

Fabrication of red organic light emitting diodes (OLEDs) using $\text{Eu}_x\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ organic complexes for solid state lighting

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ABSTRACT

The mechanism of energy transfer leading to electroluminescence (EL) of a lanthanide complex, $\text{Eu}_x\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ (TTA= thenoyltrifluoro-acetone, phen=1,10-phenanthroline), doped into TPBi(1,3,5-tris(N-Phenyl-benzimidazol-2-yl) benzene host at 15 wt% of host is investigated. With the device structure of anode/hole transport layer/ $\text{Eu}_x\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ (15%): TPBi/electron transport layer/cathode, maximum luminescence of 185.6 cd/m^2 and 44.72 cd/m^2 was obtained from device I made of $\text{Eu}_{0.4}\text{Y}_{0.6}(\text{TTA})_3\text{Phen}$ and device II made of $\text{Eu}_{0.5}\text{Y}_{0.5}(\text{TTA})_3\text{Phen}$, respectively at 18 volts. Saturated red Eu^{3+} emission based on $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition is centered at a wavelength of 612 nm with a full width at half maximum of 5 nm. From the analysis of I-V, J-V-L characteristics and electroluminescent (EL) spectra, we conclude that direct trapping of holes and electrons and subsequent formation of the excitation occur on the dopant, leading to high quantum efficiencies at low current densities. These results show that fabricated OLED devices can successfully emit saturated red light and can be used in applications such as opto-electronic OLED devices, displays and solid state lighting technology. Copyright © 2011 VBRI press.

Keywords: Electroluminescence; HOMO; LUMO; OLED device; luminance.



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Introduction

Now a day's challenging applications of organic materials for different fields, much more scientists have been prepared organic based materials by novel synthesis and reported functional physical and chemical characteristics of materials for possible applications. Recently Shukla et al. [1] prepared polyaniline thin film of 0.5 μm thickness by chemical oxidation method using CuSO_4 as initiating agent, the prepared polyaniline thin film was exposed to controlled humid condition and change in resistance has been recorded. Xinhua Yuan and Zhiwei Tian [2] using ultrasonic technology, phenylmethylsilicone/organic montmorillonite (OMMT) nanocomposites were prepared by *in situ* intercalative polymerization. The hardness of exfoliated and intercalated PLS nanocomposites are both firstly increased with the increasing of OMMT content, and then decreased, and finally increased again. Organic materials are effectively used in lamp industry as phosphors for emission of visible light by different excitation energy. Therefore, much work is possible for development of organic efficient phosphors by low cost synthesis for energy saving lamp phosphors, such as solid state lighting.

A great challenge in the field of organic light emitting diodes (OLEDs) is the realization of an efficient pure red light emitting diode with narrow emission line. Such an emitter is essential to complete the color spectrum for a full color display based on the principle of additive color mixing. One way to overcome the problem is to use an organic Eu^{3+} complex, in which Eu^{3+} ions acts as the emission centre [3-8]. The spectral properties of Eu^{3+} are ideal for use in full color displays as known from inorganic luminescent materials in cathode ray and projection television tubes. To design a red emitting OLED based on Eu^{3+} complex two criteria have to be fulfilled, namely (i) the Eu^{3+} complex has to exhibit high fluorescence quantum efficiency, and (ii) has to be integrated in the OLED structure such that high electro luminescence efficiency can be achieved. Several attempts were made by the researchers in the past to fulfill these two criteria. Many organic complexes are available from which red OLEDs can be fabricated [9-15].

Recently, Cocchi et al. [16] reported highly efficient, variable-color light-emitting diodes (LEDs) realized via mixing of molecular exciton and excimer phosphorescent emissions from a new organic phosphor have been used as either the low-concentration bluish-green (molecular) phosphorescence emitter or high-concentration red (excimer) phosphorescence emitter. Chih-Hao Chang et al. [17] recently reported successful fabrication of high-color-rendering pure-white phosphorescent organic light-emitting devices (OLEDs) by employing a true-blue iridium complex $\text{Ir}(\text{dfbppy})(\text{fbppz})_2$ and a wide-bandwidth yellow emitting osmium complex $\text{Os}(\text{bptz})_2(\text{dppee})$.

