

# 嘉南藥理科技大學專題研究計畫成果報告

合成 Imidazole 和 Polyphenylquinoxaline 衍生物在電激發光的應用

計畫類別：個別型計畫 整合型計畫

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計畫主持人：劉常興

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嘉南藥理科技大學專題研究計畫成果報告

**Bis(arylquinoxaliny)carbazole derivatives as saturated blue emitters for electroluminescent devices**

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**Abstract**

Bis(arylquinoxaliny)carbazole derivatives  
9-phenyl-3,6-bis-(3-phenyl-quinoxalin-2-yl)-9H-carbazole (PPQC),  
9-phenyl-3,6-bis-[3-(4-methoxy-phenyl)-quinoxalin-2-yl]-9H-carbazole (PMQC) and  
9-phenyl-3,6-bis-(3-p-tolyl-quinoxalin-2-yl)-9H-carbazole (PTQC) were conveniently synthesized from the corresponding tetraone and o-phenylenediamine. Electroluminescent devices using PTQC as the dopant emitters were fabricated. These devices all emit saturated blue light from the PTQC doped layer. Device C that consists of CuPc (10 nm)/TCTA (30 nm)/PTQC:TPBI (7%, 30 nm)/TPBI (40 nm) shows the highest performance. The device emits blue light at 446 nm with CIE values of (0.15, 0.08) and shows a maximum external quantum efficiency of 1.31 %, current efficiency of 1.00 cd/A, and brightness of 5067 cd/m<sup>2</sup>.

**Introduction**

Since the ground works by Tang and his coworkers [1], organic light emitting

diode (OLED) has been a great interest topic for many researchers due to its potential application in flat panel displays [2-4]. The search for new efficient and stable emitting materials with proper Commission Internationale de L'Eclairage (CIE) coordinates for full-color displays remains as one of the most active areas of these studies. Even though several reports on blue OLEDs are available in literature, efficient ones with excellent CIE coordinates and high brightness are still rare [5-7]. In the search for new blue emitters for electroluminescent devices, we design and synthesize a series of blue-light emitting material in which quinoxaline species are attached to the 9-phenylcarbazole core. It appears that no quinoxaline derivative has been used as emitter for electroluminescent devices, although an example of this type of compound as the hole blocking material was reported in the literature [8]. In this paper, we report the results of using these new materials as the dopant emitter in electroluminescent devices.

## Results and discussion

### 3.1. Physical properties of bis(arylquinoxaliny)carbazole derivatives 2

As shown in Scheme 2, bis(arylquinoxaliny)carbazole derivatives 2 were conveniently prepared in good yields from the condensation of the corresponding tetraone 1 with *o*-phenylenediamine in refluxing acetic acid. The key starting material tetraone 1 was synthesized by two steps from 3,6-diiodo-9-phenylcarbazole. First, 3,6-diiodo-9-phenylcarbazole was coupling with an excess of 1-alkyne derivatives to afford the 9-phenyl-3,6-bis-arylethynyl-9H-carbazole derivatives in the presence of palladium catalyst. Second, these derivatives were oxidized to give the corresponding tetraone 1 by using oxidizing reagent, iodine/dimethyl sulfoxide. All compounds 2 were characterized by <sup>1</sup>H NMR and high resolution mass spectral data.

The physical data of bis(arylquinoxaliny)carbazole derivatives 2 synthesized are summarized in Table 1. These compounds exhibit clear high glass transition points (T<sub>g</sub>) at 157-162 °C and melting points (T<sub>m</sub>) higher than 280 °C. As shown in Fig. 1, compounds 2 reveal similar UV-vis spectra in dichloromethane solution with two major absorptions at 294 and ~380 nm. In addition, these compounds emit

