



Different electrode anodes used in OLED devices

Diferentes eletrodos anodos usados em dispositivos OLEDs

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ABSTRACT

Transparent conductive oxides (TCOs) known as indium tin oxide (ITO) and fluorine tin oxide (FTO) deposited on glass were compared by different techniques and also as anodes in organic light-emitting diode (OLED) devices with same structure. ITO produced at laboratory was compared with the commercial one manufactured by different companies: Diamond Coatings, Displaytech and Sigma-Aldrich, and FTO produced at laboratory was compared with the commercial one manufactured by Flexitec Company. FTO thin films produced at laboratory presented the lowest performance measured by Hall effect technique and also by I-V curve of OLED device with low electrical current and high threshold voltage. ITO thin films produced at laboratory presented elevated sheet resistance in comparison with commercial ITOs (approximately one order of magnitude greater), that can be related by a high number of defects as discontinuity of the chemical lattice or low crystalline structure. In the assembly of OLED devices with ITO and FTO produced at laboratory, neither presented luminances. ITO manufactured by Sigma-Aldrich company presented better electrical and optical characteristics, as low electrical resistivity, good wettability, favorable transmittance, perfect physical-chemical stability and lowest threshold voltage (from 3 to 4.5 V) for OLED devices.

KEYWORDS: Indium tin oxide, Fluorine tin oxide, Organic light-emitting diode, I-V curve, Threshold voltage.

RESUMO

Óxidos transparentes condutivos (TCOs) conhecidos como óxido de índio e estanho (ITO) e óxido de estanho e flúor (FTO) depositados em vidros foram comparados por diferentes técnicas e também como anodos em dispositivos de diodo orgânico emissor de luz (OLED) com mesma estrutura. ITO produzido em laboratório foi comparado com o ITO comercial de diferentes empresas: Diamond Coatings, Displaytech e Sigma-Aldrich; e FTO produzido em laboratório foi comparado com o FTO comercial fabricado pela empresa Flexitec. Filmes finos de FTO produzidos em laboratório apresentaram mais baixos desempenhos medidos pela técnica de efeito Hall e também pela curva I-V do dispositivo OLED com baixa corrente elétrica e elevada tensão de limiar. Filmes finos de ITO produzidos em laboratório apresentaram elevada resistência de folha em comparação com ITOs comerciais (aproximadamente uma ordem de grandeza mais elevada), o que pode estar relacionado com a alta quantidade de defeitos como descontinuidade das cadeias químicas ou baixa cristalinidade da estrutura. Na montagem de dispositivos OLEDs com ITO e FTO produzidos em laboratório, nenhum apresentou luminâncias. O ITO fabricado pela empresa Sigma-Aldrich exibiu melhores características ópticas e elétricas, como baixa resistividade elétrica, boa molhabilidade, favorável transmitância, perfeita estabilidade físico-química e mais baixa tensão de limiar (de 3 até 4,5 V) para os dispositivos OLEDs.

PALAVRAS-CHAVE: Óxido de índio e estanho, Óxido de estanho e flúor, Diodo orgânico emissor de luz, Curva I-V, Tensão de limiar.



INTRODUCTION

Organic light-emitting diode (OLED) can be defined as organic semiconductor electroluminescent device mounted by multilayers located between two inorganic anode and cathode electrodes^{1,2}. The direct electrical polarization generates light that crosses a part of intern layers and also the insulator and transparent substrate to reach the human eyes.

In 1987, two researchers from Eastman Kodak Company called Ching Wan Tang and Steve Van Slyke developed the first monochromatic organic electroluminescent device considered with good quality of light³. Nowadays, a vast variation of different materials has provided different color emission used in displays of consumer products, such as: smartwatches, smartphones and TVs^{4,5}. These devices present some important characteristics: image with good sharpness and contrast, wide gain on vision ($\approx 180^\circ$), fidelity of color reproduction, no emission of neither ultraviolet nor infrared rays and fast time of response^{6,7}. These advantages are only found in the OLED devices compared with liquid crystal displays (LCDs), for example.

But the OLED devices are not limited only to fabric displays with polychromatic light emission. The monochromatic light has been applied on the innovative products to the lighting market for ambient and automotive area, as well^{8,9}. Monochromatic OLEDs present a method of assembly most simplified than the polychromatic and, for this reason, these devices have been very extensively investigated reaching different performances and luminances using different wavelengths¹⁰⁻¹².

In OLED structure, it has been used a multilayer concept with different materials that can be described as:

- Transparent conductive oxide (TCO) used as anode (holes injection inside the OLED devices) and deposited on glass or polyethylene terephthalate (PET, in case of flexible device) substrates. It has crucial importance, due to its favorable electrical and optical characteristics, such as: work function of ≈ 4.8 eV, low roughness surface (< 10 nm), transparency above 60%, good chemical and physical stability and low sheet resistance ($\leq 10 \Omega/\square$)¹³⁻¹⁵. Most used material is known as indium tin oxide (ITO) thin films deposited on glass substrates¹⁶⁻²¹. Another TCO that can be used as anode electrode is known as fluorine tin oxide (FTO) thin film, because it has similar electrical and optical characteristics when compared with ITO^{22,23}. The optical and electrical properties of FTO materials are very dependent on dopant (fluorine) quantity in the SnO_2 , that has direct influence on the electrical conductivity and transmittance, while in the ITO thin film production the process parameters are very known in the literature to obtain a material with good quality mixing tin and indium oxide in different percentages, as 10% of SnO_2 and 90% of In_2O_3 ²⁴⁻²⁷. The quality in TCO production is very dependent on technique and process parameters used, causing different electrical and optical characteristics, such as: transmittance, sheet resistance, carriers concentration, electrical resistance, Hall mobility, surface roughness and thickness²⁸⁻³¹. These parameters found in TCO thin film have significant influences on the final performance of the OLED devices as threshold voltage operation and electrical current³². Then, the main objective of this work are the electrical and optical analyses of different ITOs and FTOs thin films deposited on glass substrates used in the same OLED device structure;
- On the TCO layer, the deposition of an organic layer with similar electrical and optical properties of TCO anode is necessary. This material improves the charge injection inside the device causing the balancing of charges (holes and electrons). Consequently, better performance is presented. This material is called hole transport layer (HTL), and a very used commercial material is known as poly(3,4-ethylenedioxythiophene)-poly(styrene sulfonate) (PEDOT:PSS)^{33,34};
- On the HTL the electroluminescent layer (EL) that promotes the electrical charges (holes and electrons) recombination to form the exciton (light emission) is deposited^{35,36}. In the literature, several materials presenting different wavelengths have been found³⁷⁻³⁹;
- On the electroluminescent layer a thin film called as electron transport layer (ETL) is deposited. It improves electrons injection inside the emitting layer. In the literature there are several materials used as ETL^{40,41};
- On the ETL the cathode electrode is deposited using a metallic thin film to finish the assembly of OLED structure, and, in this case, aluminum has been commonly used, because it is very easy to evaporate, due to its low melting point and good electrical conductivity^{42,43}.