In this context, organic complexes of the trivalent europium ion (Eu^{3+}) are strong candidates for application in red light emission devices due to their emission at 612 nm, with essentially monochromatic emission, which minimizes the need of filtering. Therefore, it is proposed to fabricate OLED devices using binuclear $\text{Eu}_x\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ doped in TPBi by 15 wt% of host material.

Experimental

Chemicals

The purity and make of the chemicals used for the fabrication of OLED devices are as follows: Europium chloride (EuCl_3 , 99.5%), Yttrium chloride (YCl_3 , 99.5%), Terbium Chloride (TbCl_3 , 99.5%) were procured from rare earth chemicals, Kerala, India where as 2-thenoyl trifluoro acetone ($\text{C}_8\text{H}_5\text{F}_3\text{O}_2\text{S}$, 99.5%), National chemical, India; 1,10-phenanthroline ($\text{C}_{12}\text{H}_8\text{N}_2\text{H}_2\text{O}$, 99.5%), E-Merck, Germany; m-MTDATA (98%), Aldrich, USA; α -NPD (99%), Aldrich, USA; TPBi (99.99%), Aldrich, USA; LiF (99.99%); E-Merk, Germany and Alq_3 (99.99%), Aldrich, USA were used as received.

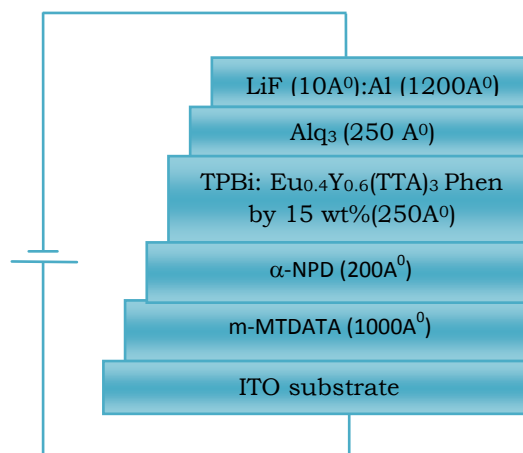


Fig. 1. Structure of fabricated OLED device-I.

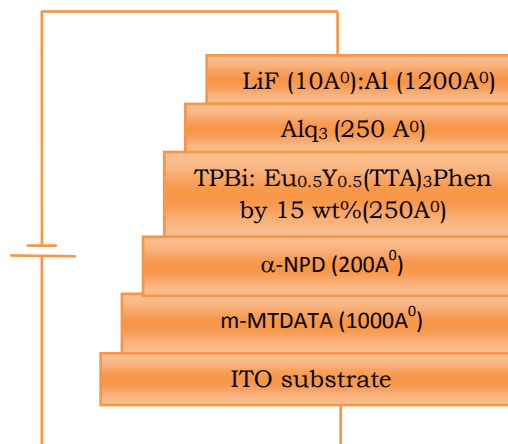


Fig. 2. Structure of fabricated OLED device-II.

Fabrication of red OLED devices

Organic EL devices with binuclear complexes as the emitter have an advantage of energy transfer process between the ligands and metal ion when the device is operated at low voltages [18]. In the present investigation, binuclear $\text{Eu}_x\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ (where $x = 0.4$ and 0.5) complexes have been chosen as emission layer material. These organic complexes are inserted between HTL and ETL as a separate emissive layer. $\text{Eu}_x\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ is sublimed in vacuum without decomposition due to the presence of phenanthroline, which saturates the metal complex coordination. As this complex has poor charge carrier transporting properties, TPBi is used as host along with the

dopant $\text{Eu}_{(x)}\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ by 15 wt% during organic layer deposition. Structures of the fabricated OLED devices used in the present study are shown in the **Fig. 1** and **2**.

The molecular structures of the materials used and the band structure of OLED devices are illustrated in **Fig. 3** and **4**, respectively.