strong blue light in dichloromethane solution with the emission maximums at 464-466 nm (Fig. 1). The quantum yields of these three compounds in dichloromethane solution as listed in Table 1 are very close to each other and are between 21.3-21.6% [13]. The HOMO and LUMO energy levels of these derivatives measured from cyclic voltametry and from UV-vis spectra are 5.8-5.9 and 2.8-2.9 eV, respectively. The PL spectra of compounds 2 depend greatly on the solvent employed. Fig. 2 displays the PL spectra of PTQC, 2c in various solvents. The emission maxima appear at 440 ~ 496 nm. In non polar solvent such as toluene, the emission occurs at 440 nm, while in the polar solvent CH<sub>3</sub>CN, the emission peak appears at 496 nm. The variation in different solvents is likely due to the polarization-induced spectral shift [14]. As the polarity of the solvent increases, the emission maximum shifts towards the longer wavelength region. Similar spectral shift is also observed for the others compounds 2a-b in different solvents.

### 3.2. Device characteristics using PTQC as a dopant

The observed strong emission at 464-466 nm in dichloromethane and high T<sub>g</sub> point for compound 2 suggests that this compound is potentially a blue dopant emitter in organic electroluminescent devices. In order to understand the electroluminescent ability of these compounds, three

devices (device A-C) using PTQC as the dopant emitters were fabricated. TPBI (1,3,5-tris[N-phenylbenzimidazol-2-yl]benzene) was chosen as the host layer for device fabrication considering that the excellent overlapping of the UV-vis absorptions of PTQC with the PL spectrum of TPBI and the HOMO/LUMO energy levels. CuPc (copper phthalocyanine), TCTA (4,4',4''-tri[N-carbazolyl]triphenylamine) and TPBI were used as the hole injection layer, hole transporting layer, and electron transporting layer, respectively. Key characteristics of these devices are listed in Table 2. The relative energy levels and molecular structures of the materials used in one of these devices are displayed in Fig. 3. Device A is a three-layer device consisting of the following layers: ITO/TCTA (30 nm)/PTQC: TPBI (7%, 20 nm)/TPBI (50 nm). The luminance and current density versus voltage characteristics of this device is shown in Fig. 4. The device emits sharp blue light at 444 nm with a FWHM of 60 nm and the EL spectra do not change significantly with variation of the applied voltage as displayed in Fig. 5. Key electroluminescent data extracted from Fig. 4 are listed in Table 2. A turn-on voltage (brightness = 1 cd/m<sup>2</sup>) of 3.3 V with maximum brightness of 3727 cd/m<sup>2</sup> at 12 V, maximum external quantum efficiency of 0.98% and maximum current efficiency of 0.69 cd/A at 5.5 V, respectively, are achieved

for this device. Based on the EL spectrum at an applied voltage of 8 V, the CIE coordinates were calculated to be (0.15, 0.08). The values are just on the recommended blue standard for a video display by the National Television Standards Committee (NTSC) [15]. Device B has the same structure as A, except that the thickness of the emitting layer is increased to 30 nm and thickness of the electron transporting layer is decreased to 40 nm. The detailed device performance data are also listed in Table 2. This device also emits pure blue light at 446 nm with maximum brightness of 4838 cd/m<sup>2</sup> and maximum external quantum efficiency of 1.06%. The CIE values do not change significantly at applied voltages of 6 V to 12 V. Comparison of these two devices indicates that device B shows higher brightness perhaps caused by the higher thickness of emitting layer. In device C, a layer of CuPc was used as the hole injection layer to improve the device performance. A turn-on voltage of 4.5 V with maximum brightness of 5067 cd/m<sup>2</sup> at 14 V, external quantum efficiency of 1.31%, current efficiency of 1.0 cd/A at 9 V, and power efficiency of 0.38 lm/W at 7.5 V, respectively, were achieved. As shown in the energy level diagram in Fig. 3, CuPc is introduced to increase the hole-injection ability from ITO to the HOMO level of TCTA, therefore the recombination of holes and electrons in the emitting layer will be more efficient.

