Received: 23 October 2017 | Revised: 8 December 2017 | Accepted: 12 December 2017
DOI: 10.1002/bio.3452

RESEARCH ARTICLE

WILEY LUMINESCENCE
The Journal of Biological and Chemical Luminescence

Synthesis and properties of blue luminescent bipolar materials constructed with carbazole and anthracene units with 4-cyanophenyl substitute at the 9-position of the carbazole unit

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Funding information
Guangzhou Institute of Technology Key Projects Foundation, Grant/Award Number: ZSH201609; Guangzhou City Education System Innovative Academic Team Foundation, Grant/Award Number: 1201610020

Abstract

With carbazole and *p*-cyanobromobenzene as raw materials, 4-(3,6-di (anthracen-9-yl)-9H-carbazol-9-yl)benzonitrile (DACB) and 4-(3,6-bis(anthracene -9-ylethynyl)-9H-carbazol-9-yl) benzonitrile (BACB) were synthesized through the Suzuki coupling reaction and the Sonogashira coupling reaction, respectively. These structures were characterized using ^{1}H nuclear magnetic resonance (NMR), elemental analysis and mass spectrometry. Their thermal properties, ultraviolet–visible (UV-vis) absorption, fluorescence emission, fluorescence quantum yields and electrochemical properties were also investigated systematically. In addition, a electroluminescence (EL) device was made with BACB as the emitting layer and performance of the EL device was studied. Results showed that: (1) the temperature points with 5% and 10% of DACB weight loss were 443°C and 461°C, respectively, and were 475°C and 506°C with BACB weight loss of 5% and 10%, respectively. When the temperature was 50–300°C, no significantly thermal transition was observed which suggested that they had excellent thermal stability. (2) DACB and BACB had single emission peaks at 415 nm, and 479 nm with fluorescence quantum yields of 0.61 and 0.87, respectively, indicating that both compounds could emit strong blue light. (3) According to electrochemical measurement on BACB and DACB, their gaps were 3.07 eV and 2.76 eV, respectively, which further showed that these two compounds were very stable and acted as efficient blue light materials. (4) The turn-on voltage of the device was 5 V, and the device emitted dark blue light with Commission Internationale de L'Eclairage (CIE) coordinates of (0.157, 0.079).

KEYWORDS

bipolar materials, blue luminescent, quantum yield

1 | INTRODUCTION

Red, green, and blue colour materials are the basis for full colour display, in which blue light material itself cannot only be used as a light-emitting layer to prepare blue-ray organic light-emitting diodes (OLEDs), but also be used to dope other luminescent materials to prepare green and red devices. Therefore, blue light materials are always the focus in studies on organic light-emitting materials.[1]

However, research progress on blue light materials is still slower than that of green and red materials, because blue light materials that exhibit high yield and good stability[2] are still very challenging due to the wider gap (3.0 eV) required by stable and efficient blue light materials, making blue light emission more difficult.[3] Therefore, in order to produce efficient blue light devices, it is especially important to prepare major blue luminescent materials with superior properties. This focus has attracted the interest of researchers[4,5] and become the focus of the OLED industry and research.[6–14] Since Pope et al.[15] observed the faint blue light for the first time when hundreds of volts of direct current were added on anthracene single crystal, scientists have continuously studied to improve their brightness and yield, and

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Abbreviations used: CIE, Commission Internationale de L'Eclairage; CV, cyclic voltammetry; DPA, 9,10-diphenylanthracene; DSC, differential scanning calorimetry; EL, electroluminescence; HOMO, highest occupied molecular orbital; HTL, hole transporting layer; LUMO, lowest unoccupied molecular orbital; NMR, nuclear magnetic resonance; OLED, organic light-emitting diodes; PL, photoluminescence; RE, reference electrode; TGA, thermogravimetric analysis; UV, ultraviolet.

prepare blue luminescent materials with good performance and good results.[15–19] Anthracene intermolecular crystals are easy to form because of the flat structure of the anthracene molecule. However, the performance of blue light material anthracene and its derivatives is still much worse than that of red and green lights, which greatly reduce the practical value of anthracene in OLEDs.