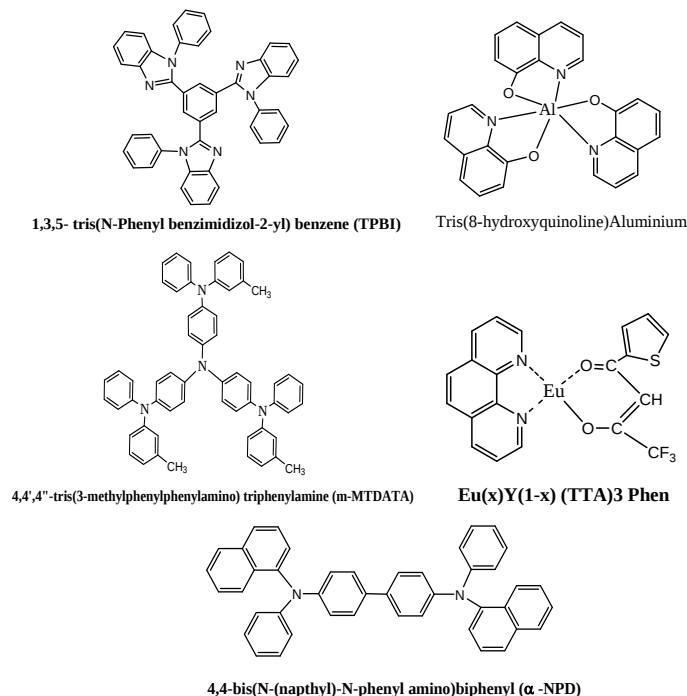


Fig. 3. Molecular structure of the materials used in fabricating OLED devices.

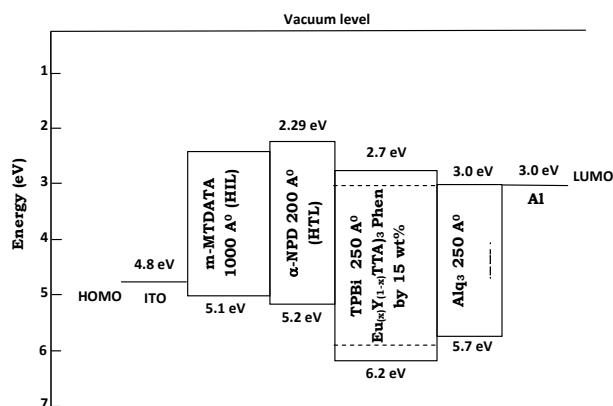


Fig. 4. The band structure of OLED devices.

OLED devices having the structure ITO/m-MTDATA/ α -NPD/TPBi: $\text{Eu}_{(x)}\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}/\text{Alq}_3/\text{LiF}/\text{Al}$ (where $x = 0.4, 0.5$) were fabricated. An hole injecting material m-MTDATA (4,4',4''tris(3-methylphenyl-phenyl amino triphenylamine) helps to balance hole and electron injection. The HIL needs enough thickness to planarize and wet the surface of the anode layer. However, since anode surfaces tend to be very rough, a thickness of up to 1000 Å may be desired for HIL in some cases. m-MTDATA has been identified as effective material in promoting injection of holes from ITO into hole transporting layers (HTL) with a hole mobility of about $3 \times 10^{-5} \text{ cm}^2/\text{Vs}$.

HTL is selected such that the HOMO and LUMO levels lie in midway between hole injection layer and electron transport layer. Since, α -NPD satisfies this requirement, it is selected as HTL layer in the present investigation. Eu complexes doped with appropriate host exhibits high efficiency, $\text{Eu}_{(x)}\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$ doped with TPBi(1,3,5-tris(N-Phenylbenzimidazol-2-yl) benzene) host at 15 wt% is used as emissive layer.

The host electron transporting material in the ETL is selected such that electrons can be efficiently injected from the cathode into the LUMO level of the electron transporting material. Alq_3 exhibits highly stable film forming characteristics, good carrier transport properties and good heat resistance and hence chosen as a model ETL. Electron and hole transporting layers play a vital role on the EL device performance. Electron injection from Al cathode to the electron transport layer is enhanced by a thin LiF buffer layer (10 Å) and hole injection from ITO anode to HTL, thereby significantly enhancing the injection of electrons and holes in the emissive layer.

