A new family of bis-DCM based dopants for red OLEDs†

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Received 4th January 2005, Accepted 30th March 2005
First published as an Advance Article on the web 12th May 2005
DOI: 10.1039/b419408j

We have synthesized a series of novel bis-DCM derivatives as candidate red dopants for use in organic light-emitting devices (OLEDs), by introducing various donor-substituted aryl rings. Compared to DCJTB (621 nm), the novel dopants (637–677 nm) showed more red-shifted emission in 1,2-dichloroethane. Using bis-DCMNEtOBu (**7**) as a dopant, we fabricated OLEDs with the configuration of ITO/4,4′,4″-tris(3-methylphenylamino)triphenylamine (*m*-MTDATA) (20 nm)/*N*,*N*′-bis(1-naphthyl)-diphenyl-1,1′-biphenyl-4,4′-diamine (NPB) (40 nm)/tris(8-quinolinolato)aluminium (Alq_3) : red dopant (35 nm, x wt%)/Alq_3 (35 nm) LiF/Al. The device with a doping concentration of 1.25 wt% showed pure red emission at $\lambda_{max} = 654$ nm (chromaticity coordinate: $x = 0.67$, $y = 0.33$) and a maximum brightness of 2500 cd m^{-2}. The chromaticity coordinates were almost independent of current density. Moreover, highly efficient red emission ($x = 0.63$, $y = 0.36$) was obtained in the 0.74 wt% doped device. The maximum external quantum efficiency was 4.46% at 7 V, the current efficiency was 3.43 cd A^{-1}, and the power efficiency was 1.64 lm W^{-1}. The highest brightness of 8300 cd m^{-2} was obtained at 19.6 V.

Introduction

Since the first report of organic light-emitting devices (OLEDs) made of thin films of organic material in the late 1980s,[1] a number of techniques for fabricating devices by doping fluorescent materials into an emitting layer have been used to achieve emissions with wavelengths corresponding to primary colors (*i.e.*, red, green and blue), which are necessary for color displays.[2,3] However, among the three primary colors, red has shown the lowest luminescence efficiency and has consequently come to be considered the most serious obstacle to attaining full-color display panels.

DCM2, a DCM derivative containing the julolidyl group, was one of the first materials used as a dopant for Alq_3 emitting layers.[2] Other extensively studied molecules include DCM derivatives,[4–8] Nile red,[9,10] perylene derivatives,[11–13] europium complexes,[14,15] and porphyrin derivatives.[16–18] In addition, nondopant type red-emitting materials have been reported recently.[19,20] Among them, 4-(dicyanomethylene)-2-*tert*-butyl-6-(1,1,7,7-tetramethyljulolidyl-9-enyl)-4*H*-pyran (DCJTB), a DCM derivative, is one of the most promising dopants and has been widely used in red OLEDs and white OLEDs.[4,5,10,21–24] However, the use of DCJTB as a red emitter is hindered by several factors: (1) preparations of asymmetric pyran derivatives entail several tedious reaction and purification steps; (2) the final yield is low;[4,5,25] and (3) the color purity of DCJTB-based devices is not good enough for display applications. Thus, the development of red emitters with a good quantum yield and chromaticity is a key issue for the production of full-color OLED-based displays.

In the present work, we report on a new family of red-emitting bis-DCM based dopants for OLED applications. This series of dopants is characterized by saturated red emission and moderately high fluorescence efficiency. In addition, these compounds can be synthesized with good yields and high product purities. Their photophysical properties and electroluminescence behavior as red emitters are investigated.

Experimental

Materials and instruments

All the reagents and solvents used for synthesis of red emitters were purchased from Aldrich and TCI companies and used without further purification. Tris(8-hydroxyquinolinato)aluminium (Alq_3) used for EL devices fabrication were purified by train sublimation.

NMR spectra were recorded on a Bruker AVANCE 400 spectrometer with tetramethylsilane as internal reference. The photoluminescence and absorption spectra of red emitters in solution and in films were recorded with a Spex Fluorolog-3 spectrofluorometer and a Jasco V-530 UV/vis spectrometer, respectively. Ionization potentials (IP) of materials were obtained using atmospheric photoelectron spectroscopy (RIKEN Keiki AC-2).

