JOURNAL OF

# A field-dependent organic LED consisting of two new high $T_g$ blue light emitting organic layers: a possibility of attainment of a white light source<sup>†</sup>

# Soon Wook Cha and Jung-II Jin\*

Department of Chemistry and the Center for Electro- and Photo-Responsive Molecules, Korea University, Seoul 136-701, Korea

Received 10th October 2002, Accepted 10th January 2003 First published as an Advance Article on the web 4th February 2003

Two new blue light emitting trimeric compounds of the Y-shape type having high glass transition temperatures were synthesized and EL behavior of LED devices consisting of bilayers of the two compounds was studied. One of the compounds is of hole-transporting type containing carbazole moieties, whereas the other is of electron-transporting type bearing phenyloxadiazole moieties. The bilayer LED devices exhibit a strong field-dependence and emit white light (simultaneous light-emittance in blue, green and red regions), at high applied electric fields. Increased interfacial formation of exciplexes at stronger external fields appears to be responsible for this field-dependence.

# Introduction

There have been extensive studies on organic light-emitting diodes (LEDs)<sup>1-11</sup> using low molecular mass organic materials, dendrimers<sup>12,13</sup> and polymers. The possibilities of making large area LEDs and of tuning the colors, covering the whole range of the visible spectrum, are especially attractive properties since this would allow the construction of full color displays. In addition, there is a strong demand for realization of efficient white light sources that can be utilized for a variety of uses such as in the application of large panel LEDs for back lighting in liquid crystal displays. Several ways to produce white light from polymeric and low molecular mass organic devices have been presented.<sup>14-21</sup> Polymers doped with an appropriate combination of red, green and blue fluorescent dyes can produce white light.<sup>14</sup> A cell structure comprised of polymeric layers each doped with blue, green and red fluorescent dyes can produce white light emission.<sup>15</sup> A luminescent conjugated polymer overlaid with a luminescent low molar mass electron transport material produced bluish-white light emission through a combined electroluminescence from both emitting layers as well as charge transfer complexes formed at the polymer/low molar mass molecule interface.16

The thermal stability of organic materials and their glassy state is an important factor governing device stability of organic light emitting diodes (OLEDs). There are a number of sources of thermal stress on OLEDs:<sup>22-24</sup> Heat evolved from deposition of the metal cathode during fabrication, Joule heating generated during device operation, and sudden exothermic crystallization of organic compounds employed in the devices are representative examples. If an OLED is heated above the glass transition temperature  $(T_g)$  of the organic materials in the device, irreversible failure can occur. In order to improve device performance, one often has to employ electron transporting and/or hole transporting materials. Poly(*N*-vinylcarbazole) (PVK)<sup>25</sup> and carbazole derivatives<sup>26</sup> are known to be excellent hole transporting materials, whereas 1,3,4-oxadizole containing compounds<sup>27</sup> have been extensively investigated as electron transporting materials. In addition,

highly efficient phosphorescent organic light-emitting devices are attracting a great deal of interest.<sup>28</sup>

In this paper, we describe characteristics of an LED device consisting of two layers of new compounds, **Cz3d** and **Oxa3d**, whose structures are shown in Schemes 1 and 2.

## **Results and discussion**

Schemes 1 and 2 illustrate the synthetic procedures used for the preparation of the two compounds. They could be readily prepared via multistep synthetic routes utilizing known reactions for each step. The structures of the intermediates and the final compounds, Cz3d and Oxa3d, were confirmed by <sup>1</sup>H NMR and IR spectroscopy and by elemental analysis as described in the Experimental section and the Supporting Information.<sup>†</sup> Both compounds are of bent-shape type. Cz3d is composed of three carbazole moieties whereas Oxa3d comprises three 1,3,4-oxadizole moieties. The three moieties in each are connected through stilbene bridges in both compounds. According to the thermal properties of these compounds investigated by DSC analysis, the glass transition temperatures  $(T_g)$  of **Cz3d** and **Oxa3d** are 250 and 156 °C, respectively (Table 1). These values are very high when compared with commonly used hole transporting materials such as 1,4-bis-(1-naphthylphenylamino)biphenyl ( $\alpha$ -NPD,  $T_g = 100$  °C) and 1,4-bis(phenyl-*m*-tolyamino)biphenyl (TPD,  $T_g = 60$  °C). Okumoto and Shirota<sup>29</sup> recently reported high  $T_g$  organic compounds which were utilized as carrier transporting materials in EL devices.