OLED devices are fabricated on Doosan DND OLED fabrication system by vacuum deposition technique [19-22]. Patterned indium tin oxide (ITO) coated glass substrate with a sheet resistance less than 10 ohm/square is taken as base for the deposition of organic layers. This ITO has a high work function ($\phi_w \approx 4.7\text{--}4.9 \text{ eV}$). ITO glass substrate was carefully cleaned under low-pressure oxygen plasma with the flow of oxygen at a rate of 50 SCCM at a pressure of 80 milli Torr before deposition organic layers. Subsequently, the oxygen plasma treated ITO substrate was transferred to organic chamber. Organic layers were sequentially deposited at a pressure of 6×10^{-8} Torr at the deposition rate of 1 Å/sec in a vacuum system consisting of two connected vacuum chambers namely organic chamber and metal chamber. The thickness of HIL, HTL, EML: dopant (15 wt%), ETL are 1000, 200, 250 and 250 Å, respectively. The substrate was transferred another chamber for deposition of the metal cathode without breaking the vacuum. LiF buffer layer and aluminum cathode (LiF (10 Å: Al:1500 Å) were deposited at a base pressure of 6×10^{-8} Torr, at the deposition rate of 0.01 Å/sec and 2 Å/sec, respectively. As deposited organic layers are sensitive to oxygen and moisture, the fabricated devices were encapsulated using a glass lid and UV sealant resin and finally UV cured in a nitrogen filled glove box (dew point $\approx 75^\circ\text{C}$) for 3 min after fabrication.

Results and discussion

The devices with the configuration of ITO/m-MTDATA(1000Å)/ α -NPD(200 Å)/TPBi: $\text{Eu}_{0.4}\text{Y}_{0.6}(\text{TTA})_3\text{Phen}$ (15 wt%, 250Å)/ Alq_3 (250Å)/LiF:Al (10:1200 Å) and ITO/m-MTDATA(1000Å)/ α -NPD (200Å)/ TPBi: $\text{Eu}_{0.5}\text{Y}_{0.5}(\text{TTA})_3\text{Phen}$ (250Å)/ Alq_3 (250Å)/ LiF:Al (10:1200 Å) were fabricated. We have selected a doping concentration of TPBi as 15% because, it was observed that when the doping concentration is taken as 2%, 5%, 10%, the intensity of EL spectra was very low, hence, the next doping concentration as 15%. This may be due to the poor charge transport characteristics of the emissive material $\text{Eu}_x\text{Y}_{(1-x)}(\text{TTA})_3\text{Phen}$. TPBi plays a vital role as charge transporting

layer i.e., the energy absorbed by TPBi molecules that is completely transported to Eu^{3+} thereby enhancing its transportation properties.

The performance of the fabricated OLED devices were tested by carrying various electrical characteristics such as current density-voltage-luminance (J-V-L), voltage-luminance-electroluminescence efficiency (V-L- η) characteristics, and electro luminescence (EL) spectra using Keithley 2400 programmable voltage current source and Minolta spectro radiometer CS-1000 connected to Keithley 2400 programmable voltage current source.

Characterization of OLED devices

J-V-L characteristics: Current density-Voltage-Luminance (J-V-L) characteristics of device I and device II are shown in **Fig. 5** and **6**, respectively. The J-V-L curve characterizes the properties among the current density (J), bias voltage (V) and the luminance output (L). The pixel having device area of 16 mm^2 , is focused to spectroradiometer in order to record current and luminescence at particular voltage. Current density as well as luminescence increased with the drive voltage, and reaching a maximum luminance value of 185.6 cd/m^2 and 44.72 cd/m^2 for device I and II, respectively at 18 volts.

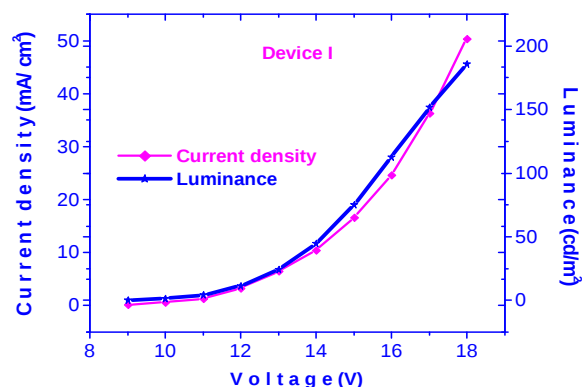


Fig. 5. J-V-L characteristics of device I.