4-(Diethylamino)-2-butoxybenzaldehyde (1)

To a solution of 4-(diethylamino)-2-hydroxybenzaldehyde (3.0 g, 15.5 mmol) in DMSO (20 mL), sodium hydroxide (0.93 g, 23.2 mmol) and 1-bromobuthane (2.5 g, 18.2 mmol) were added. The mixture was heated for 8 h at 60 °C and then cooled to room temperature. The mixture was poured into ice water and extracted with ethyl acetate. The combined organic layer was washed with water and brine and dried over $MgSO_4$.

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† Electronic supplementary information (ESI) available: synthesis of bis-DCM derivatives (**7**, **8**, **9** and **10**). See http://www.rsc.org/suppdata/jm/b4/b419408j/

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The solvent was evaporated under reduced pressure to give 3.4 g (88%) of **1** as a brown liquid. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 10.16 (s, 1H), 7.69 (d, J = 8.9 Hz, 1H), 6.25 (d, J = 8.9 Hz, 1H), 6.00 (s, 1H), 4.01 (t, J = 6.6 Hz, 2H), 3.41 (q, J = 7.1 Hz, 4H) 2.02 (m, 2H), 1.50 (m, 2H), 1.20 (t, J = 7.1 Hz, 6H), 0.97 (t, J = 7.4 Hz, 3H). ^{13}C-NMR (100 MHz): δ 187.4, 162.8, 153.6, 130.4, 115.3, 104.5, 95.6, 67.9, 44.6, 31.3, 19.4, 13.8, 12.7.

4-(Diethylamino)-2-methylbenzaldehyde (2)

Phosphous oxychloride (10 mL, 107 mmol) was slowly added to dimethylformamide (75 mL) at 0 °C. After 30 minutes, *N*,*N*-diethyl-*m*-toluidine (14.3 g, 87.6 mmol) was added and then was heated at 90 °C for 3 h. The reaction mixture was then cooled, poured in to crushed ice, and neutralized with sodium acetate. The mixture was extracted with ethyl acetate. The extract was washed with water, dried over $MgSO_4$, and then concentrated. The bright brown oil **2** (13.5 g, 81%) was obtained after column chromatography with hexane–ethyl acetate (30 : 1) as an eluent. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 9.91 (s, 1H), 7.61 (d, J = 8.8 Hz, 1H), 6.52 (dd, J = 8.8, 2.6 Hz, 1H), 6.37 (s, 1H), 3.40 (q, J = 7.1 Hz, 4H), 2.59 (s, 3H), 1.19 (t, J = 7.1 Hz, 6H). ^{13}C-NMR (100 MHz): δ 190.1, 151.4, 143.1, 134.9, 122.9, 112.9, 108.4, 44.5. 20.4, 12.5.

1-(4-Methylphenyl)-1,2,3,4-hydroquinoline (3)

Tris(dibenzylideneacetone)dipalladium(0) ($Pd_2(dba)_3$) (1.03 g, 1.12 mmol), 1,1′-bis(diphenylphosphino)ferrocene (dppf) (0.96 g, 1.73 mmol), 4-bromotoluene (12.8 g, 75 mmol) were dissolved in 500 mL of dry toluene and stirred for 15 min. Sodium *tert*-butoxide (10.8 g, 112 mmol) and 1,2,3,4-hydroquinoline (10.0 g, 75 mmol) were then added. The reaction mixture was heated to 100 °C for 24 h, and then was portioned between ethyl acetate, and the aqueous layer was extracted with ethyl acetate. The combined organic fractions were dried over $MgSO_4$, and the solvent was evaporated under reduced pressure. Column chromatography (hexane) afforded 12.6 g (75%) of product **3**. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.14 (m, 4H), 7.00 (d, J = 7.3 Hz, 1H), 6.89 (t, J = 6.6 Hz, 1H), 6.64 (m, 2H), 3.58 (t, J = 5.6 Hz, 2H), 2.84 (t, J = 6.4 Hz, 2H), 2.34 (s, 3H), 2.05 (m, 2H). ^{13}C-NMR (100 MHz): δ 145.8, 144.9, 133.6, 130.0, 129.3, 126.3, 125.3, 123.8, 117.7, 115.1, 55.1, 27.8, 22.6, 20.9.