Because of carbazole's strong absorption in the short wavelength range, its energy band gap of around 3.2 eV and its unique optical properties, carbazole and its derivatives as hole transport materials have been widely used for small molecule and polymer blue OLED.[20–23] However, the size of small molecule light materials from carbazole derivatives is small with poor thermal stability and the quantum yield of polymer materials is still low, thus their applications have been limited. In general, due to their chemical structure and energy level, most organic semiconductor unipolar materials tend to transmit a carrier (electron or hole) and have lower transmission capacity for another carrier. In the single layer devices made from unipolar materials, the electron concentration and hole concentration are unbalanced, resulting in decreased performance and reduced device life due to movement of the light-emitting layer to the electrode/organic layer interface.[24] While, as light-emitting layer, ambipolar transmission materials can also improve matching of electron and hole to obtain single layer devices with high brightness and high yields.[25] Therefore, more and more attention has been paid to ambipolar transmission materials with effectively balanced carriers in devices to improve their lighting yield and stability.[26]

To date, mobility of electrons and holes in all reported ambipolar materials with donor–acceptor structures have a difference of one to two orders of magnitude without a good balance.[27–30] Although abundant numbers and types of ambipolar transmission materials have been reported, there are very limited materials reported with excellent performance. Therefore, the development of high-performance ambipolar light-emitting materials has become the focus of studies and investigation.[31–34]

In this study, *p*-cyanophenyl with strong electronic absorbing capability was introduced into the nitrogen on carbazole position 9, followed by the introduction of a 9-anthracene group on the carbazoole positions of 3 and 6, to synthesize the conjugated carbazole–anthracene complicated structure compound 4-(3,6-bis (anthracene -9-ylethynyl)-9H-carbazol-9-yl)benzonitrile (BACB) with acetylene to connect tcarbazole and anthracene and the complicated structure compound 4-(3,6-di(anthracene- 9-yl)-9H-carbazol-9-yl)benzonitrile (DACB) with the introduced 9-anthryl to connect the carbazole and anthracene directly. These thermal and optical properties were studied to obtain ambipolar blue light-emitting materials with excellent performance.

2 | EXPERIMENTAL

2.1 | Materials and methods

Iodic acid (HIO_3), ≥99.5%, Shanghai Xinliang Chemical Reagent Co Ltd; triphenylphosphine (PPh_3), CP, Sinopharm Chemical Reagent Co Ltd; KI, >99.5%, Tianjin Damao Chemical Reagent Factory; triisopropyl borate (98%), J&K; 9-bromoanthracene (96%), carbazole (96%), n-butyllithium (1.6 M in toluene), 2-methyl−3-butyn-2-ol (98%), cesium carbonate (99.5%), 4-bromobenonitrile (98%), and tetrabutylammonium hexafluorophosphate were purchased from Acros Organic and used as received. Tetrakis(triphenylphosphine)palladium(0) (99.8%), CuI (98%), Alfa Aesar; 1,1´-bis(diphenylphosphino) ferrocene (DPPF, 99%), Strem Chemicals. anthracen-9-ylboronic acid[27] and 9-ethynylanthracene[28] were synthesized according to the literature procedures. Other reagents were all purified with standard methods if needed.

2.2 | Characterization

Melting points were measured on a SGW X-4 micro melting point apparatus. ^{1}H-NMR spectra were recorded on a Mercury-Plus 300 NMR spectrometer (Varian) in $CDCl_3$ with tetramethylsilane as an internal standard. Chemical shifts were reported in ppm relative to the internal standard tetramethylsilane. Elemental analyses were carried out on a Vario EL Elemental Analyzer (Elementar). EI-mass measurements were carried out on a DSQ EI-mass spectrometer (Thermo). UV-vis absorption spectra were recorded on a TU-1901 spectrophotometer. Emission spectra were recorded on a Perkin-Elmer LS 55 fluorescence spectrophotometer. Differential scanning calorimetry (DSC) measurements were carried out on a Netzsch DSC-204 differential scanning calorimeter (heating rate: 10°C/min; atmos.: N_2, 20.00 ml/min). Thermogravimetric analyses (TGA) were recorded on a Perkin-Elmer TGS-2 thermogravimeter (heating rate: 20°C/min; atmos.: N_2, 45.00 ml/min). Electrochemical experiments were performed on a PPS-268A potentiostat with a glassy carbon working electrode and a $Ag/AgNO_3$ (0.1 M in acetonitrile) reference electrode (scan rate: 100 mV sec^{-1}). Dichloromethane was used as the solvent, and *tetra*-*n*-butylammonium hexafluorophosphate (0.1 M) was used as the supporting electrolyte. The characteristics of voltage–current–luminance was recorded by Keithley 2400 SourceMeter (Keithley, USA) and corrected silicon photodiode, The electroluminescence spectra (EL) were recorded using a InstaspecIV CCD system (Oriel, USA).

