

# KARLSRUHE INSTITUTE OF TECHNOLOGY

# KARLSRUHE SCHOOL OF OPTICS AND PHOTONICS

# OPTICS AND PHOTONICS LABORATORY 2309491

# NANOTECHNOLOGY LAB OLED FABRICATION LAB

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## 1 INTRODUCTION

An organic light-emitting diode (OLED or Organic LED) is a light-emitting diode (LED) in which the emissive electroluminescent layer is a film of organic compound that emits light in response to an electric current. This organic layer is situated between two electrodes; typically, at least one of these electrodes is transparent. OLEDs are used to create digital displays in devices such as television screens, computer monitors, portable systems such as smartphones, handheld game consoles and PDAs. A major area of research is the development of white OLED devices for use in solid-state lighting applications.

In this report, the basic principles and theory of OLEDs and organic materials will be discussed in details to give a clear perspective on OLEDs. Afterwards, the steps for fabricating an OLED will be discussed. Then, a full analysis of the measured data will be provided to characterize the OLED.

# 2 Theory Of Operation

## 2.1 Charge Transport Mechanism

A typical OLED device is composed of a layer of organic materials situated between two electrodes, the anode and cathode, all deposited on a substrate.

Organic materials may be polymers or small molecules. Polymers are long-chain molecules that can be applied on a substrate by solution processing (eg. spincoating) while small molecules have small molecular weight and can be applied by vacuum sublimation. The development of polymer based devices is commercially limited, because polymers which can be dissolved in the same solvent cannot be deposited on top of each other, thus it is difficult to choose suitable polymers for OLED fabrication. However, due to their ease and low-cost of processing, OLEDs made of polymer materials are a big research interest and shall be the focus of this lab.

Certain polymers, which have alternating single and double bonds, are known as conjugated polymers. They are electrically conductive as a result of delocalization of  $\pi$ -electrons caused by conjugation over part or all of the molecule. The electrons move from one molecule to another by a hopping mechanism, which is shown in figure 1. These conjugated molecules have efficient hopping mechanisms, as the charge carriers do not have to jump from one molecule to another so often. They have conductivity levels ranging from insulators to conductors, and are therefore considered organic semiconductors. The highest occupied and lowest unoccupied molecular orbitals, Highest Occupied Molecular Orbital (HOMO) and Lowest Unoccupied Molecular Orbital (LUMO), of organic semiconductors are analogous to the valence and conduction bands of inorganic semiconductors.

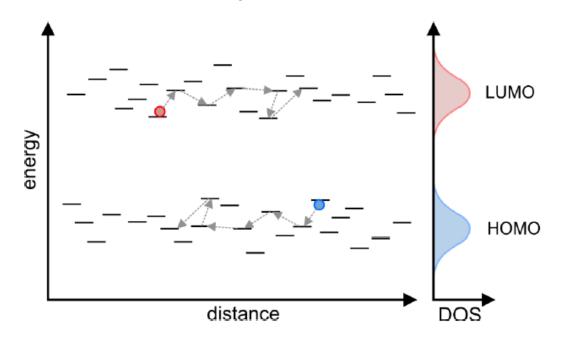


Figure 1: Charge Transport Mechanism of OLED devices.

#### 2.2 OLED Device Architecture

The OLED structure has many thin layers of organic materials, as shown in figures 2 and 5, which are explained below in detail. When direct current is applied to the electrodes, charge carriers from the anode and cathode are injected into organic layers, leading to recombination of carriers which produces radiative emission of visible light. The work function of a metal, defined as the energy difference between its Fermi level and the vacuum level, is different from the electron affinity of the organic material, which is the energy difference between its LUMO level and the vacuum level. This energy difference is the barrier for the carrier injection at the metal-organic interface. A thermally-activated electron can jump to one of these states and subsequently, undergo a hopping transport process under the influence of an external field. When an electron and a hole reach the recombination zone, they recombine to form an exciton which is a charge-less electron-hole pair that can transport energy. Their energy can be released through radiative emissions, which produces emission from the device.