1-(4-Methylphenyl)-1,2,3,4-tetrahydroquinoline-6-carbaldehyde (4)

Compound **4** was prepared by analogy to **2** using 1-(4-methylphenyl)-1,2,3,4-hydroquinoline (**3**) instead of *N*,*N*-diethyl-*m*-toluidine in 80% yield. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 9.65 (s, 1H), 7.51 (s, 1H), 7.34 (dd, J = 8.7, 2.1 Hz, 1H), 7.24 (d, J = 6.1 Hz, 2H), 7.11 (d, J = 8.2 Hz, 2H), 6.44 (d, J = 8.6 Hz, 1H), 3.64 (t, J = 5.6 Hz, 2H), 2.89 (t, J = 6.3 Hz, 2H), 2.37 (s, 3H), 2.05 (m, 2H). ^{13}C-NMR (100 MHz): δ 190.2, 150.6, 143.6, 136.2, 131.1, 130.6, 129.8, 126.7, 126.1, 122.0. 112.9, 51.8, 27.7, 21.8, 21.0.

1-Hexyl-1,2,3,4-hydroquinoline (5)

1,2,3,4-Hydroquinoline (10.0 g, 7.5 mmol) was reacted with 1-bromohexane (18.6 g, 11 mmol) in a two-phase system composed of 20 mL toluene and 50 mL of 50 wt% aqueous NaOH using tetra(*n*-butyl)ammonium bromide as the phase transfer catalyst at 100 °C for 3 days. After diluting the reaction mixture with ethyl acetate, the organic layer was washed with water several times to remove excess NaOH. The organic layer was separated and dried over $MgSO_4$. The yellow liquid **5** (8.1 g, 50%) was obtained after column chromatography with hexane–ethyl acetate (10 : 1) ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.03 (t, J = 7.3 Hz,1H), 6.91 (d, J = 7.3 Hz, 1H), 6.53 (m, 2H), 3.26 (t, J = 5.5 Hz, 2H), 3.21 (t, J = 7.6 Hz, 2H), 2.73 (t, J = 6.4 Hz, 2H), 1.92 (m, 2H), 1.57 (m, 2H), 1.31 (m, 6H), 0.89 (t, J = 6.6 Hz, 3H). ^{13}C-NMR (100 MHz): δ 145.4, 129.1, 127.0, 122.1, 115.1, 110.4, 51.5, 49.4, 31.8, 28.2. 26.9. 26.2, 22.7, 22.3, 14.0.

1-Hexyl-1,2,3,4-tetrahydroquinoline-6-carbaldehyde (6)

Compound **6** was prepared by analogy to **2** using 1-hexyl-1,2,3,4-hydroquinoline (**5**) instead of *N*,*N*-diethyl-*m*-toluidine in 78% yield. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 9.62 (s, 1H), 7.52 (d, J = 8.6 Hz, 1H), 7.50 (s, 1H), 6.53 (d, J = 8.6 Hz, 1H), 3.36 (t, J = 5.6 Hz 2H), 3.29 (t, J = 7.6 Hz, 2H), 2.75 (t, J = 6.4 Hz, 2H), 1.92 (m, 2H), 1.59 (m, 2H), 1.33 (m, 6H), 0.88 (t, J = 6.6 Hz, 3H). ^{13}C-NMR (100 MHz): δ 190.0, 150.3, 131.2, 130.4, 124.5, 121.6, 109.3, 51.5, 49.8, 31.6, 28.0, 26.7, 26.3, 22.6, 21.5, 14.0.