2.3 | Preparation of blue luminescent bipolar materials

2.3.1 | Synthesis of compound 4-(9H-carbazol-9-yl) benzonitrile (CBN)[29]

In the glove box, DPPF (400 mg, 0.72 mmol), $Pd(PPh_3)_4$ (346.5 mg and 0.3 mmol) and newly distilled toluene (15 ml) after 30 min of oxygen removal with the flux of nitrogen 30 min were added into a 100 ml single-neck bottle. After 30 min of stirring, carbazole (2.004 g, 12 mmol), *p*-cyanobenzyl bromide (2.715 g, 15 mmol), Cs_2CO_3 (4.890 g, 15 mmol) and toluene (55 ml) were added, sealed, moved out from the glove box and reacted for 24 h in an oil bath at 100°C under nitrogen protection. After the reaction, the mixture was filtered and the filter residual was washed three times with ethyl acetate. Organic phases were combined together, washed twice with both distilled water and saturated salt water, and dried with sodium sulfate overnight. The solvent was removed with a rotary vapour. The solid was dissolved with a small number of tetrahydroflurans (THF) under heating, recrystallized with the dropwise addition of anhydrous methanol. After filtration, the crystal was washed three times with methanol to obtain a white crystal (2.95 g) with a yield of 92% and a melting point of 176–177°C. ^{1}H-NMR (300 MHz, $CDCl_3$): d(ppm) 8.14 (d,J = 7.8 Hz, 2H), 7.91 (d,J = 8.Hz, 2H), 7.74 (d,J = 8.7 Hz, 2H), 7.40–7.48 (m, 4H), 7.30–7.37 (m, 2H).

2.3.2 | Synthesis of compound 4-(3,6-diiodo-9H-carbazol-9-yl) benzonitrile (DICBN)

A mixture of CBN (2.95 g, 11 mmol), KI (2.74 g, 14.5 mmol), and HIO_3 (3.53 g, 16.5 mmol) in Acetic Acid (AcOH) (40 ml) was stirred at 115°C for 30 min. Then the mixture was filtered when it was warm. The obtained precipitates were washed with AcOH. The crude product was purified by recrystallization from THF/CH_3OH. Yellow solid, 4.51 g; yield 79.0%. mp. 249–250°C. ^{1}H-NMR: δ 8.39 (s, 2H), 7.92 (d, *J* = 8.4 Hz, 2H), 7.68 (t, *J* = 8.4 Hz, 4H), 7.18 (d, *J* = 8.4 Hz, 2H). Anal. calcd: $C_{19}H_{10}I_2N_2$, C, 43.88; H, 1.94; I, 48.80; N, 5.39; found: EI MS *m/z* 519.89; found: 520.