The OLED devices in general contain several layers, the simple structure is made of:

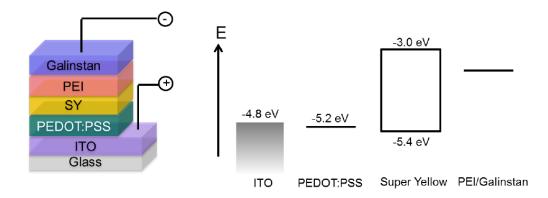


Figure 2: Device architecture of a simple OLED device with PEDOT:PSS as the hole transport layer and Super Yellow as the emitting polymer..

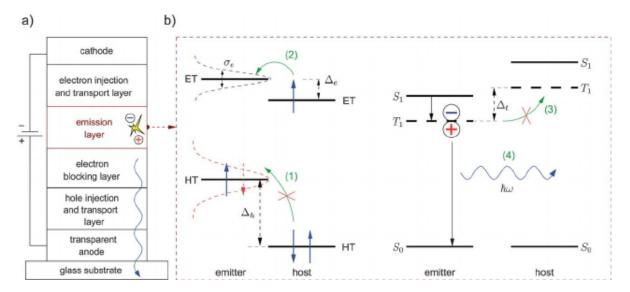


Figure 3: Functionality of different layers of OLED

- 1. Substrate Layer: This layer is a thin sheet of glass, which supports the OLED structure.
- 2. Anode Layer: This layer must be conducting and transparent with large work function. The function of this layer is to transfer holes to the organic material on top of it, or to remove electrons from the organic material. In this lab, Indium Tin Oxide (ITO) will be used as an anode.
- 3. Hole Transport (Conductive) Layer: This layer injects holes from the anode

layer to the organic emission layer on top of it. The hole and electron transport layers should be made of organic materials that are not soluble in the same material. In this lab, PEDOT:PSS is used as a hole transport layer.

- 4. Electron Transport (Emissive) layer: This layer receives electrons from cathode layer as well as holes from the hole transport layer. Electrons and holes are collected in this layer, thus, they recombine together and produce emission of light. In this lab, Super Yellow will be used as the emissive layer. Super Yellow is soluble in toluene, while PEDOT-PSS is soluble in water. Thus, they are suitable for placing them on top of each other.
- 5. Cathode Layer: Cathode layer is responsible for injection of electrons to the organic layer beneath it. It is made of a conducting metal which must have a low work function, to be less or equal than the LUMO level of the electron transport layer, to enable carrier injection. In this lab, Aluminum will be used as the cathode layer. We could also use another metal, such as Galinstan, with a surface modifier material (such as PEI) to reduce the work function of Galinstan. [1]

## **OLED** Characterization

To characterize an OLED, physical and optical technical values have to be considered. One way to determine these values is to analyze a calibrated emission spectrum of an OLED.

## 2.3 Colorimetry

To characterize the color emitted from the OLED device, the standard tristimulus values X, Y, Z should be calculated. They are calculated by multiplying the spectral power distribution by  $\bar{x}, \bar{y}, \bar{z}$ , which are numerical descriptions related to the retina response of a standard observer to different wavelengths, and integrating over the wavelengths that the eye is sensitive to, which are from 380nm to 780 nm.

The obtained tristimulus values are related to the brightness, or luminance, of the emitted colour (Y), the eye's response to the blue colours (Z) and a linear combination of the retina cones' responses (X). The values of X, Y and Z can be obtained

in the following equation.

$$X = \int_{380}^{780} \phi(\lambda) . \bar{x}(\lambda) d\lambda \tag{1}$$

$$Y = \int_{380}^{780} \phi(\lambda) . \bar{y}(\lambda) d\lambda \tag{2}$$

$$Z = \int_{380}^{780} \phi(\lambda).\bar{z}(\lambda)d\lambda \tag{3}$$

(4)

From the tristimulus values, the derived values x, y, z in the CIE color space can be retrieved. These values can be plotted on the CIE color diagram. The values of x, y and z can be obtained from the tristimulus values in the following equation.

$$x = \frac{X}{X + Y + Z}$$
  $y = \frac{Y}{X + Y + Z}$   $z = 1 - x - y$  (5)

The CIE color diagram is shown in figure 4

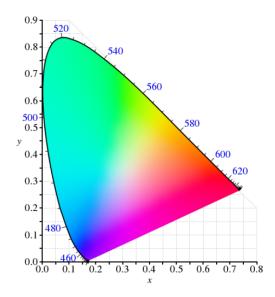


Figure 4: CIE 1931 color space chromaticity diagram.