Fig. 1 compares the UV-Vis absorption and photoluminescence (PL) spectra of **Cz3d** and **Oxa3d** in dilute solutions and in thin films. The solid thin films were prepared by vacuum deposition. The PL spectra were taken at an excitation wavelength of 300 nm. Fig. 1(a) shows that a dilute solution of **Cz3d** ( $10^{-5}$  mol L<sup>-1</sup>) absorbs at 300 and 349 nm with the tail extending to about 425 nm. The two absorptions are ascribed to the carbazole and phenylcarbazole moieties, respectively. The extended tail part originates from the  $\pi$ - $\pi$ \* transition of the whole conjugated  $\pi$ -electron system. The two absorptions show a slight red shift in the solid film and appear at 304 and 355 nm, respectively (Fig. 1(b)). In addition, the absorption

<sup>†</sup>Electronic supplementary information (ESI) available: experimental section. See http://www.rsc.org/suppdata/jm/b2/b209949g/



Scheme 1 Reagents and conditions: (i) 4-bromotoluene, Cu,  $K_2CO_3$ , dibenzo-18-crown-6, o-dichlorobenzene, reflux; (ii) Br<sub>2</sub>, CCl<sub>4</sub>, room temperature; (iii) *N*-bromosuccinimide (NBS), CCl<sub>4</sub>, reflux; (iv) triphenylphosphine, benzene, reflux; (v) 1 M NaOH; formaldehyde (37% water solution), room temperature; (vi) (PPh<sub>3</sub>)<sub>4</sub>Pd (cat.), toluene/Na<sub>2</sub>CO<sub>3</sub>(2 M in H<sub>2</sub>O), 4-*tert*-butylphenylboronic acid; (vii) palladium(n) acetate, tri-o-tolylphosphine, DMF, triethylamine, 90 °C.

beyond 550 nm with a shoulder clearly discernible at 395 nm. The optical band gap energy estimated from the absorption edge (420 nm) of the solid thin film is 2.96 eV. The two sharp peaks at 414 and 434 nm (Fig 1(a)) in the PL spectrum of the **Cz3d** solution also displayed a red-shift and moved to 426 and 444 nm with a longer tail (Fig. 1(b)) in the solid film. Additionally, the emission intensity ratio between the two peaks became reversed in the solid film, indicating the lower energy deactivation or longer wavelength emission is more preferred in the solid state.

We observe similar phenomena in UV-Vis absorption and PL spectra of **Oxa3d** as shown in Fig. 1(c) and (d). **Oxa3d** as a dilute solution  $(10^{-5} \text{ mol L}^{-1})$  absorbs at 279, 334 and 337 nm with a shoulder peak appearing at 368 nm (Fig. 1(c)). The first three absorptions arise from the substituted oxadiazole structures and the shoulder from the  $\pi$ - $\pi$ \* transition of the extended  $\pi$ -electron system. The optical band gap energy for **Oxa3d** is estimated to be 3.13 eV. The solution PL spectrum shown in Fig. 1(c) exhibits an emission intensity maximum at

much less resolved absorption spectrum (Fig 1(b)), which shows a strong absorption at 339 nm with a shoulder at 378 nm. The solid film shows a strong PL emission at 438 nm with weak shoulders overlapped around at 495 and 518 nm. Although it is not definitive, it appears that there is a shoulder at about 420 nm. It is clear that both Cz3d and Oxa3d are bluelight emitters. We observe the occurrence of a weak long tail in the PL spectrum of Oxa3d, implying possible additional emission from excimers and/or exciplexes. The PL quantum yields in chloroform solutions were 0.28 for Cz3d and 0.93 for Oxa3d, respectively (Table 1). The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) levels determined by cyclovoltametry and from optical bandgaps are at 5.64 and 2.68 eV for Cz3d and 5.91 and 2.78 eV for Oxa3d, respectively. According to these values, Oxa3d is expected to have a higher affinity for electrons and also to act as a hole-blocker. For the sake of comparison, data for the EL spectra of Cz3d and Oxa3d obtained from ITO/Cz3d