All bis-DCM derivatives (**7**, **8**, **9** and **10**) were synthesized using Knoevenagel condensation between (2,6-dimethyl-4*H*-pyran-4-ylidine)propanedinitrile and the corresponding aldehyde (**1**, **2**, **4** and **6**), analogous to previously described methods.[25]†

Device fabrication and characterization

OLEDs were fabricated on glass substrates precoated with a layer of indium tin oxide (ITO), which were precleaned by ultrasonic treatment in detergent, de-ionized water, acetone and methanol, respectively, and were pretreated with oxygen plasma cleaner before use. Organic layers and cathode layers (LiF/Al) were deposited by conventional vacuum vapor deposition below 1×10^{-6} Torr. The emitting area was 2 × 2 mm. The doped devices have a structure of ITO/4,4′,4″-tris(3-methylphenylamino)triphenylamine (*m*-MTDATA) (20 nm)/*N*,*N*′-bis(1-naphthyl)-diphenyl-1,1′-biphenyl-4,4′-diamine (NPB) (40 nm)/tris(8-quinolinolato)-aluminium (Alq_3) : red dopant (35 nm)/Alq_3 (35 nm)/LiF (1 nm)/Al (100 nm). The electroluminescence (EL) spectrum and Commission International del'Eclairage (CIE) color coordinates were measured with a spectroradiometer (Minolta CS-1000). Current–voltage–luminance (*I*–*V*–*L*) measurements were performed with a current/voltage source (Keithley 238) and a luminescence meter (Minolta LS-100). Devices were tested in air without further encapsulation.

Results and discussion

Molecular design and synthesis

The structures of the novel red dopants (**7**–**10**), widely studied red dopants (DCM and DCJTB), and other bis-DCM derivatives (DADB and bis-DCJTB) are shown in Fig. 1. We