2.3.3 | Synthesis of compound BACB

To a two-necked 250 ml flask, 0.416 g (0.8 mmol) of DICBN, 0.45 g (2.23 mmol) of 9-ethynylanthracene, 12.5 mg of $Pd(PPh_3)_2Cl_2$, 15.3 mg of CuI, and 23.2 mg of triphenylphosphine were added. The flask was vacuumed and refilled with N_2. This process was repeated three times. A mixture of 60 ml freshly distilled toluene and 20 ml of freshly distilled Et_3N, degassed by bubbling N_2 under stirring for 30 min, was added to the flask. The resulting mixture was stirred for 1 h at room temperature, then overnight at 80°C. After the reaction, the mixture was cooled and filtered. The filtrate was washed with water, and saturated NaCl solution successively, dried with anhydrous Na_2SO_4, then filtered. The solvent in the filtrate was removed by rotary evaporation. The crude product was purified by recrystallization from THF/CH_3OH after column chromatography on silica gel with petroleum/THF (v/v = 4/1). Yellow solid 0.358 g; yield 67%. mp 328–329°C. ^{1}H-NMR: δ 8.76 (d, *J* = 8.7 Hz, 4H), 8.65 (s, 2H), 8.45 (s, 2H), 8.05 (d, *J* = 8.4 Hz, 4H), 8.00 (d, *J* = 8.1 Hz, 2H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 8.4 Hz, 2H), 7.61–7.69 (m, 4H), 7.50–7.58 (m, 6H). Anal. calcd: $C_{16}H_{21}IO$, C, 91.59; H, 4.22; N, 4.19; found: C, 91.71; H, 4.37; N, 3.98. EI MS *m/z* calcd.: 668.23; found: 668.

2.3.4 | Synthesis of compound DACB

To a 250 ml flask, 1.56 g (3 mmol) of DICBN, 2 g (9 mmol) of anthracen-9-ylboronic acid, 346.5 mg (0.3 mmol) of $Pd(PPh_3)_4$, and 157.2 mg (0.6 mmol) of triphenyl-phosphine were added. The flask was vacuated and refilled with N_2 for three times. A mixture of 90 ml freshly distilled toluene, 30 ml of freshly distilled methanol, and 15 ml of 2 M aqueous solution of K_2CO_3, degassed by bubbling N_2 under stirred for 30 min, was added to the flask via a syringe. The resulting mixture was stirred under N_2 for 48 h at 50°C. After reaction, the reaction mixture was filtered. The obtained precipitates were washed with water and toluene successively, then dissolved with THF, and filtered through a short column of silica gel to remove the catalyst. The crude product was purified by recrystallization from THF/CH_3OH. Yellow solid 1.37 g; yield 74%. mp 357–359°C. ^{1}H NMR: δ 8.50 (s, 2H), 8.16 (s, 2H), 7.98–8.08 (m, 8H), 7.70–7.77 (m, 6H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 4H), 7.32 (t, *J* = 7.5 Hz, 4H). Anal. calcd.: $C_{16}H_{21}IO$, C, 90.94; H, 4.55; N, 4.51; found: C, 90.79; H, 4.82; N, 4.27. EI MS *m/z* calcd.: 620.23; found: 620.

3 | RESULTS AND DISCUSSION

All compounds were stable at atmospheric temperature and pressure and were soluble in common organic solvents. Their spectroscopic data were in good agreement with the proposed structures in Scheme 1 and Scheme 2.

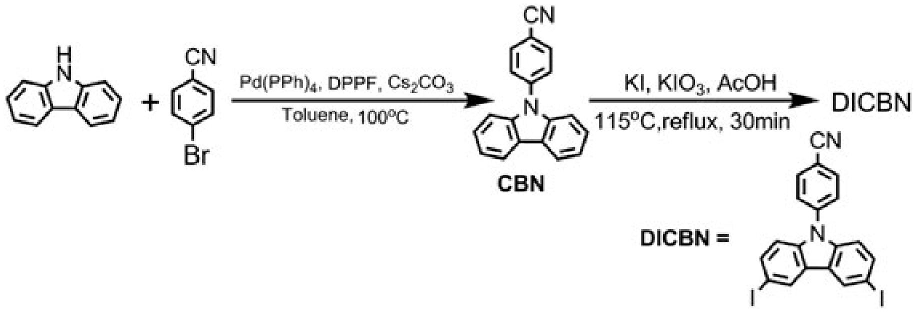
3.1 | Structural description

The synthetic routes of all compounds are shown in Scheme 1 and Scheme 2. The compounds were characterized by ^{1}H-NMR, ESI-MS, TGA, DSC and UV-vis analysis.