## Experimental

Melting points ( $T_m$ ), glass transition temperature ( $T_g$ ) and crystallization temperature ( $T_c$ ) were determined by differential scanning calorimetry (DSC) using a TA DSC Q10 instrument. The oxidation potentials were measured by cyclic voltammetry (CV) using an electrochemical analyzer CHI600A. The HOMO levels were calculated from the oxidation potentials, while the LUMO levels were calculated based on the HOMO levels and the lowest-energy absorption edges of the UV-vis absorption spectra [9]. UV-vis absorption spectra were recorded using a Hitachi U-3300 spectrophotometer while photoluminescence (PL) and electroluminescence (EL) spectra were measured on a Hitachi F-4500 fluorescence spectrophotometer.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded with Bruker 200 spectrometers. High resolution mass spectra were taken with a Thermo Finnigan MAT 95 XL instrument. Elemental analyses were performed by Elementar vario EL instrument.

The EL devices were fabricated by vacuum deposition of the materials at  $<5 \times 10^{-6}$  Torr onto a clean glass precoated with a layer of indium tin oxide with a sheet resistance of 25 ohm/square. The deposition rate for organic compounds was 1-2 Ås $^{-1}$ . The cathode was a Mg:Ag alloy (10:1, 55 nm) and was deposited by co-evaporation of Mg and Ag metals with deposition rates of 5 and 0.5 Ås $^{-1}$

respectively. The cathode was then capped with Ag metal (100 nm) by evaporation of Ag with a rate of 3 Ås $^{-1}$ . The effective area of the emitting diode is 9.00 mm $^2$ . Current, voltage and light intensity measurements were made simultaneously using a Keithley 2400 source meter and a Newport 1835-C optical meter equipped with a Newport 818-ST silicon photodiode. All chemicals used for EL devices were sublimed in vacuum prior to use.

### 2.1. General procedure for the synthesis of tetraone derivatives (1a-1c)

Tetraone derivatives (1a-1c) were obtained from the corresponding 3,6-dialkynyl-carbazole compounds. The synthetic route to the tetraone derivatives is shown in Scheme 1. 3,6-Diiodo-9-phenylcarbazole was synthesized from carbazole in two steps according the literature methods [10-11]. The synthetic procedure for tetraone 1a is as follows.

3,6-Diiodo-9-phenylcarbazole (2.00 g, 4.03 mmol), Pd(PPh $_3$ ) $_4$  (0.46 g, 0.40 mmol) and cuprous iodide (0.11 g, 0.58 mmol) were dissolved in 40 mL of diethylamine under a nitrogen atmosphere. Phenylacetylene (1.03 g, 10.06 mmol) was added to the above solution and the mixture was stirred at 60 oC for 8 h. After removal of diethylamine under reduced pressure, ethyl acetate was added to the residue and the precipitate was filtered off. The concentrated ethyl acetate-extract was purified by silica gel column using

hexane/ethyl acetate as the eluent to give 1.59 g of

9-phenyl-3,6-bis-phenylethynyl-9H-carbazole.

9-Phenyl-3,6-bis-phenylethynyl-9H-carbazole (1.00 g, 2.26 mmol) and I2 (1.20 g, 4.51 mmol) were dissolved in 12 mL of DMSO and the solution was heated at 150 °C for 12 h [12]. After cooling down, water was added to the solution and brown precipitate formed. The solid was collected and washed with saturated Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> aqueous solution, H<sub>2</sub>O and dried to give 1.05 g of

1-[6-(2-oxo-2-phenyl-acetyl)-9-phenyl-9H-carbazol-3-yl]-2-phenyl-ethane-1,2-dione (1a) (yield = 92.1%). By the similar procedure,

1-(4-methoxy-phenyl)-2-{6-[2-(4-methoxy-phenyl)-2-oxo-acetyl]-9-phenyl-9H-carbazol-3-yl}-ethane-1,2-dione (1b) and 1-[6-(2-oxo-2-p-tolyl-acetyl)-9-phenyl-9H-carbazol-3-yl]-2-p-tolyl-ethane-1,2-dione (1c) were obtained in 87.2 and 73.4% yields, respectively.