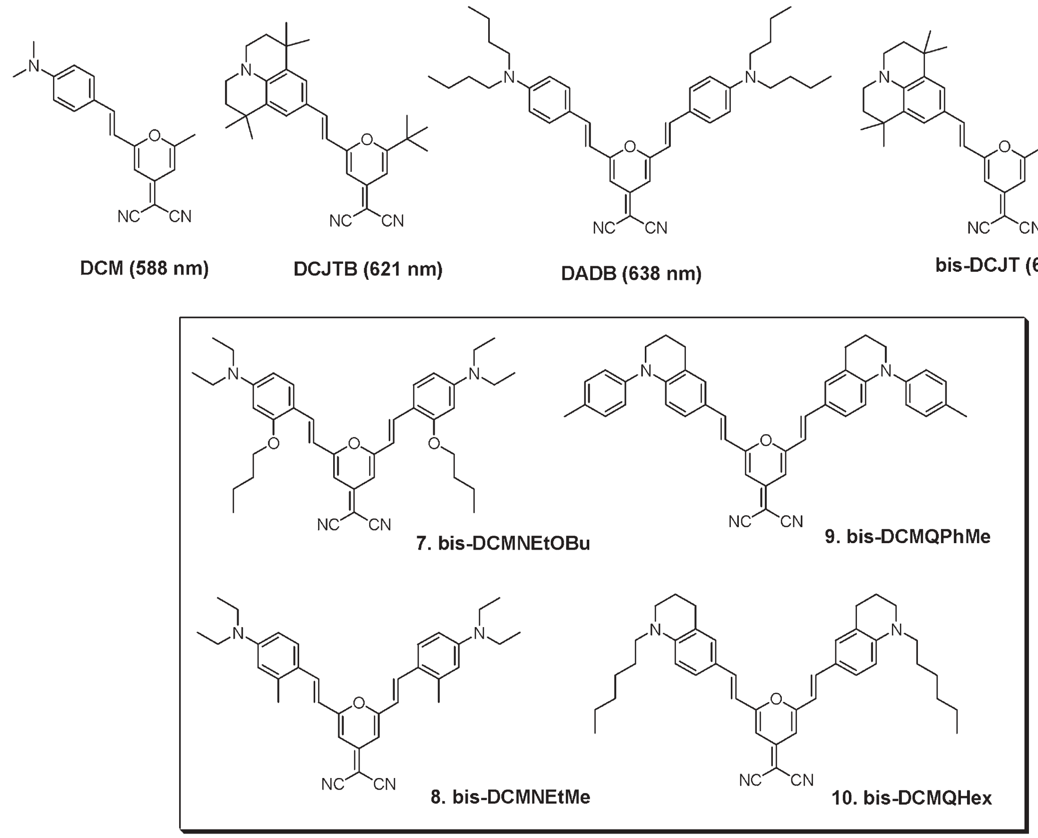
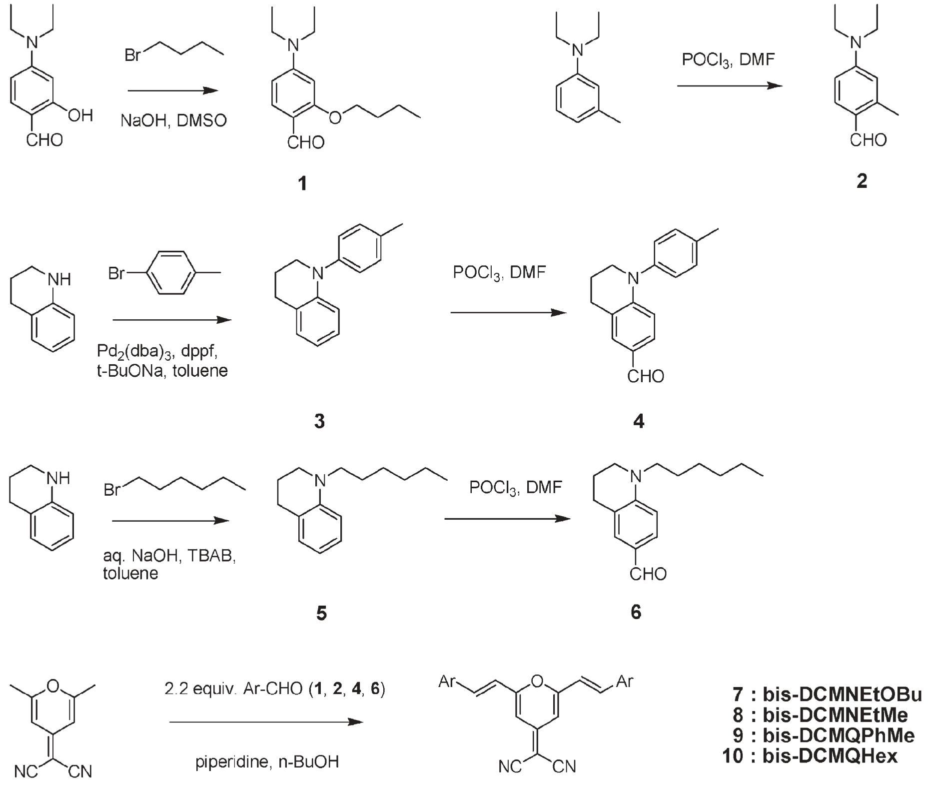


Fig. 1 Molecular structures of the new bis-DCM based red dopants, reported red dopants and other bis-DCM derivatives.



Scheme 1 Synthesis of the new bis-DCM based red dopants.

have previously reported that some bis-condensed DCM derivatives with moderate efficiency could be used as red dopants, in contrast to bis-DCJTB, which is known as an impurity.[5,26,27] The bis-DCM derivatives have a lower band gap (longer wavelength emission) and PL efficiency than their mono-DCM analogues. Generally, in doped red OLEDs there is a compromise between device efficiency and color purity due to concentration quenching.[5,28] Although the PL efficiency of DADB is lower than that of DCJTB, at the optimal doping concentration, the performance of red OLEDs using DADB was found to be comparable to that of red OLEDs using DCJTB.[29] These red dopants are intramolecular charge-transfer compounds with both strong electron donor and electron acceptor groups. The emission wavelength of these compounds can be tuned by changing the relative electron donating and accepting abilities of the donor and acceptor groups.[8,30,31] Our strategy in designing the new red-emitting materials discussed here was to maintain the bis-condensed conjugation system but replace its electron-donating group with one of higher electron donating power, thereby creating new red dopants that should show longer wavelength emission than DADB.