403 nm with a shoulder at 430 nm. The solid film reveals a



**Scheme 2** Reagents and conditions: (i)  $NH_2NH_2\cdot H_2O$ , ethanol, reflux; (ii) 4-*tert*-butylbenzoylchloride, THF, pyridine, 0 °C; (iii) POCl<sub>3</sub>, reflux; (iv) *N*-bromosuccinimide (NBS), CCl<sub>4</sub>, reflux; (v) triphenylphosphine, benzene, reflux; (vi) 1 M NaOH; formaldehyde (37% water solution), room temperature; (vii) 3,5-dibromobenzoylchloride, THF, pyridine, 0 °C; (viii) palladium(II) acetate, tri-*o*-tolylphosphine, DMF, triethylamine, 90 °C.

Table 1 Physical data for Cz3d and Oxa3d

Compound	$T_{\rm g}/^{\circ}{\rm C}$	$T_{\rm m}/^{\circ}{\rm C}$	$\lambda_{\max}^{a}$ /	nm $\lambda_{em}^{a}/r$	$m \Phi_{\rm f}^{\ b}$
Cz3d Oxa3d	250 156	NA <sup>c</sup> 218	334 339	444 438	0.28 0.93
<sup><i>a</i></sup> Measured in ${}^{c}NA:T$ not	film. <sup>b</sup> Flu	iorescence	quantum	efficiency in a	chloroform.

or **Oxa3d** (50 nm)/Li: Al devices are included in Fig. 1. Although there exist some differences in details the general profile of their PL and EL spectra are very similar.

A double layer EL device was fabricated using **Cz3d** as a hole transporting, emitting layer and **Oxa3d** as an electron transporting, emitting layer, with the configuration ITO/**Cz3d** (40 nm)/**Oxa3d** (20 nm)/Li–Al. The two organic layers were consecutively vacuum deposited onto indium–tin oxide (ITO) coated glass (anode) followed by vacuum deposition of Al–Li alloy (cathode) containing 0.26% of Li.

Fig. 2(a) shows how the EL spectrum of the device depends on the applied electric field and clearly demonstrates that the EL spectrum of this device changes drastically when the applied field was higher than 2.5 MV cm<sup>-1</sup>. At electric fields of 1.5 and 1.83 MV cm<sup>-1</sup> the device emits at 400–550 nm with maximum emission intensity being observed at 451 nm overlapped with a shoulder at about 424 nm. As the applied field is increased to 2.5 MV cm<sup>-1</sup>, the emission intensity of the shoulder part (424 nm) greatly increases in intensity and becomes the major emission peak. This emission originates from the excitons formed in the **Cz3d** and **Oxa3d** layers (see EL spectra shown in Fig. 1(b) and (d)) and is thus an overlapped emission from the two layers. When the external field is further increased,



**Fig. 1** Absorption, PL and EL spectra of (a) **Cz3d** in solution (CHCl<sub>3</sub>), (b) **Cz3d** film (50 nm), (c) **Oxa3d** in solution (CHCl<sub>3</sub>), and (d) **Oxa3d** film (50 nm).



Fig. 2 Dependence of EL spectrum on applied electrical field (device: (a) ITO/Cz3d (40 nm)/Oxa3d (20 nm)/Li–Al and (b) ITO/Cz3d (20 nm)/Oxa3d (40 nm)/Li–Al).

surprisingly, two new peaks appear; one in the green light region (526 nm) and the other in the red light region (592 nm) region, in addition to the original blue light emission. Above an applied electric field of 2.8 MV cm<sup>-1</sup> the emitted light appears visually to be white. It is believed that the red light emission originates from the exciplexes (Oxa3d\*...Cz3d) formed at the interface between the excited species of Oxa3d and Cz3d in the ground state. The peak in the green region appears to result from the exciplexes (Cz3d\*...Oxa3d) formed between the excited species of Oxa3d and Cz3d in the ground state. This conclusion can be easily derived by comparing the EL spectra (Fig. 2(a)) of this device with those of ITO/Cz3d (20 nm)/ Oxa3d (40 nm)/Li-Al presented in Fig. 2(b), considering the relative thickness of the two emitting layers. The former device favors the accumulation of more Oxa3d polarons on the interface between the two layers, whereas in the latter accumulation of more Cz3d polarons on the interface is favored. Such a conjecture is based on the assumption that the hole mobility in the Cz3d layer and the electron mobility in the Oxa3d layer are comparable. Increasing the applied external electrical field is expected to increase the number of carriers injected, which, in turn, enhances the probability for the formation of exciplexes at the interface. This is exactly what we observe in the present devices.