## 2.4 Photometry

Photometry is the science of the measurement of light, in terms of its perceived brightness to the human eye. It is distinct from radiometry, which is the science of measurement of radiant energy (including light) in terms of absolute power. [4]

#### Photometry

Luminous Flux  $(\Phi_v)$  is energy per unit time (dQ/dt) that is radiated from a source over visible wavelengths. More specifically, it is energy radiated over wavelengths sensitive to the human eye, from about 330 nm to 780 nm. Thus, luminous flux is a weighted average of the Radiant Flux in the visible spectrum.

It is defined as

$$\Phi_v = K_m \int_{\lambda 1}^{\lambda 2} \phi(\lambda) . V(\lambda) d\lambda$$
(6)

where  $K_m = 683 lm/W, \lambda_1 = 380 nm$  and  $\lambda_1 = 780 nm$  which defines the visible area.  $V(\lambda)$  describes the spectral sensitivity of the human eye.

Luminance $(l_v)$  is the intensity of light emitted from a surface per unit area in a given direction. It can be related to the luminous flux by

$$L = \frac{d^2 \Phi_v}{d\omega dA \cos\epsilon} \tag{7}$$

where A is the area of the surface  $(m^2)$ , and  $d\omega$  is the solid angle (sr),  $\epsilon$  is the angle between the axis perpendicular to the emitting surface and the axis of the viewer.

Likewise, it is also possible to deduce the luminous flux from a known luminance distribution. For a Lambertian emitter (In reality, an ideal Lambert emitter does not exist.), the luminous flux is not dependent of the viewing angle and the total flux of an area A into the half space is then given by:

$$\Phi_v = \pi A L_\nu \tag{8}$$

The following comparison between radiometric and photometric units of light may be referred.

Radiometry	Unit	Photometry	Unit
Radiant flux $\Phi_e$	W	Luminous flux $\Phi_v$	lm
Radiance L <sub>e</sub>	$W/m^2sr$	Luminance $L_v$	$cd/m^2$
Radiant intensity $I_e$	W/sr	Luminous intensity $I_v$	cd
Irradiance E <sub>e</sub>	$W/m^2$	Illuminance $E_v$	$lx=lm/m^2$

## 3 Experiment

The steps conducted in this lab to fabricate OLED structures are explained in the following section.

1. Cut the Substrates:

Four ITO substrate of 25nm  $\times$  25nm were taken for the experiment. Glass cutter was used to cut the glass.

2. Structure the ITO electrode:

The substrates were covered by stickers and were immersed in 37 percent hydrochloric acid for 7 minutes. Afterwards the samples were rinsed with water and the sticker was removed (This process was already done by the tutor). To figure out where the ITO is on the glass surface multimeter is used.

3. To Clean the substrate:

1. Substrates were poured into acetone and were put into the ultrasonic bath for 5 minutes. Then it is dried with the nitrogen gun.

2. After that the same process is repeated with isopropanol as the solvent.

4. Prepare PEDOT:PSS Solution:

For each sample 150  $\mu$ l PEDOT:PSS solution is required. The solution is mixed of PEDOT:PSS and ethanol in 1:3 ratio(This process was already done by the tutor). The bottle containing the solution is then put into the ultrasonic bath for 5 minutes.

5. Substrate Pre-Treatment:

Before the substrate is coated with the [U+FB01] rst layer, potential organic residues has to be removed from the ITO surface. For this reason the substrates are exposed to oxygen plasma for 5 minutes.

6. Prepare Polymer Solution and Spin Coating:

As the emission layer, the polymer Super Yellow is used. The polymer was dissolved in toluene with a concentration of 3mg/ml. The pure material was weighed and dissolved with the respective amount of toluene. Whereas PEI was dissolved in isoproponal with a concentration of 3.86mg/ml. To completely dissolve the materials, a few days are required (This process was already done by the tutor). To cover the whole substrate  $180\mu$ l of Super Yellow and  $120\mu$ l of PEI solutions were used.