MATERIALS AND METHODS

Production of ITO thin films at laboratory

The initial and exploratory process to fabric ITO thin films manufactured at laboratory used microscopic glass slides as substrate with geometry of 2.5×7.5 cm and ITO (90% of indium oxide and 10% of tin oxide in mass) pellets manufactured by Kurt J. Lesker Company.

Preparation was carried out by reactive thermal evaporation using oxygen atmosphere inside the chamber. After deposition, the ITO thin films presented opacity, then the samples were heated at room temperature and laminar air flow to obtain good transmittance. The process parameters used were: oxygen flow rate at 100 sccm; pressure inside the chamber at 1.10^{-3} torr; electrical current at 120 A; process time from 5 to 10 minutes; and temperature at 350°C.

Production of FTO thin films at laboratory

The initial and exploratory process to fabric FTO thin films manufactured at laboratory also used glass slides as substrate at the same geometry of ITO/glass. The FTO thin films were produced using Reactive Sputtering DC technique and deposited on microscopic glass slides with the same geometry used in the production of ITO thin films. In the equipment, there are plasma generation by high voltage of 5,000 V, electrical current of 100 mA and intern pressure inside the chamber at 8.10^{-2} torr. In the method, pure oxygen gas with 99.999% was used, as well as pure target of tin oxide (SnO_2) with 95% and polytetrafluorethylene (PTFE) plate, to introduce fluorine inside the FTO thin film formed. The process parameters used were: oxygen flow rate at 25 sccm; pressure inside the chamber at $1.9.10^{-1}$ torr; electrical current at 12.5 mA; voltage at 1,200 V; process time from 3 to 4 hours; and 2.5-cm-distance between sample and target.

Commercial ITO and FTO thin films

Commercial TCOs of different companies were analyzed. ITO thin films deposited on glass substrates were supplied at different companies: Diamond Coatings, Displaytech, and Sigma-Aldrich.

FTO thin films deposited on glass substrates were supplied by Flexitec Eletrônica Orgânica company.

Before optical/electrical analyses and OLED devices assembling, the TCO surfaces were cleaned with current water, neutral detergent scrubbing with gloves. This procedure was complemented using a commercial product of cleaning surfaces called Aqua Brilho. Some drops were placed on the surfaces of TCOs and scrubbed with cotton until the complete solvent evaporation and remnants removal.

Measurement techniques used in TCOs

The TCO thicknesses were obtained using an equipment manufactured by Veeco company, model DekTak 6M stylus profiler. Before measurements, a part of TCO was corroded with hydrochloric acid (HCL), zinc powder and cotton to create a step between TCO and glass substrate, and each sample was measured by three times at different regions.

Parameters as sheet resistance, resistivity, carriers concentration and Hall mobility analyses were obtained using Hall effect technique with equipment manufactured by Swin Company, model Hall 8800, configured by Van der Pauw electrical circuit, permanent magnetic field at 5,300 G and electrical current at 100 mA. All parameters were obtained at the same time, and each sample was measured four times.

Transmittance analyses were obtained with equipment manufactured by Varian company, model Cary 50, measured from 200 to 800 nm and using the atmospheric air as reference. Each sample was measured four times at different regions.

Images of TCO surfaces were carried out with scattering electronic microscopy (SEM) using equipment manufactured by FEI Company. Images of 120,000, 50,000 and 25,000 X magnifications were obtained. A gold thin layer to better resolution of images was deposited on surfaces before analyses. This technique was complemented with chemical element composition analyses carried out by energy dispersion scattering (EDS).

Contact angle technique was used with some droplets of PEDOT:PSS deposited on TCO surfaces by a micro syringe. The images of droplets (no distortion) were captured using a digital camera manufactured by FujiFilm company, model S1800, configured as super macro mode. The contact angles of PEDOT:PSS were measured by left and right sides using ImageJ software. For each sample, five droplets were used.

Assembling and electrical analyses of OLED devices

All OLED devices used the same structure – glass/TCO/PEDOT:PSS/PVK/Alq₃/Al – and were assembled inside the glove box under nitrogen atmosphere and relative humidity below 20%, measured with thermo hygrometer manufactured by Minipa company, model MT-241.

In PEDOT:PSS (as HTL) and poly(*N*-vinylcarbazole) (PVK) (as EL) depositions, a spinner apparatus (developed in another work) with 3,000 rpm by 60 seconds and dried with temperature at $\approx 55^{\circ}\text{C}$ was used⁴⁴. The Alq₃ (as ETL) material was synthesized at laboratory, and Al (as cathode) was obtained commercially (pure 99.9%). Both thin films were obtained by thermal evaporation technique⁴⁵. The PEDOT:PSS was deposited as received and PVK was diluted in organic solvent as trichlorobenzene supplied by Tedia Company with concentration of 10 mg/mL⁴⁶.

Four OLED devices were mounted at the same time on each glass/TCO sample and I-V curves analyzed using a source power manufactured by Keithley Instruments, model 2400 Series, and LabTracer software 2.0 version⁴⁷. The OLED devices were not encapsulated.

RESULTS

Analyses of different TCO thin films

Table 1 shows the thicknesses for all TCOs analyzed, showing a large difference mainly between ITO of Displaytech and Diamond Coatings, revealing a difference of six times longer. This thickness result to ITO from Diamond Coatings was similar as the one presented by FTO thin film produced at laboratory, but generally the FTOs are thicker when compared with similar conductivity, as found in the ITO thin films⁴⁸.

Table 1: Thicknesses for all TCOs analyzed.

TCO types	Thickness (nm)
ITO at laboratory	228
ITO from Diamond Coatings	656
ITO from Displaytech	130
ITO from Sigma-Aldrich	140
FTO at laboratory	633
FTO from Flexitec	780

TCO: transparent conductive oxide; ITO: indium tin oxide; FTO: fluorine tin oxide.

The TCOs thickness is a very important parameter, because, if TCO is very thick, the light produced by OLED device is decreased; and if it is very thin, the threshold voltage of OLED is considerable increased. Moreover, in TCOs with thin thicknesses there is the probability of occurring electrical short-circuit or low injection of holes, leaving practically inoperative the OLED device.

The TCO thickness is related by other electrical characteristics, as sheet resistance and electrical resistance, by Eq. 1⁴⁹:

$$\rho = Rt \quad (1)$$

in which: ρ = electrical resistivity; R = sheet resistance; t = TCO thickness.

Hall effect results revealed discrepant values comparing commercial and laboratory TCOs.

In relation to the FTO produced at laboratory, it presented the highest sheet resistance value, and this aspect decreases considerably the final performance of OLED devices, as low luminance, with increase of the threshold voltage, or causes a different electrical behavior compared by common I-V curve of diode⁵⁰. Performance presented by FTO produced at laboratory can be related by its amorphous structure, interfering in the locomotion of electrical charges (as observed by low carriers concentration and Hall mobility), once a crystalline structure promotes better injection of electrical charges by fluorine in the FTO, as observed in some semiconductor materials (silicon, as example).

Table 2 shows the Hall effect results for all analyzed TCOs.

Table 2: Hall effect for all TCOs analyzed.