Therefore, it is as expected to observe that the total intensity of emitted light also depends on the applied electric field. Fig. 3 shows that the intensity of emitted light increases to  $410 \text{ cd m}^{-2}$ 



Fig. 3 Luminance versus applied electric field of the device ITO/Cz3d (40 nm)/Oxa3d (20 nm)/Li–Al and EL spectra at two different applied electric fields.



**Fig. 4** Comparison of UV-Vis absorption ( $\bullet$ ,  $\bigstar$ ,  $\times$ , +) and PL spectra ( $\bigcirc$ ,  $\triangle$ ) of the double layer and blend films.

as the applied field is increased to 2.5 MV cm<sup>-1</sup>. However, it then starts to decrease as the field is further increased. This point occurs with the appearance of the red emission (see Fig. 2). The external quantum efficiency was about 0.2% at an applied field of 2.0 MV cm<sup>-1</sup> and it decreases steadily when the field is higher than 2.8 MV cm<sup>-1</sup>.

Recently, Jenekhe and coworkers<sup>30</sup> reported photoluminescence properties of double layers of poly(p-phenylene benzobisoxazole) (PBO) and tris(p-toyl)amine. They observed an emission at 474 nm which was ascribed to the exciplexes formed between the electron-donating amine compound and electronaccepting PBO. Epstein and coworkers<sup>31</sup> also reported PL and EL emissions from exciplexes from blends of PVK and an electron-accepting conjugated polymer. Separately, we constructed an LED device by codepositing a 60 nm thick film of Cz3d and Oxa3d in 1:1 weight ratio onto an ITO glass and tried to study the device characteristics. Unfortunately, the device revealed very poor device performance due to very high turn-on electric field and we failed in obtaining an acceptable EL spectrum. Thus, we obtained only a PL spectrum (Fig. 4) of the blend film and we compared it with the PL spectrum of the double layered film. We note that the two PL spectra are very different from each other. While the PL spectrum of the double layered film is practically the same as its EL spectrum obtained at low applied fields as shown in Fig. 1, the PL spectrum of the blend exhibited a significant degree of red shifting and showed an emission maximum at about 475 nm; the spectrum was also much broader. A close examination of the PL spectrum of the blend showed shoulders around 524 and 600 nm. The positions of the shoulders coincide with the positions of the new EL emission peaks for the double layered film at high applied electric fields as shown in Fig. 2. This implies that the blend, due to much greater contact area between Cz3d and Oxa3d molecules, exhibit stronger emissions from exciplexes than the bilayered film.

### Conclusion

We have synthesized thermally stable, high  $T_g$ , and bentshaped conjugated molecules, **Cz3d** and **Oxa3d**, both of which are blue light emitting materials. A double layer EL device consisting of **Cz3d** and **Oxa3d** layers exhibited a strong dependence of its EL spectra on the applied electric field and emitted white light at high field (>2.8 MV cm<sup>-1</sup>). This is ascribed to the increased formation of exciplexes at the interface of the two layers. High applied electric fields are expected to increase the number of the two opposite carriers injected into the layers, which would confer a greater chance to form exciplexes at the interface between the two layers. Our observation leads to a novel method to design a white light source by employing two layer LEDs consisting of an electrondonating layer and an electron-attracting layer both of which are blue-emitting, but are able to form green and red-light emitting exciplexes. This work also provides us with an excellent example that reveals a color tuning by changing the applied field. The light intensity and the device efficiency of the present devices, however, require much further improvement to be acceptable for practical application.

# Experimental

### Synthesis

The synthetic route to the two compounds are shown in Scheme 1 and 2.