As illustrated in Scheme 1, the bis-DCM derivatives can be easily synthesized by two- or three-step processes including the synthesis of an aldehyde. Aldehyde **1** was synthesized by converting the hydroxy group to an alkoxy group. Other aldehydes **2**, **4**, and **6** were synthesized using a traditional synthetic procedure, known as the Vilsmeier reaction. Quantities in the range of several grams of the bis-DCM derivatives were obtained without byproducts. The samples were fully characterized by ^{1}H-NMR, ^{13}C-NMR and elemental analysis, and all findings were consistent with the proposed structures. The synthesis and purification procedures are simple, and most of the reagents are cheap. This is a significant advantage for large-scale commercial applications where minimizing production costs is important.

Photophysical properties and energy levels

Fig. 2 shows the absorption and emission spectra of the red emitters in 1,2-dichloroethane. The fluorescence maximum is dramatically red-shifted from 588 nm for DCM to 667 nm for **10**. The emission wavelengths for the new red emitters are longer than that for DADB, which can be explained by the fact that the additional donating groups are attached to the dialkylaminobenzene ring. The photophysical properties and energy level data for these red emitters are summarized in Table 1. They show moderately strong luminescence in 1,2-dichloroethane and indicate that **7**, in which the butoxy group enhances the steric hindrance of the molecules, has a higher PL quantum yield than DADB. Based on these findings, **7** is expected to be as highly efficient a red emitter as DADB. Other red emitters show deeper red emission than DADB but their PL quantum yields are lower than that of DADB. This is probably because they have relatively small energy gaps between the ground state and the lowest excited state, allowing more routes of nonirradiative decay.[32] Compared with the HOMO/LUMO energy levels of DADB, the DADB analogues **7** and **8**, which contain additional alkoxy and alkyl groups in *meta* positions to amino groups, have similar values, and **9** and **10** with tetrahydroquinoline donors have lower values.

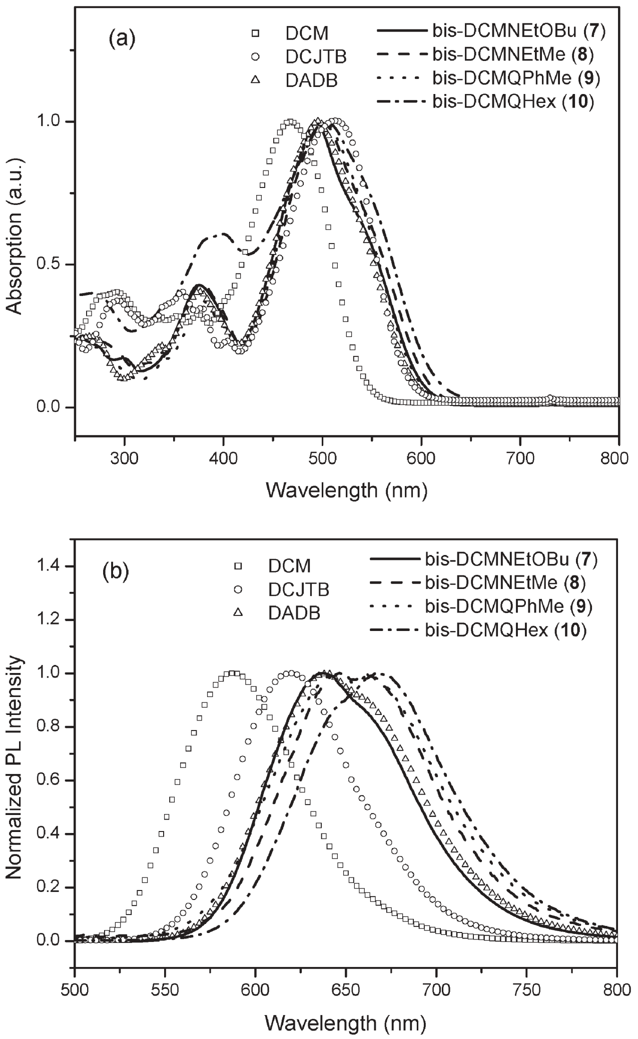


Fig. 2 (a) UV/vis absorption and (b) photoluminescence spectra of red dopants.