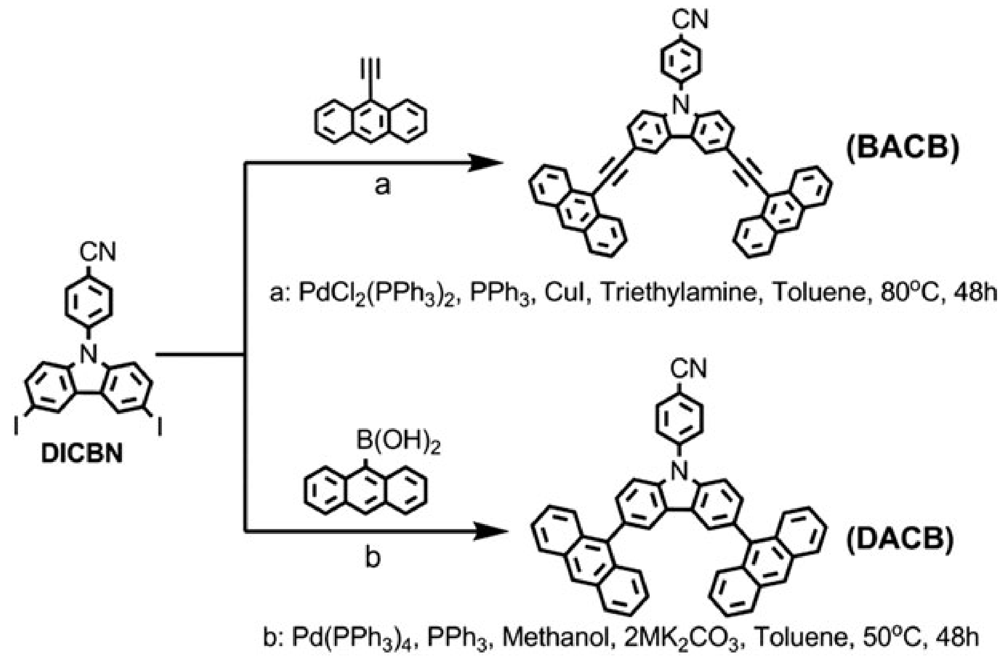
3.2 | Thermal properties

After the sample was dried for 12 h at 40°C under vacuum in advance, its thermogravimetry (TG) and DSC experiments were conducted in a N_2 atmosphere with a TG heating rate of 20°C/min, DSC heating rate of 10°C/min, TG test temperature range of 50–700°C and DSC test temperature range of 50–300°C. TG and DSC results for DACB and BACB are shown in Figures 1 and 2, respectively.

As shown in Figure 1, in N_2, DACB started to lose weight at around 410°C with 5% weight loss at 443°C and 10% weight loss at 461°C. BACB started to lose weight at around 260°C with 5% weight loss at 475°C and 10% of weight loss at 506°C. The weight loss of BACB at 220–320°C might be due to evaporation of solvent or volatile impurities in its crystal lattice. According to their thermogravimetric analysis, these two compounds both had high thermal stability and



SCHEME 1 Synthetic route of CBN and DICBN



SCHEME 2 Synthetic route of BACB and DACB

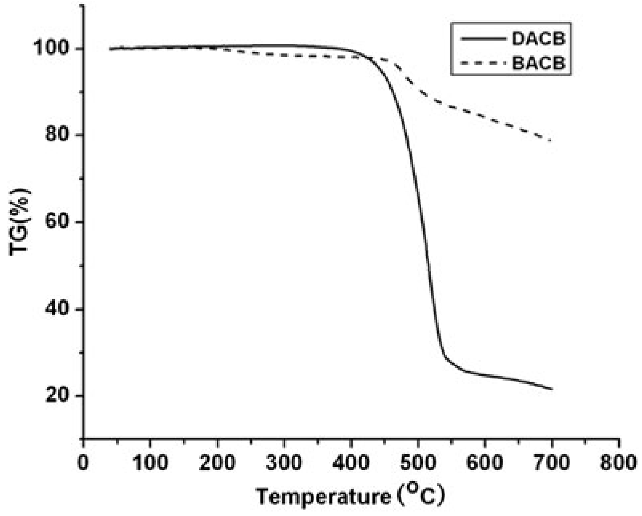


FIGURE 1 TG curves for DACB and BACB

the thermal decomposition temperature of DACB without an alkynyl group was higher than that of BACB with an alkynyl group, probably due to the instability of alkynyl compounds.