TCOs types	Sheet resistance (Ω/\square)	Electrical resistivity ($10^{-4} \cdot \Omega \cdot \text{cm}$)	Carriers concentration ($10^{20}/\text{cm}^3$)	Hall mobility ($\text{cm}^2/\text{V} \cdot \text{s}$)
ITO at laboratory	400 ± 110	93 ± 26.0	1.5 ± 0.2	5 ± 1
ITO from Diamond Coatings	12 ± 1	8 ± 0.6	2.6 ± 0.2	30 ± 1
ITO from Displaytech	11 ± 0	1.5 ± 0.1	10 ± 1	42 ± 3
ITO from Sigma-Aldrich	11 ± 0	1.6 ± 0	9.5 ± 0.4	41 ± 1
FTO at laboratory	$(1.1 \pm 0.1) \cdot 10^6$	69 ± 11	0.6 ± 0.5	$(5 \pm 4.8) \cdot 10^{-3}$
FTO from Flexitec	13 ± 3	10 ± 2	2.3 ± 0.4	27 ± 2

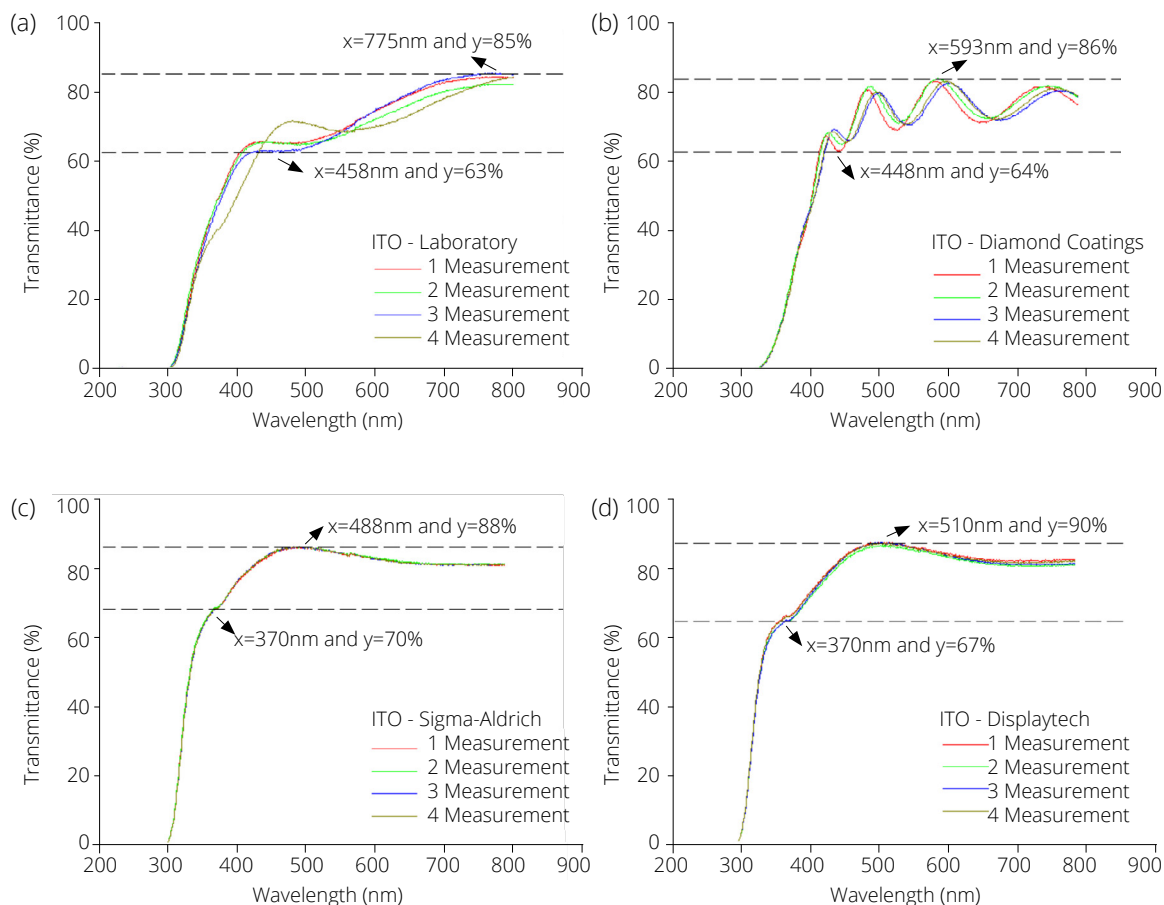
TCO: transparent conductive oxide; ITO: indium tin oxide; FTO: fluorine tin oxide.

ITO produced at laboratory also presented discrepant results (high electrical resistivity, low carriers concentration and low Hall mobility) as compared with ITO thin films manufactured by companies. The hypothesis for this behavior result can be related to its chemical composition with low quantity of dopants or oxygen vacancies, low crystalline structure and discontinuance of the chemical lattice, as well. After preparation, these TCOs presented visible appearance, as yellowish color instead complete transparency, as easily observed in commercial ITO thin films. In the literature, it is found that the visual aspect of ITO thin films is related to sheet resistance and chemical composition⁵¹. The hypothesis regarding with elevated sheet resistance found can be related to the high concentration of oxygen in the chemical composition of ITO produced at laboratory, causing low quantity of oxygen vacancies and decreasing the electrical conductivity property. The different results found by ITO and FTO produced at laboratory can be also linked to the process parameters not well known or controlled used during the fabrication, jeopardizing the homogenization.

Transmittance results presented good reproducibility to the commercial ITOs in comparison with the ones produced at laboratory. Figures 1a to 1d show the results of transmittance vs. wavelengths for ITO thin films produced at laboratory, Diamond Coatings, Sigma-Aldrich and Displaytech, respectively.

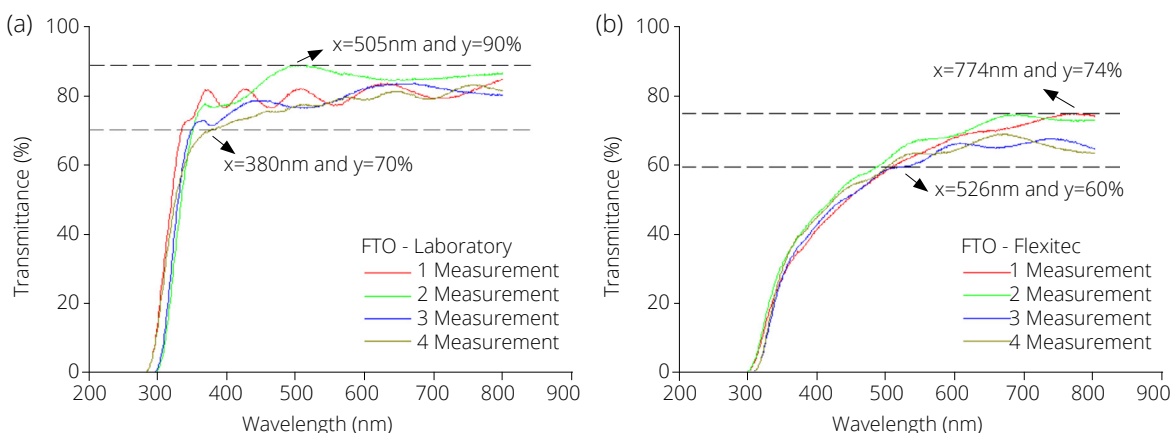
In general terms, it is possible to observe that the ITO thin film manufactured by Displaytech presented the highest transmittance, reaching maximum value of $\approx 90\%$ for maximum wavelength peak of ≈ 510 nm (with better efficiency for blue-green light), as used by OLED structure of this work (glass/TCO/PEDOT:PSS/PVK/Alq3/Al). In relation to the ITO manufactured by Diamond Coatings and compared with the one produced at laboratory, both presented maximum transmittance of $\approx 85\%$ for maximum wavelength peak of ≈ 593 nm (better efficiency for yellow-orange light) and maximum wavelength peak of ≈ 775 nm (better efficiency for red light), respectively.