Now the pipette was adjusted to a volume of  $150\mu$ l and the PEDOT:PSS solution was spread on the substrate. After spincoating PEDOT:PSS, the substrates were dried at 100 degrees for 10 minutes.

For super yellow the pipette was adjusted to  $180\mu$ l the spin coating was done by a two step process of 1000rpm with an acceleration of 500rpm/s for 45s followed by 4000rpm, 800rpm/s for 3s. Then the samples were baked at 80 degree centigrade for 10 minutes.

7. Fabrication of Cathodes:

Aluminum is used to prepare the cathode. Each substrate could be divided into four parts(like quadrants) and from each part emitted light could be collected.

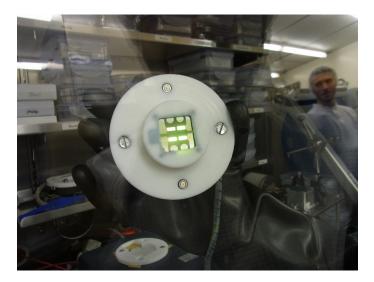


Figure 5: Emission of light from OLED

Accordingly the coating of the super yellow and ITO is removed from the substrates by scratching. Then the layers of Lithium Fluoride and Aluminum is put by vapour coating.

8. Data Collection: After the fabrication process voltage is applied across the prepared OLED. A spectrometer is connected with the set up with the help of a fiber optics cable. Voltage vs Current and Intensity vs Wave length values are measured and the characteristics are plotted.

## 4 ANALYSIS

In this section, the fabricated OLED is characterised based on the photometric units and color tristimulus values explained previously.

#### 4.1 Current Density Vs. Voltage Characteristics

Using the Optical Characterization System (OCS) for OLEDs, various electrodes are applied to the anodes and cathodes of the four devices on the substrate. The current density is measured with different voltage values, by sweeping the voltage from 0-8V. As expected, the current density vs. voltage follow the same trend as that of a conventional (inorganic) LED. At small voltages, as the voltage increases, the current density is approximately zero and the device off. When the voltage increases beyond a certain value, the current density increase becomes exponential and the device is ON. Beyond a certain voltage value, the current density saturates. This is shown in figure 6.

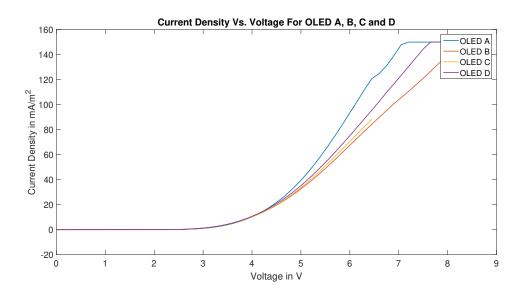


Figure 6: Current Density vs. Voltage characteristics of A, B, C and D OLEDs, from 0 to 8 Volts.

The current density is proportional to the number of carriers generated in the device. This means that is also proportional to the number of recombination processes that occur in the OLED device, and thus, proportional to the intensity of the emission, or the luminance of the device. According to figure 7, the peak luminance increases from  $0.25Cd/m^2$  at applied voltage V = 3V to a peak luminance of  $1.08Cd/m^2$  at

applied voltage V = 7V. Beyond a voltage of 6.5, the luminance remains at a constant value. The same trend can be expected for devices OLED B, C and D.

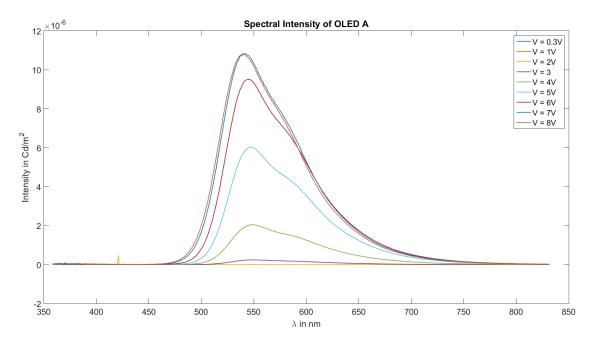


Figure 7: Intensity vs. Wavelength characteristics of OLED A, for different applied voltages.