EL performances

The red dopant used in the EL devices was compound **7**, which had the highest PL quantum yield among the new bis-DCM

Table 1 Physical data for previous red dopants and new bis-DCM classes (**7**, **8**, **9**, **10**)

	Solution[a]		Film		
Compd.	λ_{max}/nm (ε_{max}/ M^{-1} cm^{-1})	λ_{em} (Φ_f[b])/ nm	λ_{max}/ nm	λ_{em}/ nm	HOMO[c]/LUMO[d] (band gap)/eV
7	494 (82800)	637 (0.52)	493	676	5.15/3.24 (1.91)
8	499 (71600)	649 (0.23)	500	695	5.15/3.27 (1.88)
9	500 (71000)	646 (0.33)	507	681	5.26/3.34 (1.92)
10	505 (49300)	667 (0.35)	509	688	5.34/3.48 (1.86)
DADB	496 (76100)	638 (0.47)	493	668	5.16/3.19 (1.97)
DCM	468 (38900)	588 (0.85)			
DCJTB	513 (41200)	621 (0.78)			

[a] In 1,2-dichloroethane. [b] The fluorescence quantum yield determined relative to DCJTB (0.78).[7,8] [c] Measured by a RIKEN Keiki AC-2. [d] Estimated from the HOMO and optical band gap.

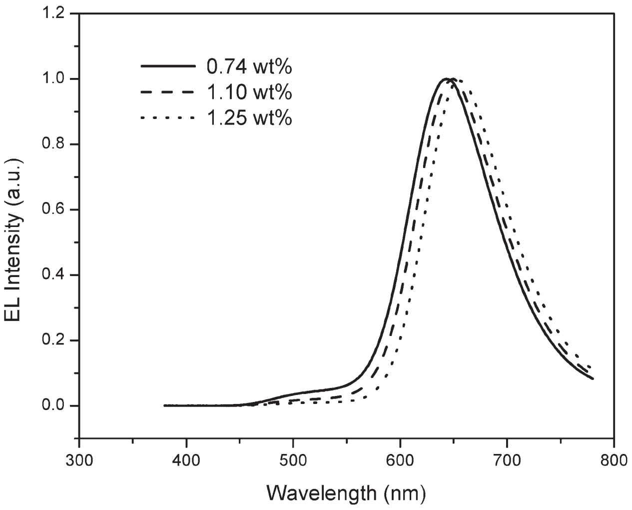


Fig. 3 EL spectra of red OLEDs with the configuration ITO/*m*-MTDATA (20 nm)/NPB (40 nm)/Alq_3 : red dopant (**7**) (35 nm, *x* wt%)/Alq_3 (35 nm)/LiF/Al at 20 mA cm^{-2}.

derivatives. The EL emission spectra of the devices at doping concentrations of 0.74 wt%, 1.10 wt% and 1.25 wt% are shown in Fig. 3. The EL performances of these devices are summarized in Table 2. It can be seen that the devices emit saturated red emission ranging from 643 nm to 654 nm, depending on the doping concentration. In particular, the device with 1.25 wt% of **7** shows pure red emission whose color coordinates are the same as that of the National Television System Committee (NTSC) standard red (0.67, 0.33). The brightness and current efficiency reach 2500 cd m^{-2} and 1.29 cd A^{-1}, respectively. In addition, the color coordinates of the device with 1.25 wt% of **7** are almost independent of current density, as demonstrated in Fig. 4. The EL device has a high brightness of several hundred cd m^{-2} while maintaining good color purity at a practically applicable current density of several tens of mA cm^{-2}.

Fig. 5 shows the voltage–efficiency characteristics of the device doped with 0.74 wt% of **7**. By a trade-off between efficiency and color, a relatively high efficiency and red emission with moderate color purity can be achieved. The external quantum efficiency is 4.46% at 7 V, the current efficiency is 3.43 cd A^{-1} and the power efficiency is 1.64 lm W^{-1}. The maximum brightness is greater than 8000 cd m^{-2}, suggesting that this material is quite good as a red dopant. While the EL performance of the present red OLEDs is still not ideal, it can be seen that the EL device is as good as those made from DCJTB and the color coordinates of the devices are better.[4,5,10,21,22] Recently, some methods have been reported for the enhancement of color purity or performance in red OLEDs. These methods are introducing an emitting assisted dopant,[21,33] improving the host material,[22,34] and using a phosphorescent sensitizer.[17,35,36] Compound **7** can be a promising red dopant in OLED applications through the device structural optimization,[37] which is under investigation.