FTO thin film produced at laboratory had different behavior compared with commercial FTO on the transmittance results. Transmittance of FTO produced at laboratory in Fig. 2a had the most elevated values inside the range of visible light, presenting a maximum peak of $\approx 90\%$ for maximum wavelength peak of ≈ 505 nm (with better efficiency for green light), while the FTO from Flexitec in Fig. 2b had a maximum peak of $\approx 74\%$ and a maximum wavelength peak of ≈ 774 nm (with better efficiency for red light). The percentage of fluorine and deposition of layer-by-layer to produce a thin film changes the transmittance (easily observed by visual aspect) of FTO formed as revealed the results by Mulyadi et al.⁵². Both FTO thin films presented similar transmittances in the range of visible light compared with FTO results found in the literature^{53,54}. However, overall, the FTO produced at laboratory had the lowest transmittance in the range of visible light in comparison with TCOs^{55,56}.



ITO: indium tin oxide.

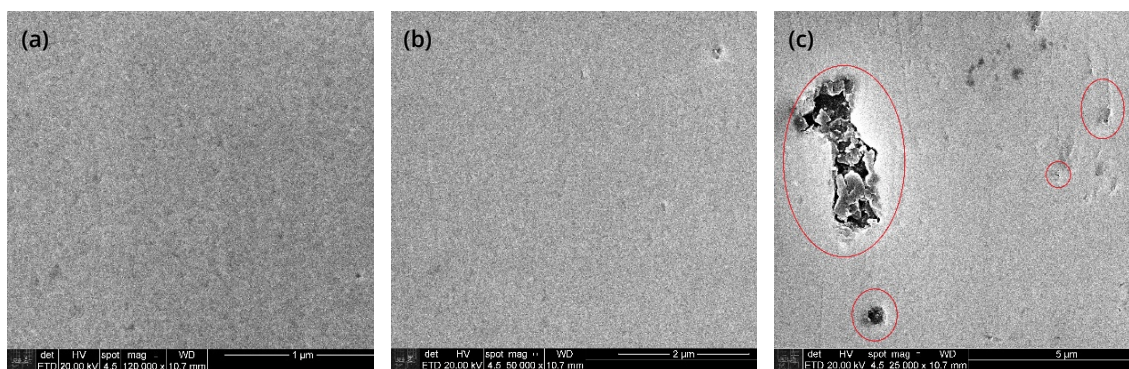
Figure 1: (a) Transmittance vs. wavelength for ITO produced at laboratory; (b) transmittance vs. wavelength for ITO from Diamond Coatings; (c) transmittance vs. wavelength for ITO from Sigma-Aldrich; (d) transmittance vs. wavelength for ITO from Displaytech.



FTO: fluorine tin oxide.

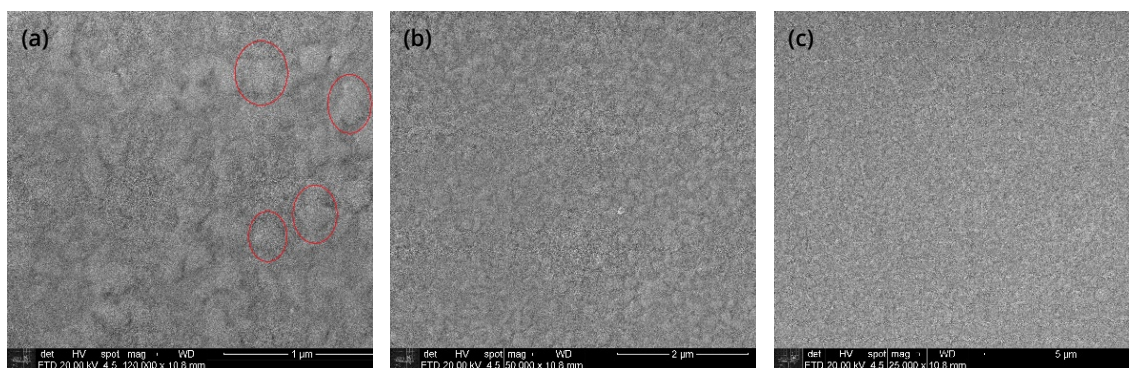
Figure 2: (a) Transmittance vs. wavelength for FTO produced at laboratory; (b) transmittance vs. wavelength for FTO of Flexitec.

SEM images of ITO thin films produced at laboratory are represented by Figs. 3a to 3c; Diamond Coatings from figs. 4a to 4c; Displaytech from Figs. 5a to 5c; and Sigma-Aldrich from Figs. 6a to 6c. ITO thin film produced at laboratory did not reveal grain contours or polycrystalline characteristics in the material structure. More visible eventual characteristic in Fig. 3d can be related to the gold thin film deposited on surface of ITO before analyses. This same figure presents some defects (indicated by red color) that probably can be related to the particles created during the manipulation or physical force caused during the cleaning method, showing ITO low chemical stability.



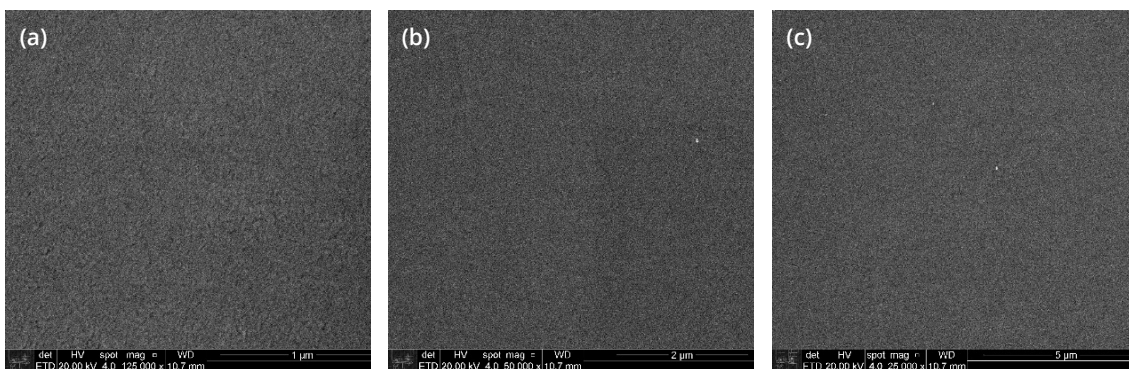
ITO: indium tin oxide.