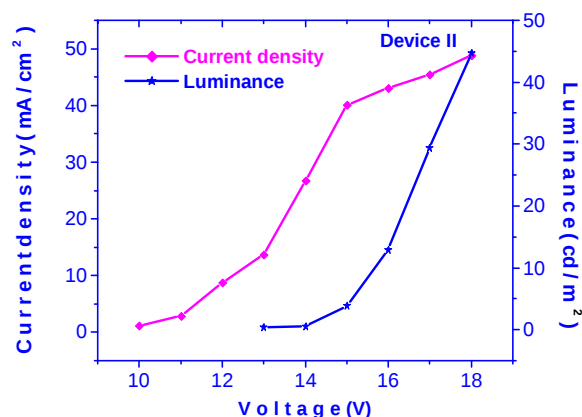


Fig. 6. J-V-L characteristics of device II.

It was observed that the current density and luminance increased with voltage in both the devices. This may be due to the fact that transportation of more charge carriers is possible at higher voltages.

V-L- η characteristics: V-L- η curve characterizes the variation of luminance output (L) and electro luminescence efficiency (η) with voltage (V). V-L- η characteristics of the device I and II for 16 mm^2 device area is shown in **Fig. 7**. It is observed that the luminance increases exponentially with voltage for both the devices. Maximum luminance of 185.6 cd/m^2 and 44.72 cd/m^2 was obtained at 18 V for device I and device II, respectively. This reveals that the Luminance in device I is about 4 times as strong as that in device II and the EL efficiency in device I is better than the device II as shown in **Fig. 7**. Ideally, the turn-on voltage should be as low as possible, but in many lanthanide-based OLEDs the value is between 5 and 10 V [23].

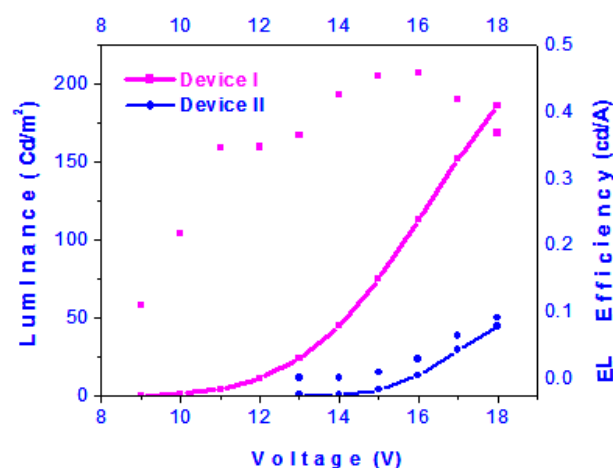


Fig. 7. V-L- η characteristics of device I and device II.

Electroluminescence (EL) spectra

EL spectra of device I and II for 16 mm^2 device area is shown in **Fig. 8** and **9**, respectively at different bias voltage. With host: dopant combination, maximum luminance of 187.6 cd/m^2 and 47.72 cd/m^2 is achieved at 612 nm for device I and II, respectively at a bias voltage of 18 volts.

The spectra contain five primary peaks at 525, 590, 612, 650, 704 nm, corresponding to $^5\text{D}_0 \rightarrow ^7\text{F}_j$ transitions ($j = 0, 1, 2, 3, 4$) while a broad band at 525 nm in the green region is due to emission from Alq_3 , which is used as ETL in the present device structure. Molecular origin of these peaks is from Eu^{3+} moieties a sharp emission peak at 612 nm is due to $^5\text{D}_0$ state and long life of electric dipole induced $^5\text{D}_0 \rightarrow ^7\text{F}_2$ transition of Eu^{3+} ion. Due to this long lifetime this state can be easily filled very quickly and the emission accordingly saturates [24]. In figure 9, this band is more intensified, where as in device II, this green emission is suppressed because the EL intensity of device II is low even at a voltage of 18V.