Details are available in the Supporting Information. Only structural characterization data for Cz3d and Oxa3d are given below.

3,6-Bis(2-{4-[3,6-bis(4-tert-butylphenyl)cabazole-9-yl]phenyl}vinyl)-9-p-tolyl-9H-cabazole (Cz3d). <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>, ppm):  $\delta$  1.40 (s, 36H, C(CH<sub>3</sub>)<sub>3</sub>,), 2.53 (s, 3H, CH<sub>3</sub>), 7.34-7.80 (m, 54H, Ar-H), 8.40 (s, 6H, Ar-H). FTIR (KBr, cm<sup>-1</sup>): 3064 (Ar C-H stretching), 2965 (aliphatic C-H stretching), 1596, 1508 (Ar C=H stretching), 1276, 1220 (C-N stretching). Elemental analysis: Calc. for C<sub>99</sub>H<sub>89</sub>N<sub>3</sub>: C, 90.03; H, 6.79; N, 3.18%. Found: C, 90.12; H, 6.88; N, 3.20%. MALDI-TOF MS: Calc. for C<sub>99</sub>H<sub>90</sub>N<sub>3</sub> (MH<sup>+</sup>) m/z 1320.7, found: 1321.0.

2-[3,5-Bis(2-{4-[5-(4-tert-butylphenyl)-1,3,4-oxadiazole-2yl]phenyl}vinyl)phenyl]-5-(4-methylphenyl)-1,3,4-oxadiazole (Oxa3d). Yield = 62% (1.20 g); mp 218 °C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, ppm): δ 1.39 (s, 18H, C(CH<sub>3</sub>)<sub>3</sub>,), 2.44 (s, 3H, CH<sub>3</sub>), 7.30 (s, 4H, CH=CH), 7.36 (d, 2H, Ar-H), 7.57 (d, 4H, Ar-H), 7.72 (d, 4H, Ar-H), 7.81 (s, 1H, Ar-H), 8.05-8.19 (m, 12H, Ar-H). FTIR (KBr, cm<sup>-1</sup>): 3064 (Ar C-H stretching), 2964 (aliphatic C-H stretching), 1613, 1494 (Ar C=H stretching), 1575 (C=N stretching), 1012 (C-O-C stretching). Elemental analysis: Calc. for C<sub>55</sub>H<sub>48</sub>N<sub>6</sub>O<sub>3</sub>: C, 78.55; H, 5.75; N 9.99%. Found: C, 78.45; H, 5.80; N 9.89%. MALDI-TOF MS: Calc. for  $C_{55}H_{49}N_6O_3$  (MH<sup>+</sup>) *m*/*z* 841.4, found: 841.6.

## Identification and measurement

<sup>1</sup>H NMR (300 MHz) and IR spectra were recorded on a Varian AM 300 spectrometer and on a Bomem MB FT-IR instrument, respectively. Elemental analyses were performed by the Center for Organic Reactions, Sogang University, Seoul, Korea, using an Eager 200 elemental analyzer. Thermal properties were studied under a nitrogen atmosphere on a Mettler DSC 821<sup>e</sup> instrument. The molecular mass of each compound was measured using a matrix-assisted laser desorption/ionization time of flight (MALDI-TOF) PerSeptive Biosystems Voyager-DE<sup>m</sup>RP mass spectrometer. The UV-vis absorption and luminescence spectra were recorded on an HP8452A Diode Array spectrophotometer and an AMINCO-Bowman Series 2 luminescence spectrometer, respectively, at room temperature. Fabrication methods and conditions can be found elsewhere.<sup>1,16,20,21</sup> The current and luminescence intensity as a function of applied field were measured using an assembly consisting of a PC-based DC power supply (HP 6623A) and a digital multimeter (HP 34401). A light power meter (Newport Instruments, Model 818-UV) was used to measure the device light output in µW. Luminance was measured by a MINOLTA LS-100 luminance meter.

## Acknowledgements

This work was supported by the Korea Science and Engineering Foundation through CMR of Korea University. S. W. Cha is a recipient of the Brain Korea 21 scholarship from the Ministry of Education and Human Resources, Korea.

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