Figure 3: Images of ITO produced at laboratory with magnification: (a) 120,000 x (with scale of 1 μm); (b) 50,000 x (with scale of 2 μm); (c) 25,000 x (with scale of 5 μm).



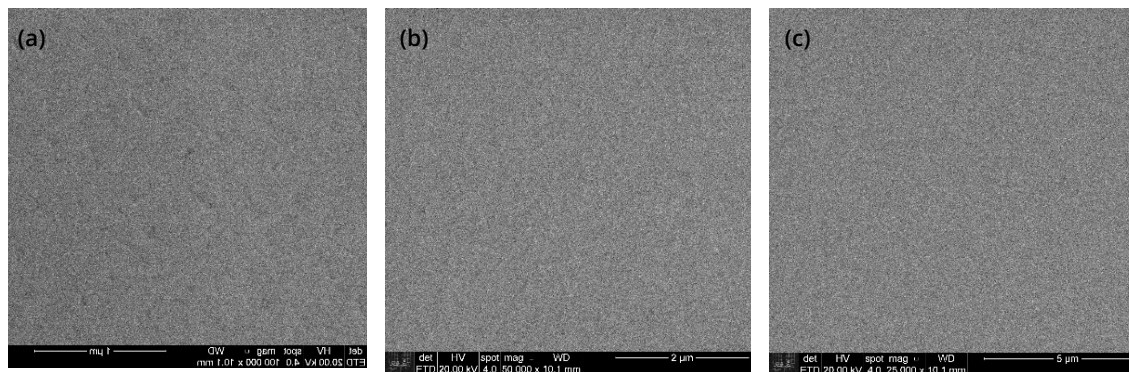
ITO: indium tin oxide.

Figure 4: Images of commercial ITO manufactured by Diamond Coatings with magnification: (a) 120,000 x (with scale of 1 μm); (b) 50,000 x (with scale of 2 μm); (c) 25,000 x (with scale of 5 μm).



ITO: indium tin oxide.

Figure 5: Images of commercial ITO manufactured by Displaytech with magnification: (a) 120,000 x (with scale of 1 μm); (b) 50,000 x (with scale of 2 μm); (c) 25,000 x (with scale of 5 μm).



ITO: indium tin oxide.

Figure 6: Images of commercial ITO manufactured by Sigma-Aldrich with magnification: (a) 120,000 x (with scale of 1 μm); (b) 50,000 x (with scale of 2 μm); (c) 25,000 x (with scale of 5 μm).

In the images of ITO manufactured by Diamond Coatings, the polycrystalline characteristic of material structure with formation of grains (indicated) is evident. Little defects were also observed on the surface, showing the material good physical stability.

ITO thin film manufactured by Displaytech revealed characteristics as little grain contours, but with imperfections (as occurred with the ITO produced at laboratory), and this characteristic can be related to the gold thin film deposited on the surface before the analyses.

SEM images obtained to the ITO thin film manufactured by Sigma-Aldrich presented similar aspect as it was seen for ITO manufactured by Diamond Coatings with little grain contours.

Table 3 shows the chemical element, percentage in mass and percentage in mols for all analyzed ITO thin films. The results show similar composition between ITO thin films produced at laboratory and the one from Displaytech. An unbalancing of chemical elements between oxygen and indium in the composition to the ITO thin film manufactured by Diamond Coatings was found, and this behavior was very different in comparison with other ITOs.

Table 3: Results obtained by EDS technique for all analyzed ITO thin films.

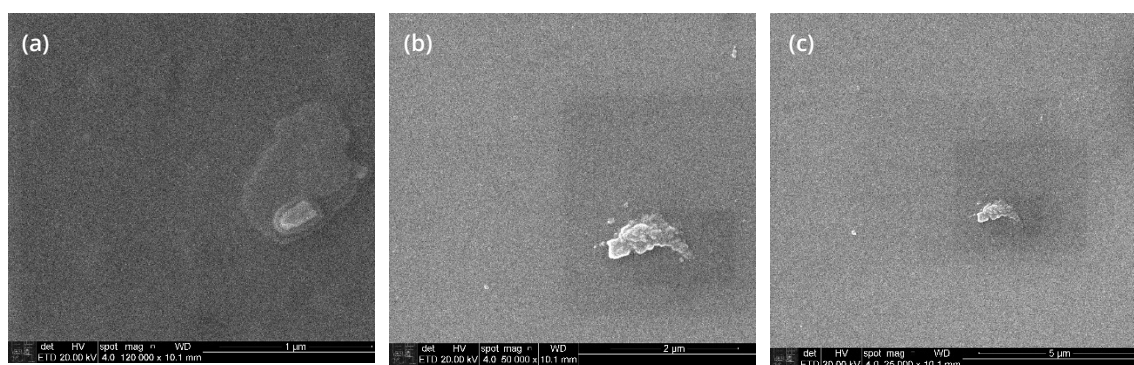
ITO produced at laboratory		
Chemical element	Percentage in mass (%)	Percentage in mols (%)
Oxygen (O)	46.92	86.45
Indium (In)	44.35	11.39
Tin (Sn)	8.73	2.17
ITO manufactured by Diamond Coatings		
Chemical element	Percentage in mass (%)	Percentage in mols (%)
Oxygen (O)	19.44	63.49
Indium (In)	71.48	32.52
Tin (Sn)	9.08	4
ITO manufactured by Displaytech		
Chemical element	Percentage in mass (%)	Percentage in mols (%)
Oxygen (O)	47	86.49
Indium (In)	43.74	11.22
Tin (Sn)	9.26	2.30
ITO manufactured by Sigma-Aldrich		
Chemical element	Percentage in mass (%)	Percentage in mols (%)
Oxygen (O)	42.62	84.30
Indium (In)	43.94	12.11
Tin (Sn)	13.45	3.59

EDS: energy dispersion scattering; ITO: indium tin oxide.

SEM images of FTO thin films produced at laboratory are represented by Figs. 7a to 7c, and the ones from Flexitec are presented by Figs. 8a to 8c.