As shown in figure 7, the peak wavelength of emission and the spectrum trend of the OLED remains the same with different applied voltages. The only difference between the emission spectra at different voltages is the luminance amplitude of the emission peak. This is because the wavelength at which the emission peak occurs depends on the bandgap of the emissive layer (Super Yellow), which is the energy difference of the LUMO and HOMO layers of the material. To change the wavelength of maximum emission, we would need to change the material of the emissive layer.

The conclusion to be drawn from this observation is that it is required to input sufficient voltage to the OLED device to get a good luminance value. For low voltages, the emission luminance will be too weak. If we assume the turn-on voltage is the voltage at which the peak emission is approximately 10  $Cd/m^2$ , then from figure 7, we can estimate the turn-on voltage to be approximately 6 Volts.

#### 4.2 Spectral Intensity Measurements

From figure 8, it can be observed that the spectral intensity of the OLED device has a peak value at wavelength  $\lambda_{peak} = 541nm$ , at which the peak luminance is

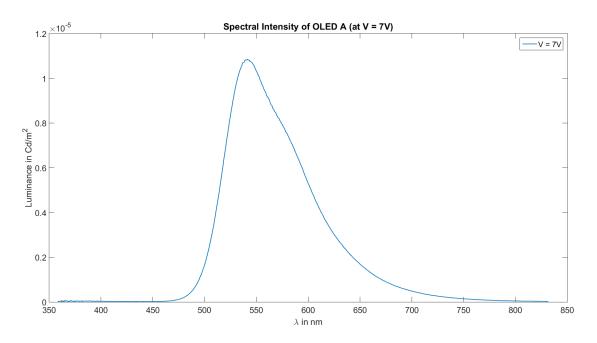


Figure 8: Intensity vs. Wavelength characteristics of OLED A, for applied voltage V = 7V.

 $I_{peak} = 1.08Cd/m^2$ . The bandwidth, or full width half maximum (FWHM), of the emission spectrum is FWHM = 83.3nm.

Similar spectral intensity characteristics are expected for the other devices on the substrate, which are OLED B, C and D. The spectral intensity characteristics of the four devices, at applied voltage V = 6.3 V, are given in figure 9.

Emissions of longer wavelengths also help in understanding radiative emissions from lower energy levels that stem from temperature/vibronic effects and from recombination at defects and impurities. For example, there seems to be another resonant mode at around 600nm where all spectra show a small bump.

## 4.3 Characterizing the Color of the OLED Emission

In order to characterize the color of the OLED emission, it is required to find the tristimulus values corresponding to the spectral intensity of the OLED. In order to find the values, the color matching functions are used, obtained from The Colour Vision Research laboratory database [2]. The color matching functions, which are  $\bar{x}, \bar{y}, \bar{z}$ , are given in figure 10.

These color matching functions are multiplied by the measured spectral intensity of the OLED and summed for all wavelengths from 380-780 nm, which is the sensitivity

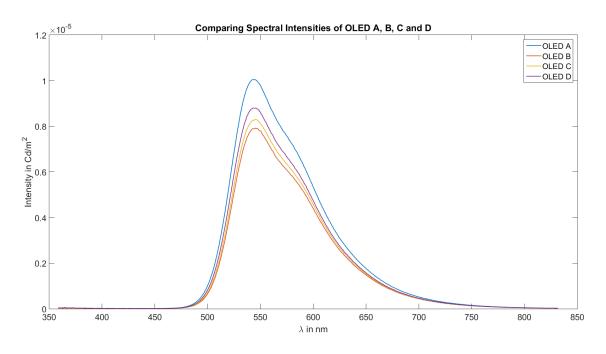


Figure 9: Intensity vs. Wavelength characteristics of OLED A, B, C and D for applied voltage V = 6.3V.