As shown in Figure 2, there were not significant heat changes for DACB and BACB at 50–300°C, indicating that they had good thermal stability. On melting point apparatus, their melting points was determined as 357–359°C and 328–330°C.

Therefore, both DACB and BACB had excellent thermal stabilities according to their TG and DSC results.

3.3 | Photophysical properties

At room temperature, with tetrahydrofuran as solvent, ultraviolet absorption spectra of DACB and BACB solution are shown in Figure 3.

According to the ultraviolet absorption spectra in THF solution, both compounds had four peaks within 320–450 nm at 387 nm, 368 nm, 349 nm and 334 nm for DACB, and at 434 nm, 409 nm,

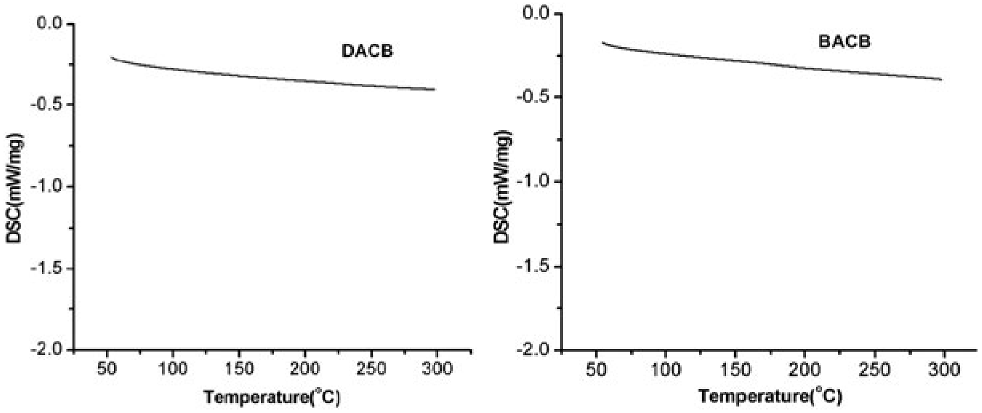


FIGURE 2 DSC curves for DACB and BACB

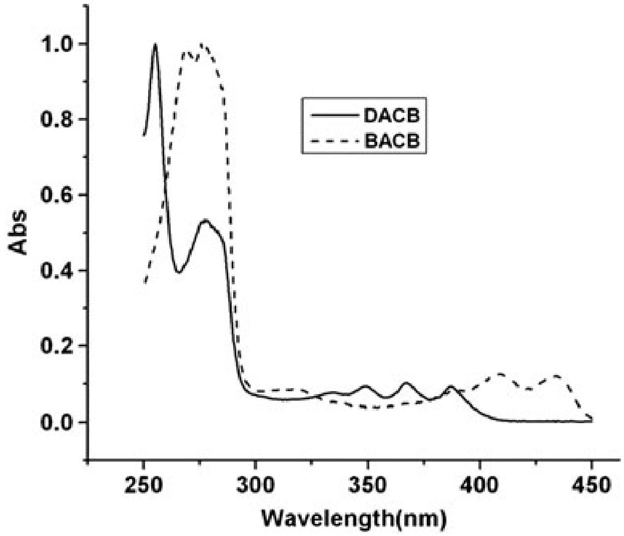


FIGURE 3 UV absorption spectra of DACB and BACB

388 nm and 367 nm for BACB, respectively. DACB had the characteristic absorption peaks of the anthracene group[30] while UV absorption peaks of BACB with an alkynyl group had a relative red shift.

Figure 4 shows the fluorescence spectra of DACB and BACB solution in THF at room temperature.