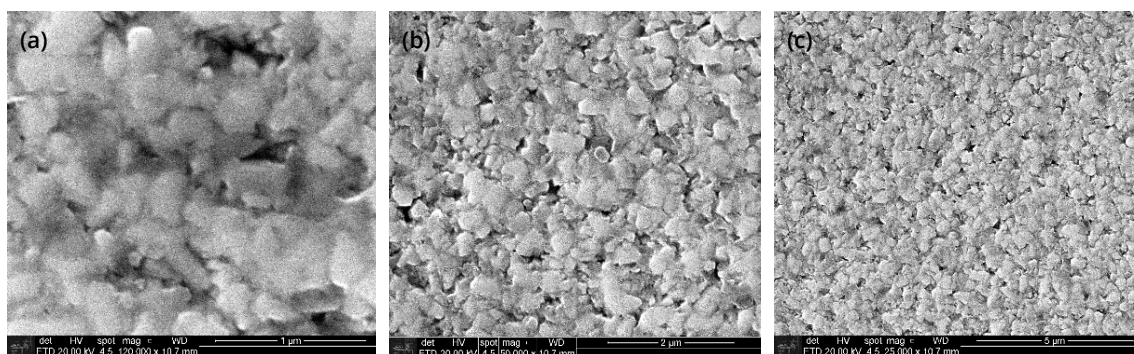
As observed by FTO produced at laboratory, the behavior of this material presented a very smooth aspect on the surface with similar results, as presented by ITO thin films. These results were very different as compared with FTO thin films manufactured by Flexitec, as seen in figs. 8a to 8c, showing the largest grain contours and the most roughness surface. There is a hypothesis that a very rough surface decreases the performance of OLED devices, due to the different regions created causing mobility difficult for charge carriers in the interface TCO/HTL created.

Another hypothesis can be related by charge carriers loss in discontinued chemical lattice.



FTO: fluorine tin oxide.

Figure 7: Images of FTO produced at laboratory with magnification: (a) 120,000 x (with scale of 1 µm); (b) 50,000 x (with scale of 2 µm); (c) 25,000 x (with scale of 5 µm).



FTO: fluorine tin oxide.

Figure 8: Images of commercial FTO manufactured by Flexitec with magnification: (a) 120,000 x (with scale of 1 µm); (b) 50,000 x (with scale of 2 µm); (c) 25,000 x (with scale of 5 µm).

Table 4 shows the chemical element, percentage in mass and percentage in mols for both analyzed FTO thin films. Comparing the commercial FTO and the one produced at laboratory, it is possible to notice that the former had low quantity of fluorine. Normally high quantity of fluorine to the FTO produced at laboratory indicates high electrical conductivity compared with FTO manufactured by Flexitec. On the other hand, the good electrical conductivity was not verified to the FTO manufactured at laboratory, as observed by Hall effect results, and the acting of dopants requires formation of crystalline structure of the material. This behavior can be confirmed by the hypothesis that the FTO produced at laboratory presents amorphous structure.

Table 4: Results obtained by EDS technique for FTO produced at laboratory and manufactured by Flexitec.

FTO produced at laboratory		
Chemical element	Percentage in mass (%)	Percentage in mols (%)
Oxygen (O)	17.17	20.19
Fluorine (F)	21.32	13.35
Tin (Sn)	61.51	73.15
FTO manufactured by Flexitec		
Chemical element	Percentage in mass (%)	Percentage in mols (%)
Oxygen (O)	12.40	49.18
Fluorine (F)	1.43	4.76
Tin (Sn)	86.17	46.06

EDS: energy dispersion scattering; FTO: fluorine tin oxide.

The contact angle revealed good scattering of PEDOT:PSS droplets on the TCO surfaces with similar values for all samples. These analyses also showed good hydrophilic of surfaces (water is used as solvent in the PEDOT:PSS), and, in this case, this presented behavior can be attributed to the good performance of the cleaning process used with commercial product instead organic solvents or other normally used methods⁵⁷⁻⁵⁹. Table 5 presents the contact angle results for TCOs, showing good wettability and similar values for all analyzed TCOs.

Contact angle measurements using distilled water droplets on the ITO surfaces were carried out by Davenas et al. and they reported similar values⁶⁰.

In another work, Morais treated the surface of ITO thin films with ultraviolet-ozone by some times and measured the contact angle of PEDOT:PSS droplets obtaining similar results⁶¹. After the cleaning method, the TCO surfaces became more reactive chemically, modifying its surface energy. For this reason, the contact angle analyses were quickly performed. This procedure avoids the fast adsorption of chemical composts on surface of TCOs that can interfere in the measurements, as observed in Morais⁶¹.

Table 5: Contact angle results for all analyzed TCOs.

TCO type	Contact angle (degree)
ITO produced at laboratory	32.9 ± 4.2
ITO from Diamond Coatings	33.4 ± 5
ITO from Displaytech	34.8 ± 2.9
ITO fromSigma-Aldrich	33.0 ± 1.7
FTO produced at laboratory	30.5 ± 2.5
FTO from Flexitec	31.2 ± 2.9

TCO: transparent conductive oxide; ITO: indium tin oxide; FTO: fluorine tin oxide.

Analyses of OLEDs with different TCO thin films

Only OLED devices using the TCOs supplied by companies presented green light emission, showing the hypothesis that the recombination of charges (holes and electrons) occurred inside the Alq₃ material (used as ETL) instead the PVK layer (used as light emitting). This aspect was also observed in the same structure of OLED mounted by Guerra⁶². This author assembled a similar structure using glass/ITO/PEDOT:PSS/PVK/Butyl-PBD (instead Alq₃)/Al, and the experiment revealed light-blue emission. Moreover, Guerra⁶² mounted the structure of OLED device with glass/ITO/PVK/Al (without Alq₃ layer), and the OLED devices presented no luminances and elevated electrical current up to 20 mA.

OLEDs mounted by Berthelot et al. used a similar structure (Mg-Ag instead Al as electrode cathode), and the devices presented green light emission⁶³.

Hebner et al. reported that the PVK layer has a function of HTL instead emissive layer in devices. Then, the hypothesis is that the PVK layer used in the structure as emitting layer glass/TCO/PEDOT:PSS/PVK/Alq₃/Al contributes as holes injection, increasing the performance of PEDOT:PSS layer and, consequently, improving the OLEDs performance⁶⁴.

Figures 9 to 14 show the I-V curves with respective OLED device turned-on for each TCO used in the structure glass/TCO/PEDOT:PSS/PVK/Alq₃/Al:

- Figures 9a and 9b: the range of threshold voltage was obtained from 3.5 to 7.5 V with low electrical current level (maximum peak of 5.5 mA only) for all devices polarized. The I-V curves for these OLEDs presented no reproducibility of results showing distinct performances;
- Figures 10a and 10b: compared with the ITO from Diamond Coatings, it promoted low electrical current level (maximum peak of 2.5 mA only) and also high threshold voltage, from 4.5 to 6.5 V. As observed by ITO of Diamond Coatings, the I-V curves presented variations with low electrical current;
- Figures 11a and 11b: the results of I-V curves presented the lowest threshold voltage, from 3 to 4.5 V, compared with all ITO thin films, and the highest electrical current peak, from 8 mA, and little variations in curves were observed. The hypothesis for this performance can be related to the percentage of tin (Sn) in the composition of ITO, that decreased the electrical resistivity, and it can be related to the threshold voltage;

- Figure 12: in comparison for all OLEDs using ITO thin films as electrode anode, only the devices with ITO produced at laboratory presented no luminances. The hypothesis for this behavior can be attributed to the elevated sheet resistance, as observed by Hall effect measurements. Then, decreases of this electrical parameter are necessary to present similar values, as observed by commercial ITOs tested. Moreover, the tangent line performed to obtain the threshold voltage is very sloped. This indicates electrical resistive characteristics in the I-V curves. The other two devices polarized showed this hypothesis, obtaining the lowest current level with elevated voltage operation;
- Figures 13a and 13b: in general terms, the I-V curves revealed low variation on the threshold voltage compared with OLED devices using commercial ITO thin films. An increase of ≈ 28 mA of the electrical current was obtained, but large variations of electrical current values were obtained as well;
- Figures 14: the I-V curves clearly show the influence caused by elevated sheet resistance (or electrical resistivity), presenting low performance of OLED devices. FTO produced at laboratory needs to have similar results as presented by commercial FTO. In this case, the chemical composition of FTO produced at laboratory is a very important factor, and it has total influence on the OLED performance to decrease the threshold voltage and increase the luminance. Then, it needs to have similar chemical composition, as found by commercial FTO, to improve its electrical and optical characteristics.