The host TPBi is a well known blue emitting material at 460 nm [25]. No emission in blue region indicates that the energy absorbed by the TPBi molecules is completely

absorbed by the Eu^{3+} complex molecules. The emission properties of the device are controlled by the Eu^{3+} complex molecules. The emission in these devices is due to recombination of trapped electron and holes on the binuclear Eu^{3+} complex, resulting in high turn-on voltage. Earlier, it was reported that the high turn-on voltage in phosphorescent devices is due to recombination of trapped electron and trapped holes on the LUMO and HOMO levels, respectively, of phosphorescent molecules [26, 27]. The PL intensity of the emissive materials in device I and II was found to be 604 and 500 a.u., respectively. In both the devices, the shape of the PL spectra remained at the same position at 590, 612, 650 and 704 nm with maximum peak intensity at 612 nm, which is due to Eu^{3+} moieties.

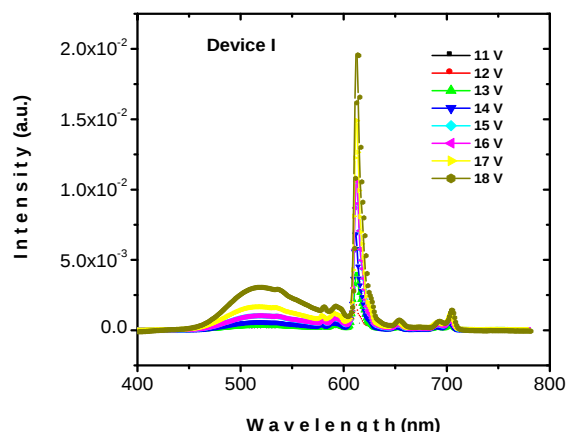


Fig. 8. Electroluminescence (EL) spectra of device I.

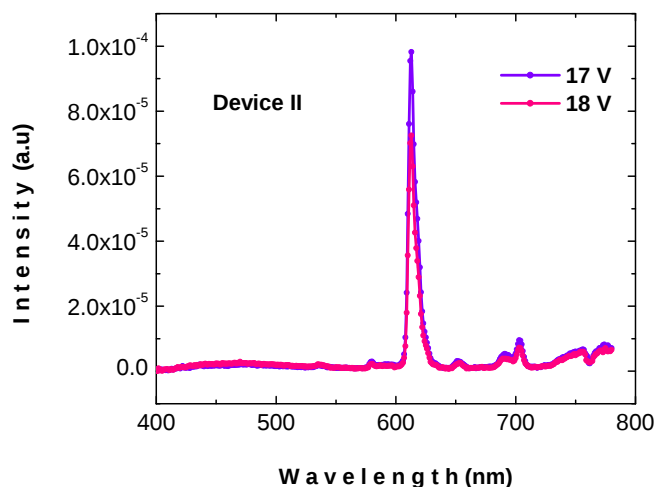


Fig. 9. Electroluminescence (EL) spectra of device II.

Conclusion

Multilayer OLED devices have been fabricated with binuclear $\text{Eu}_{0.5}\text{Y}_{0.5}(\text{TTA})_3\text{Phen}$ and $\text{Eu}_{0.4}\text{Y}_{0.6}(\text{TTA})_3\text{Phen}$ complexes as organic emissive layer. Different characterization techniques such as I-V, J-V-L, V-L- η characteristics and electroluminescence (EL) spectra were carried out for the fabricated devices at room temperature in ambient atmosphere. Bright and efficient EL device with a

narrow luminescent emission and narrow bandwidth in the red region at 612 nm has been obtained. Full width at half maxima (FWHM) was found to be less than 5 nm for both the devices. The turn on voltage of device I and II are 13V and 17V, respectively. Electroluminescence of these devices increased with voltage, maximum brightness of 185.6 cd/m^2 and 44.72 cd/m^2 was observed for device I and II, respectively. Although the driving voltage is rather high compared to the other OLED devices, the device performance can be improved by employing highly purified organic complexes, implementing the method of co-doping (organic emitting material can be co-deposited with an appropriate host), controlling the thickness of each layer, proper selection of HIL, HTL, ETL as well as the device structure. Emission color is basically determined by the energy difference of HOMO and LUMO of the emitting organic material. Consequently by changing these active materials the emission color can be varied across the entire visible spectrum.

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