As shown in Figure 4, emission spectra of both DACB and BACB were single peaks with shoulders, in which the maximum emission wavelength (λ_{max}) of DACB was 415 nm while the λ_{max} of BACB was 479 nm. Similar to their corresponding UV absorption spectra, fluorescent emission peaks of BACB had a red shift of 64 nm compared with that of DACB because the introduction of alkynyl molecules resulted in an increase in the π-electron conjugated system. Meanwhile, 9,10-diphenylanthracene (DPA) was chosen as the standard ($\Phi_{f(cyclohexane)}$ = 0.9)[31] to determine fluorescence quantum yields of DACB and BACB and the results are listed in Table 1.

As listed in Table 1, these three compounds all had fluorescence quantum yields above the average, in which the fluorescence quantum yield of BACB was similar to that of the standard DPA. Through comparison, for compounds with similar structures, the fluorescence quantum yield of BACB with the introduction of an alkynyl group was increased significantly over that of DACB, indicating that the proper introduction of an alkynyl group could increase the π-electron conjugated system in the molecule and also increase the fluorescence quantum yield.

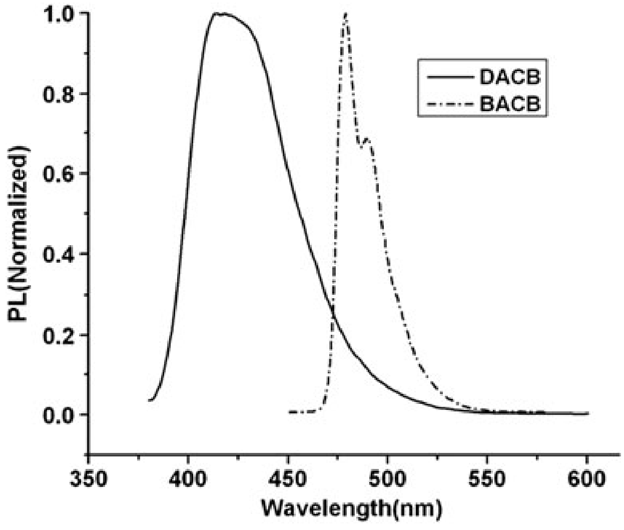


FIGURE 4 Fluorescence emission spectra of DACB and BACB

TABLE 1 Fluorescence quantum yields of DACB and BACB in THF

	λexmax (nm)	Adsorption (A)	Fluorescence strength (I)	Fluorescence quantum yield (Φ_f)
The standard	373	0.0355	9168	0.90
DACB	367	0.0349	6255	0.61
BACB	409	0.0237	6097	0.87

3.4 | Electrochemical properties

Cyclic voltammetry (CV) was done on a PS-268A electrochemical measuring instrument (Beijing Middle Corrosion Protection Engineering Technology Ltd) with a three-electrode system and a glassy carbon electrode as the working electrode, Ag/Ag+ (0.1 M acetonitrile solution) as the reference electrode (RE), platinum electrode as the auxiliary electrode (AE), a scanning rate of 20 mV/sec, dichloromethane as solvent, and tetrabutylammonium hexafluorophosphate (0.1 M) as the supporting electrolyte. After the analysis on electrochemical properties of the synthesized materials, their highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) energy levels could be calculated according to the following formula[32,33]:

$$E_{HOMO} = -4.71 - E_{pa\ (onset)},\ E_{LUMO} = Eg - |E_{HOMO}|,\ Eg = 1240/\lambda_{abs}$$

where Epa (onset) is the initial oxidation potential, E_{LUMO} is the LUMO energy level, Eg is the band between HOMO and LUMO, λabs is the intersection point of the tangential curve on the direction of UV-vis adsorption wave and the baseline. The results are shown in Figure 5.

After the starting oxidation potentials of DACB and BACB were obtained as shown in Figure 5, with the intersection point of tangential curve on the direction of UV-vis adsorption wave and the baseline, the above-mentioned formula could be used to calculate their HOMO and LUMO energy levels as well as the gaps between them, and the results are shown in Table 2.

As listed in Table 2, the energy barrier between the LUMO energy level (−2.37) of DACB and Mg:Ag cathode (−3.7 eV) was small and in favour of electronic injection, therefore, DACB was more suitable as a blue light material for electronic transmission, probably because the alkynyl group in BACB might enhance the conjugation and flatness of the molecule.