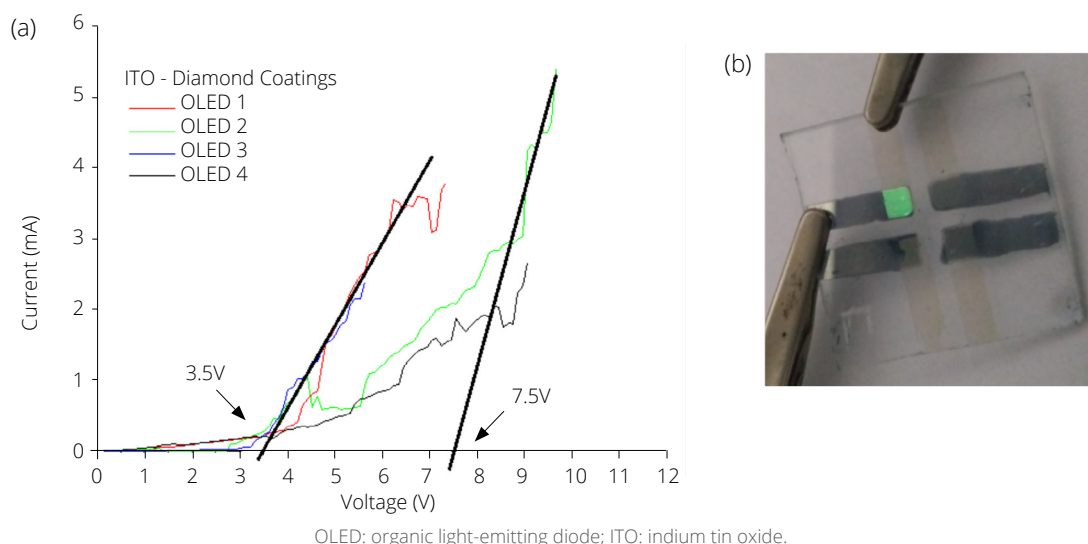


Figure 9: (a) OLEDs mounted with ITO manufactured by Diamond Coatings; (b) better result for OLED turned-on with ITO manufactured by Diamond Coatings.

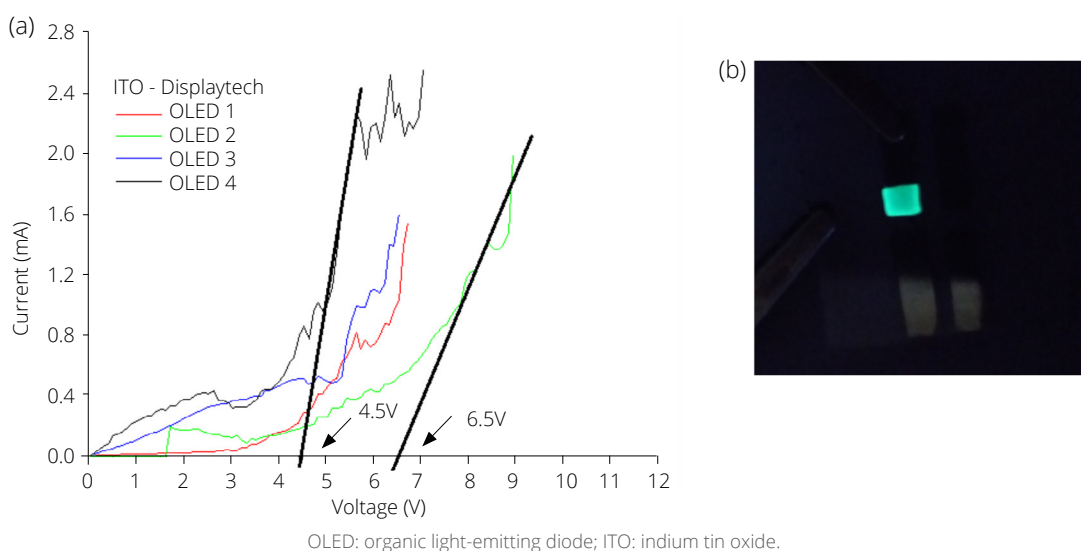


Figure 10: (a) OLEDs mounted with ITO manufactured by Displaytech; (b) better result for OLED turned-on with ITO manufactured by Displaytech.

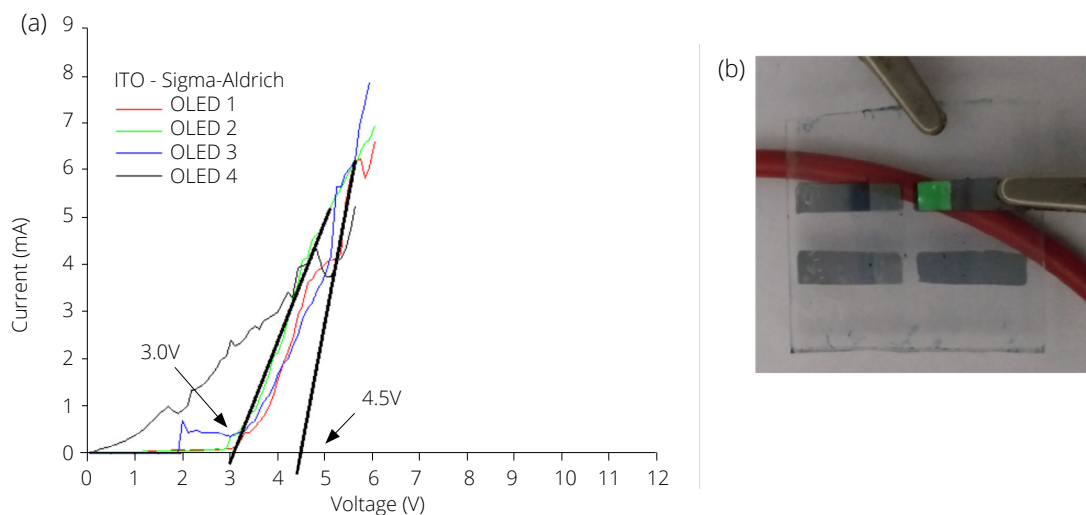


Figure 11: (a) OLEDs mounted with ITO manufactured by Sigma-Aldrich; (b) better result for OLED turned-on with ITO manufactured by Sigma-Aldrich.

