that the photoluminescence and electroluminescence spectra are similar, i.e., they have the same characteristics in terms of peak position and line width. This means that the emission processes have the same origin in the PPV layer. In addition, the PAni absorption spectrum presents an optical ‘window’ in the 450–600 nm range, approximately, which is totally compatible with the PPV emission.

4. Conclusions

In summary, we have presented the optical and electrical characterization of PPV polymer light-emitting diodes with ITO/PAni as the hole-injector and transparent electrode. The PAni films were synthesized and processed *in situ*. The PPV active layer was obtained by a low-temperature conversion procedure substituting the chloride counter-ion of the PTHT by DBS. The analysis of electrical conductivity and absorption curves of the Au/PAni and Au/N-PAni hole-injector devices show the co-doping effects from the PPV thermal conversion. In addition, we could observe that the PPV acts as a protector for the PAni layer against thermal degradation effects, i.e., the oxidative process and cross-linking, principally. The enhancement observed in the electrical properties of PAni was lowered when DBS was used as a lateral group in the PTHT polymer. Nevertheless, when making use of this process, the optical properties of the PPV are conserved due to the lower conversion temperature. The inclusion of a PAni layer between the ITO electrode and the PPV active layer has caused a significant reduction in the hole potential barrier and in the operating voltage for the PLEDs. The best result was obtained for the ITO/PAni/PPV+DBS/Al structure, with a 72% reduction in operating voltage, for T_{conv} 150 °C, in comparison with the conventional ITO/PPV/Al PLED. This condition is suitable for processing multi-layer polymer films with different thermal properties. Finally, the EL measurement was obtained using a direct and constant current (1.0 mA) that reveals a satisfactory efficiency of the device PLED150.

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