2.1.1. 1a:

1-[6-(2-Oxo-2-phenyl-acetyl)-9-phenyl-9H-carbazol-3-yl]-2-phenyl-ethane-1,2-dione

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 200 MHz), δ (ppm): 7.45 (d, J = 8.7 Hz, 2H, H-1, H-8), 7.71-7.50 (m, 11H), 8.06 (dd, J = 8.2 Hz, J = 1.5 Hz, 4H), 8.17 (dd, J = 8.7 Hz, J = 1.6 Hz, 2H, H-2, H-7), 8.75 (d, J = 1.6 Hz, 2H, H-4, H-5). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 50 MHz), δ (ppm): 111.0, 123.3, 124.4, 126.6, 127.1, 128.6, 129.0, 129.2, 130.1, 130.5, 133.2, 134.9, 135.6, 145.5, 194.4,

193.8. HRMS: calculated M<sup>+</sup> 507.1471; observed M<sup>+</sup> 507.1473.

2.1.2. 1b:

1-(4-Methoxy-phenyl)-2-{6-[2-(4-methoxy-phenyl)-2-oxo-acetyl]-9-phenyl-9H-carbazol-3-yl}-ethane-1,2-dione

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 200 MHz), δ (ppm): 3.89 (s, 6H, OCH<sub>3</sub>), 6.99 (d, J = 8.9 Hz, 4H), 7.43 (d, J = 8.7 Hz, 2H, H-1, H-8), 7.58-7.78 (m, 5H, 9-phenyl), 8.02 (d, J = 8.9 Hz, 4H), 8.16 (dd, J = 8.7 Hz, J = 1.6 Hz, 2H, H-2, H-7), 8.70 (d, J = 1.6 Hz, 2H, H-4, H-5). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 50 MHz), δ (ppm): 55.6, 110.9, 114.4, 123.3, 124.5, 126.3, 126.8, 127.1, 128.5, 129.2, 130.4, 132.5, 135.6, 145.4, 165.0, 193.6, 194.2. HRMS: calculated M<sup>+</sup> 567.1682; observed M<sup>+</sup> 567.1683.

2.1.3. 1c:

1-[6-(2-Oxo-2-p-tolyl-acetyl)-9-phenyl-9H-carbazol-3-yl]-2-p-tolyl-ethane-1,2-dione

<sup>1</sup>H NMR (CDCl<sub>3</sub>, 200 MHz), δ (ppm): 2.45 (s, 6H, CH<sub>3</sub>), 7.33 (d, J = 8.4 Hz, 4H), 7.43 (d, J = 8.7 Hz, 2H, H-1, H-8), 7.70-7.47 (m, 5H, 9-phenyl), 7.93 (d, J = 8.4 Hz, 4H), 8.15 (dd, J = 8.7 Hz, J = 1.7 Hz, 2H, H-2, H-7), 8.69 (d, J = 1.7 Hz, 2H, H-4, H-5). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 50 MHz), δ (ppm): 22.0, 111.0, 123.4, 124.5, 126.7, 127.1, 128.5, 129.2, 129.8, 130.2, 130.5, 130.8, 135.7, 145.5, 146.3, 194.1, 194.7. HRMS: calculated M<sup>+</sup> 535.1784; observed M<sup>+</sup> 535.1782.

2.2. General Procedure for the Synthesis of bis(arylquinoxaliny)carbazole derivatives (2a-2c)

Tetraone 1a (2.0 g, 3.94 mmol) and

o-phenylenediamine (0.92 g, 8.68 mmol) were dissolved in THF (40 mL) in a 100 mL round-bottomed flask. 15 mL of acetic acid was added to the flask and the mixture was stirred under refluxing for 10 h. After cooling to room temperature, methanol was poured into the mixture with stirring. The precipitate was collected and washed with methanol and then dried in vacuum to give a crude product of 2a. The product was further purified by vacuum sublimation technique at 250 °C and 3-5x10<sup>-3</sup> Pa. The other derivatives 2b and 2c were prepared according to a similar procedure from the corresponding tetraone. The yield and important spectral data are given below.