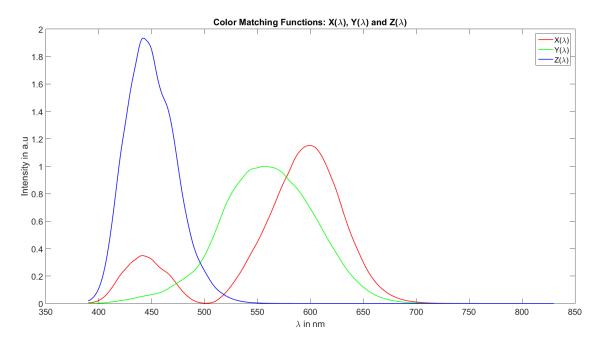


Figure 10: Color matching functions,

range of the human eye. The following equations are applied to obtain the tristimulus values X, Y and Z.

$$X = \int_{380}^{780} \phi(\lambda) . \bar{x}(\lambda) d\lambda \tag{9}$$

$$Y = \int_{380}^{780} \phi(\lambda) . \bar{y}(\lambda) d\lambda \tag{10}$$

$$Z = \int_{380}^{780} \phi(\lambda).\bar{z}(\lambda)d\lambda \tag{11}$$

(12)

By applying these equations for the measured spectral intensity of OLED A and the color matching functions, the following tristimulus values are obtained.

$$X = 5.55 x 10^{-4} \tag{13}$$

$$Y = 7.55 x 10^{-4}$$
(14)

$$Z = 2.55 x 10^{-4} \tag{15}$$

(16)

To find the point that these tristimulus values correspond to on the CIE color space, a normalization is needed to find the derived values of x and y.

$$x = \frac{X}{X + Y + Z} \qquad \qquad y = \frac{Y}{X + Y + Z} \tag{17}$$

The values of the derived values of x and y, are found to be as follows:

$$x = 0.4154 \qquad \qquad y = 0.5655 \tag{18}$$

By plotting the CIE color space, the exact point corresponding to the calculates x and y values can be marked. This is marked in figure 11, where the desired point labelled as 'OLED Emission'.

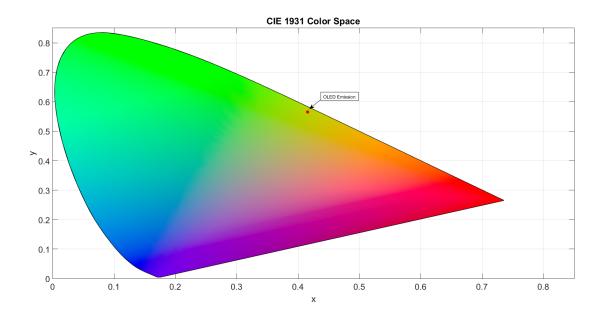


Figure 11: CIE 1931 Color Space, with the OLED emission point marked, according to the calculated x and y point, as 'OLED Emission'.

#### 4.4 Luminance and Efficiency Calculations

The luminous flux of the spectral emission of the OLED device can be obtained by multiplying the spectral intensity by the spectral sensitivity of the human eye and summing over the wavelengths from 380 to 780 nm, which the sensitivity range of the eye.

$$\Phi_v = K_m \int_{380}^{780} \phi(\lambda) . V(\lambda) d\lambda$$
(19)

In the previous equation,  $K_m = 683 lm/W$  and  $V(\lambda)$  describes the spectral sensitivity of the human eye. Our eyes are more sensitive to green light than to red or blue with the same physical radiant flux. The spectral sensitivity function, obtained from the Colour Vision Research laboratory database [2], is shown in figure 12.

By multiplying the measured spectral intensity by the spectral sensitivity function and summing over the wavelength range, the luminous flux is calculated (OLED A).