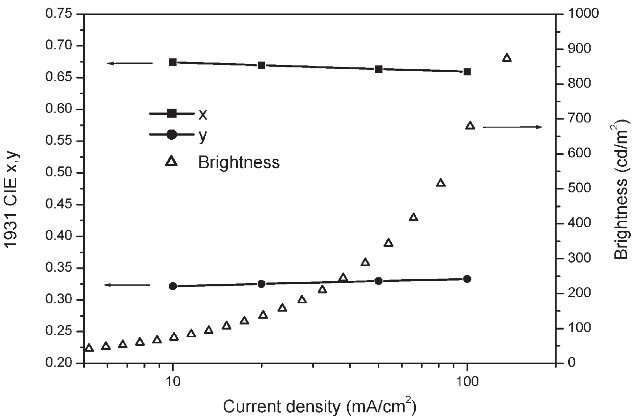


Fig. 4 Current density *vs.* CIE (*x*,*y*) coordinate for emission of selected pure red OLED (1.25 wt%) and luminance (*J*–*L*) characteristics for OLED (1.25 wt%).

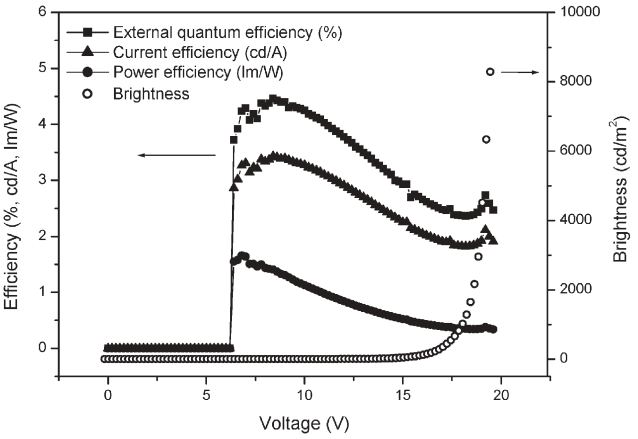


Fig. 5 Efficiency (external quantum, power and current efficiency) *vs.* voltage characteristics for the selected OLED (0.74 wt%) and luminance (*V*–*L*) characteristics for the OLED (0.74 wt%).

Conclusions

A series of bis-DCM compounds with various donor groups have been designed and synthesized. Their saturated red emissions, which are of longer wavelength than those of the

Table 2 Electroluminescence performance for the present red OLEDs with the configuration ITO/*m*-MTDATA (20 nm)/NPB (40 nm)/Alq_3 : red dopant (**7**) (35 nm, *x* wt%)/Alq_3 (35 nm)/LiF/Al

	At 20 mA cm^{-2}					
%	Brightness/cd m^{-2}	1931 CIE (*x*,*y*)	η/cd A^{-1}	λ_{em}/nm	B_{max}/cd m^{-2} (at voltage/V, current density/mA cm^{-2})	η_{max} (%, cd A^{-1}, lm W^{-1}
0.74	397	(0.63, 0.36)	1.99	643	8290 (19.6, 434)	4.46, 3.43, 1.64
1.10	236	(0.65, 0.34)	1.18	649	5070 (21.4, 394)	4.42, 2.91, 1.69
1.25	137	(0.67, 0.33)	0.69	654	2500 (21.2, 423)	2.95, 1.29, 0.99

mono-DCM derivatives, make them excellent candidates for red dopants in red OLEDs. In particular compound **7** is an excellent choice as a red dopant, as it affords improved color purity and high external quantum efficiency.

Acknowledgements

This work was supported by the Center for Advanced Functional Polymers (CAFPoly) through KOSEF and the Ministry of Information and Communications (MIC) of Korea.

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