#### 2.2.1. 2a:

9-Phenyl-3,6-bis-(3-phenyl-quinoxalin-2-yl)-9H-carbazole (PPQC)

Yield: 88%. Mp. = 284 oC. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 200 MHz), δ (ppm): 7.28 (d, J = 8.6 Hz, 2H, H-1, H-8), 7.31-7.35 (m, 7H), 7.47 (dd, J = 8.6 Hz, J = 1.7 Hz, 2H, H-2, H-7), 7.55-7.62 (m, 8H), 7.75-7.80 (m, 4H, quinoxaline H's), 8.17-8.24 (m, 4H, quinoxaline H's), 8.40 (d, J = 1.7 Hz, 2H, H-4, H-5). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 50 MHz), δ (ppm): 109.5, 122.4, 123.6, 126.9, 127.8, 128.3, 128.4, 128.8, 129.1, 129.2, 129.6, 129.7, 129.8, 130.0, 131.3, 137.1, 139.5, 141.0, 141.4, 141.5, 153.6, 153.8. HRMS: calculated M<sup>+</sup> 651.2423; observed M<sup>+</sup> 651.2430. Anal. Calcd for C<sub>46</sub>H<sub>29</sub>N<sub>5</sub>: C, 84.77; H, 4.48; N, 10.75. Found: C, 84.72; H, 4.44; N, 10.70.

#### 2.2.2. 2b:

9-Phenyl-3,6-bis-[3-(4-methoxy-phenyl)-quinoxalin-2-yl]-9H-carbazole (PMQC)

Yield: 86%. Mp. = 302 oC. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 200 MHz), δ (ppm): 3.39 (s, 6H, OCH<sub>3</sub>), 6.84 (d, J = 8.8 Hz, 4H), 7.31 (d, J = 8.6 Hz, 2H, H-1, H-8), 7.45 (dd, J = 8.6 Hz, J = 1.7 Hz, 2H, H-2, H-7), 7.53-7.61 (m, 9H), 7.73-7.78 (m, 4H, quinoxaline H's), 8.15-8.21 (m, 4H, quinoxaline H's), 8.52 (d, J = 1.7 Hz, 2H, H-4, H-5). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 50 MHz), δ (ppm): 55.3, 109.5, 113.8, 122.3, 123.8, 126.9, 127.8, 128.3, 129.0, 129.5, 129.9, 131.3, 131.7, 131.8, 137.1, 141.1, 141.2, 141.5, 153.2, 153.8, 160.1. HRMS: calculated M<sup>+</sup> 711.2634; observed M<sup>+</sup> 711.2636. Anal. Calcd for C<sub>48</sub>H<sub>33</sub>N<sub>5</sub>O<sub>2</sub>: C, 80.99; H, 4.67; N, 9.84. Found: C, 80.62; H, 4.63; N, 9.71.

#### 2.2.3. 2c:

9-Phenyl-3,6-bis-(3-p-tolyl-quinoxalin-2-yl)-9H-carbazole (PTQC)

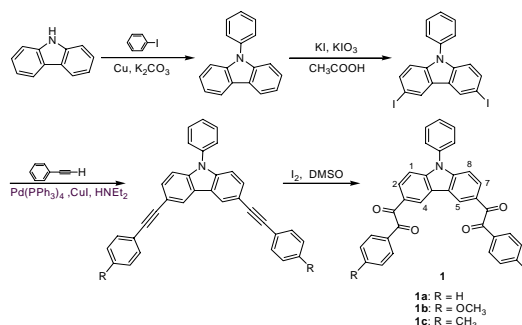
Yield: 87%. Mp. = 309 oC. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 200 MHz), δ (ppm): 2.34 (s, 6H, CH<sub>3</sub>), 7.13 (d, J = 7.9 Hz, 4H), 7.31 (d, J = 8.6 Hz, 2H, H-1, H-8), 7.58-7.42 (m, 11H), 7.73-7.78 (m, 4H, quinoxaline H's), 8.17-8.20 (m, 4H, quinoxaline H's), 8.48 (d, J = 1.6 Hz, 2H, H-4, H-5). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 50 MHz), δ (ppm): 21.4, 109.5, 122.4, 123.8, 126.9, 127.8, 128.4, 129.1, 129.2, 129.5, 129.7, 129.9, 131.6, 136.6, 137.2, 138.7, 141.1, 141.3, 141.5, 153.7, 153.9. HRMS: calculated M<sup>+</sup> 679.2736; observed M<sup>+</sup> 679.2728. Anal. Calcd for C<sub>48</sub>H<sub>33</sub>N<sub>5</sub>: C, 84.81; H, 4.89; N, 10.30. Found: C, 84.77; H, 4.92; N, 10.25.