From the calculated luminous flux  $(\Phi_v)$  and knowing the active area of the device (A) to be 5mm x 5 mm, we can assume the emitter to be a Lambert emitter. Thus, we can apply the following equation to find the luminance  $(L_v)$ :

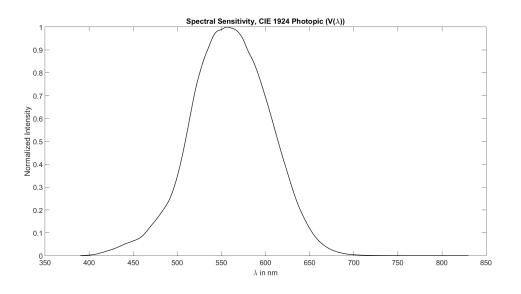


Figure 12: CIE 1924 Photopic spectral sensitivity function, used for luminous flux calculations.

$$L_v = \frac{\Phi_v}{pi.A} \tag{20}$$

Next, the power efficiency is calculated by dividing the luminous flux by the applied electrical power. The applied electrical power is the product of current and voltage. This is shown in the following equation.

$$P_{eff} = \frac{\Phi_v}{P_{tot}} \tag{21}$$

$$P_{tot} = I.V \tag{22}$$

At applied voltage V = 7V, the current density is  $J = 147.9mA/mm^2$  on an active area of 5mm x 5mm. This gives a power of 25.9W. Thus, the power efficiency is calculated.

Furthermore, to calculate the current efficiency, the luminance is divided by the current density of the OLED device. This is shown in the following equation.

$$I_{eff} = \frac{L_v}{J} \tag{23}$$

Finally, the results are plotted in the following table for OLED A, B, C and D.

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Measurement	OLED A	OLED B	OLED C	OLED D			
Luminous Flux (lm)	51.6	40.7	40.5	46.1			
Luminance $(cd/m^2)$	6570.5	5188.6	5156.1	5875.5			
Power Efficiency $(lm/W)$	199.4	157.4	156.4	178.2			
Current Efficiency (cd/A)	4.44	3.51	3.48	3.97			

Table 1: Luminance and Efficiency Calculations For OLEDS A,B,C and D

## 4.5 Luminance vs. Voltage Characteristics

In order to find the variation of luminance with voltage, the luminance is calculated at different voltages, in intervals of 0.5V. The plot of luminance vs. voltage is shown in the following figure 13.

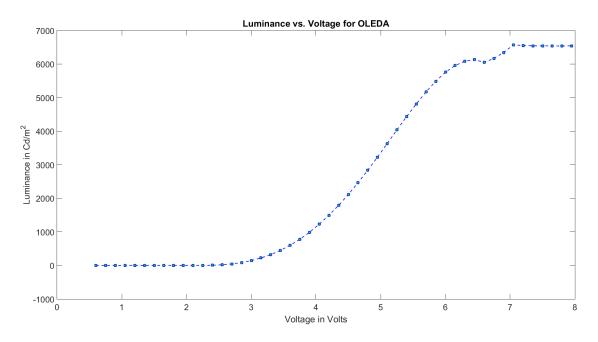


Figure 13: Calculated Luminance vs. Voltage characteristics, for OLED A.

Based on figure 13, the turn-on voltage, defined as the voltage at luminance of 10  $cd/m^2$ ,  $isV_{on} = 2.5V$ . The reason this calculated value differs from the theoretical value, is that this value takes into account the response of the eye and the sensitivity of the eye retina. This means that not all the emitted radiant flux is converted into luminous flux. Hence, the luminance value includes some losses due to the response of the eye retina.

## 4.6 Comparison with Literature

By comparing with the reported values in the literature, taken from [3], we find the following table.

Comparison of Luminance and Tower Linetency with					
Measurement		Measured	Literature		
_	Luminance $(cd/m^2)$	6570	2000		
	Current Efficiency (cd/A)	4.4	4.3		

Table 2: Comparison of Luminance and Power Efficiency with Literature

The efficiency of the OLED device may be increased, by applying the method in the reference, by doping the emissive layer with a dopant material to increase its emission efficiency.

## 5 Conclusion

From the previous results, we conclude that OLEDs formed of polymer layers are easily processed and can be fabricated by solution-processing, such as spin-coating processes. By fabricating an OLED device, certain parameters can be obtained to characterize the device, such as the current density vs. voltage characteristics, the tristimulus values which indicate the color observed from the OLED emission, and the spectral intensity of the OLED emission. Also, a conversion from the radiometric units to the photometric units can be made to obtain the luminance of the device. Thus, the power efficiency and current efficiency of the OLED can be calculated.

# References

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