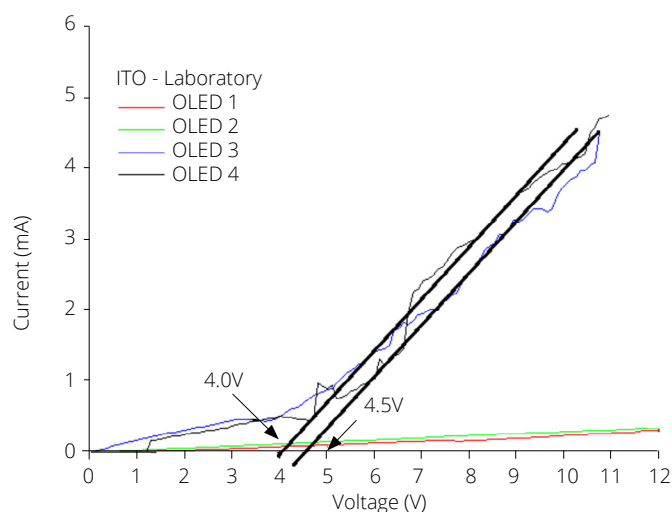


Figure 12: OLEDs mounted with ITO produced at laboratory.

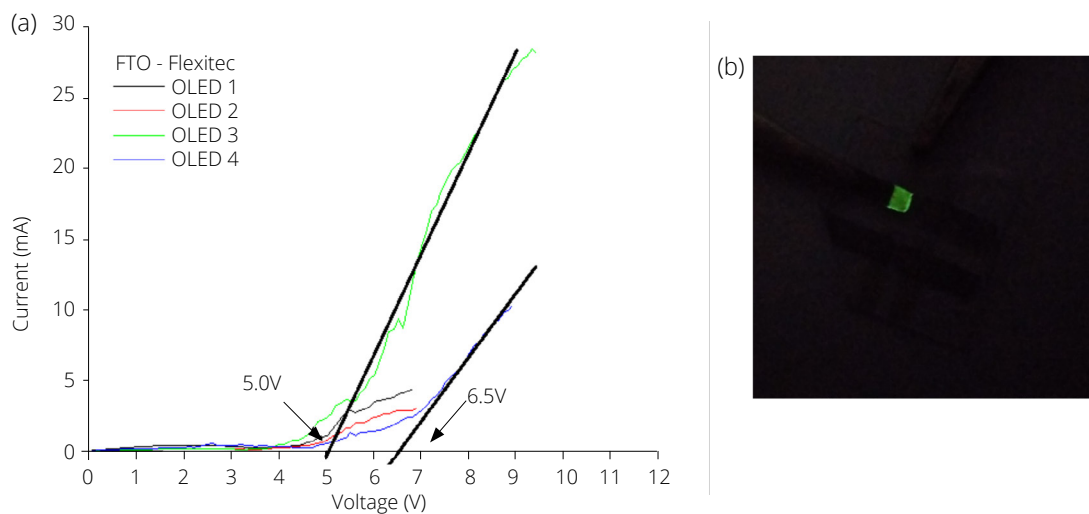
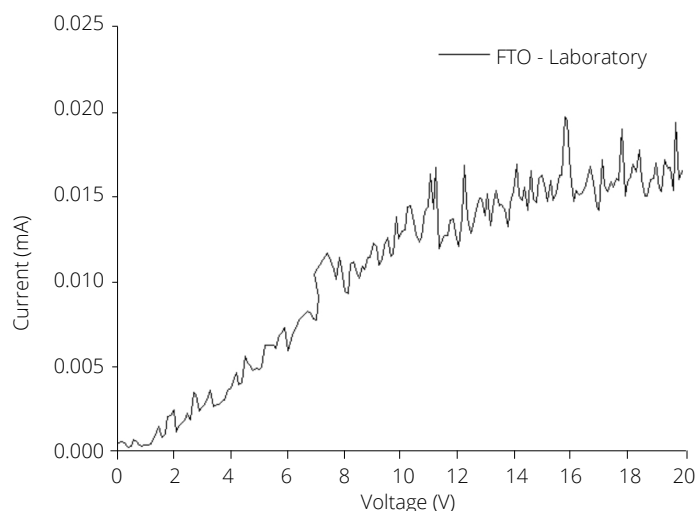


Figure 13: (a) OLEDs mounted with FTO manufactured by Flexitec; (b) better result for OLED turned-on with FTO manufactured by Flexitec.



OLED: organic light-emitting diode; FTO: fluorine tin oxide.

Figure 14: OLED mounted with FTO produced at laboratory.

CONCLUSION

Commercial and produced at laboratory TCOs known as ITO and FTO were analyzed by different techniques and compared as electrode anode in OLED devices using the same structure (glass/TCO/PEDOT:PSS/PVK/Alq₃/Al).

FTO thin films produced at laboratory presented the lowest performance compared with commercial FTO, measured by parameters in the Hall effect results, SEM images and also by I-V curve of OLED devices. This low performance can be attributed by its elevated sheet resistance and electrical resistivity, but FTO/glass is promissory, due to its great potential to substitute the ITO thin films (OLEDs, organic photovoltaics – OPVs, LCDs and other devices), that have high price offered by international companies. In addition, no company produces TCO in Brazil.

ITO thin films produced at laboratory presented elevated electrical resistivity in comparison with commercial ITOs (approximately one order of magnitude greater), what can be related to the high quantity of defects as discontinuity of the chemical lattice in the structure or low crystallinity of material.

Commercial ITO manufactured by Sigma-Aldrich Company presented better electrical and optical characteristics as: low electrical resistivity ($1.6 \Omega/\square$), good wettability (33 ± 1.7), favorable transmittance in the range of visible light (up to 80%), perfect physical-chemical stability (as observed during the cleaning method) and the lowest range of threshold voltage (from 3 to 4.5 V) in comparison with all TCOs used in the OLED devices analyzed.

In the assembling of OLED devices with ITO and FTO produced at laboratory, neither presented green light emission as obtained by commercial TCOs, due to the low performances presented by technique measurements. The hypothesis related for this green emission can be caused by Alq₃ (used as ETL) instead PVK (used as emitting material) in the structure tested, because the PVK has HTL function, as reported by literature.

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AUTHOR'S CONTRIBUTIONS

Conceptualization: Santos ER, Yuki EY, Shu Hui W; **Data Curation:** Yuki EY, Santos ER; **Formal Analysis:** Yuki EY, Santos ER; **Funding Acquisition:** Santos ER, Yuki EY, Shu Hui W; **Methodology:** Yuki EY, Santos ER; **Resources:** Santos ER, Yuki EY, Shu Hui W; **Software:** Shu Hui W; **Supervision:** Santos ER; **Validation:** Santos ER; **Visualization:** Yuki EY, Santos ER; **Writing – Original Draft:** Yuki EY, Santos ER; **Draft Preparation:** Yuki EY, Santos ER; **Writing – Review & Editing:** Yuki EY, Santos ER.

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DATA AVAILABILITY STATEMENT

Data are available in a data repository.

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