## Conclusions

We have successfully synthesized three bis(arylquinoxaliny)carbazole derivatives. These materials show excellent physical properties such as remarkable thermal stability, high melting and glass-transition points. The PL spectra of these compounds exhibit strong blue emission in solution state. The PTQC-based EL devices emit blue light at ~445 nm with good brightness and excellent CIE coordinates. To the best of our knowledge, these are the only few devices that emit saturated blue light with CIE coordinates on the NTSC standard primary blue. Extension of the studies to other compounds of this series is still in progress.

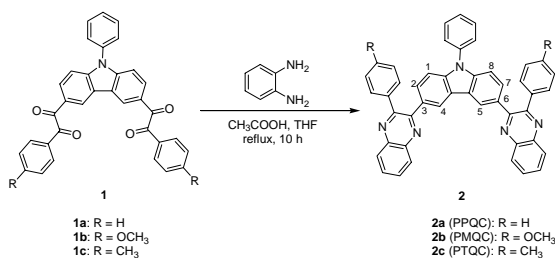
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Scheme 1. Synthesis of tetraone compounds (1a-c).





Scheme 2. Synthesis of bis(arylquinoxaliny)carbazole derivatives (2a-c).

Table 2. Performance of PTQC-Based OLEDs

Device	turn-on voltage (V)	$\eta_{ext}$ (% V)	$L$ (cd/m <sup>2</sup> , V)	$\eta_e$ (cd/A, V)	$\eta_p$ (lm/W, V)	$\lambda_{max}$ (nm)	CIE <sub>s</sub> , 8 V (x, y)
A	3.3	0.98,	3727,	0.69,	0.43,	444	(0.15, 0.08)
		5.5	12	5.5	4.5		
B	4.2	1.06,	4838,	0.79,	0.28,	446	(0.15, 0.08)
		9	13.5	8.5			
C	4.5	1.31,	5067,	1.00,	0.38,	446	(0.15, 0.08)
		9	14	7.5			

<sup>a</sup>The data for external quantum efficiency ( $\eta_{ext}$ ), brightness ( $L$ ), current efficiency ( $\eta_e$ ) and power efficiency ( $\eta_p$ ) are the maximum values of the device.

<sup>b</sup>Device A: TCTA (30 nm)/PTQC:TPBI (7%, 20 nm)/TPBI (50 nm); B: TCTA (30 nm)/PTQC:TPBI (7%, 30 nm)/TPBI (40 nm); C: CuPc (10 nm)/TCTA (30 nm)/PTQC:TPBI (7%, 30 nm)/TPBI (40 nm).

## Figure Legends.

Fig. 1. The UV absorption and PL spectra of bis(arylquinoxaliny)carbazole derivatives 2 in dichloromethane solution (concentration = 10<sup>-5</sup> M).

Fig. 2. The PL spectra of 2c (PTQC) in different solvents (concentration = 10<sup>-5</sup> M).

Fig. 3. The relative energy levels and molecular structures of the materials used in device C [CuPc (10 nm)/TCTA (30 nm)/PTQC: TPBI (7%, 30 nm)/TPBI (40 nm)].

Fig. 4. Luminance and current density versus voltage characteristics of device A [TCTA (30 nm)/PTQC: TPBI (7%, 20 nm)/TPBI (50 nm)].

Fig. 5. The EL spectra of device A at various applied voltages.

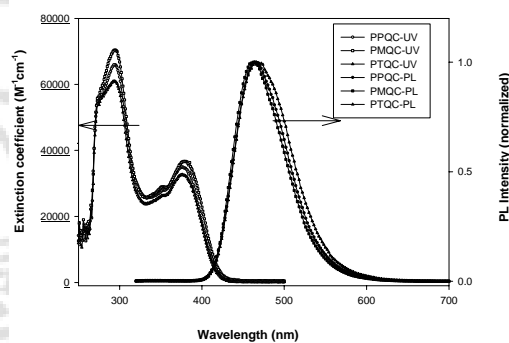


Fig. 1

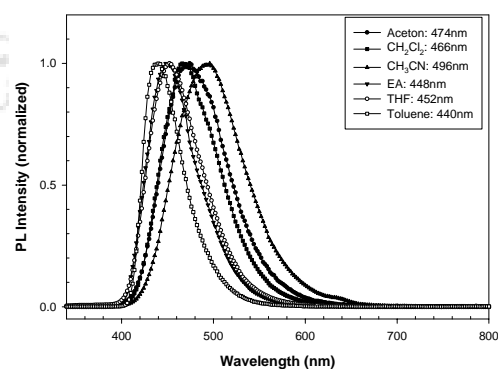


Fig. 2

Table 1. Physical properties of the

bis(arylquinoxaliny)carbazole derivatives 2

Compound	$T_m/T_g/T_c^a$ (°C)	$\lambda_{max}(abs)^b$ (nm)	$\lambda_{em}^c$ (nm)	Q.Y. <sup>d</sup> (%)	HOMO/LUMO (eV)
2a (PPQC)	284/157/ND	294, 376	464	21.5	5.9/2.9
2b (PMQC)	302/159/263	294, 380	464	21.3	5.8/2.8
2c (PTQC)	309/162/260	294, 377	466	21.6	5.9/2.9

<sup>a</sup>Obtained from DSC measurement; ND = not detected.

<sup>b</sup>Measured in a CH<sub>2</sub>Cl<sub>2</sub> solution.

<sup>c</sup>Measured in a CH<sub>2</sub>Cl<sub>2</sub> solution.

<sup>d</sup>Measured in a CH<sub>2</sub>Cl<sub>2</sub> solution by using coumarin 1 as a reference.

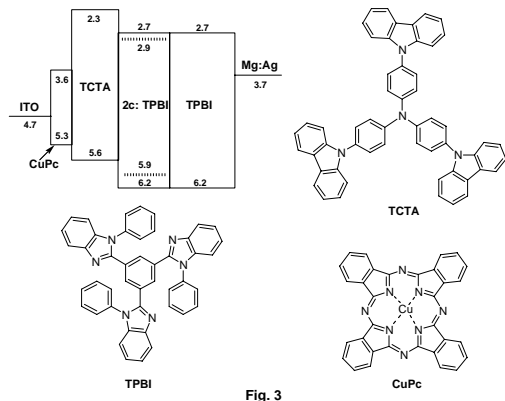


Fig. 3

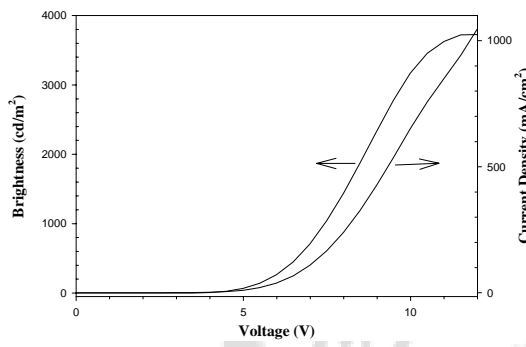


Fig. 4

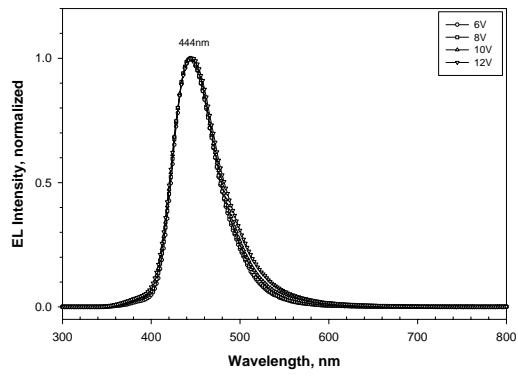


Fig. 5