3.5 | Electroluminescent properties

Firstly, *N,N´*-di-[(1-naphthyl)-N,N´-diphenyl]-1,1´-biphenyl)-4,4´-diamine (NPB) and BACB were used as the hole transporting layer (HTL) and luminescent layer respectively, and the organic layers and the top cathode of Al/LiF were successively vacuum deposited onto an indium–tin oxide (ITO)-coated glass substrate to fabricate the electroluminescence (EL) device. The EL device structure is ITO/NPB

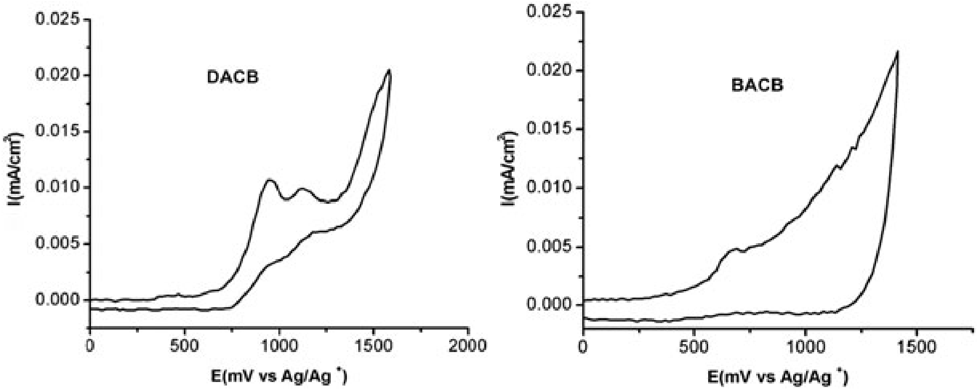


FIGURE 5 Cyclic voltammetry curves of DACB and BACB

TABLE 2 HOMO and LUMO energy levels of DACB and BACB

	$E_{pa(onset)}$ (eV)	Eg (eV)	E_{HOMO} (eV)	E_{LUMO} (eV)
DACB	0.73	3.07	−5.44	−2.37
BACB	0.51	2.76	−5.22	2.46

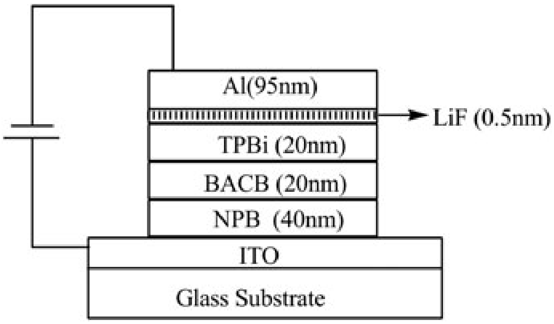


FIGURE 6 The structure of the EL device

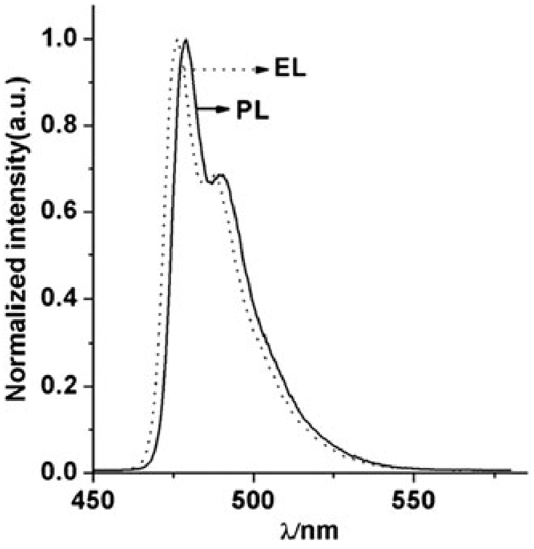


FIGURE 7 El spectrum and PL spectrum of the device

(40 nm)/BACB (20 nm)/TPBi (20 nm)/LiF (0.5 nm)/Al (95 nm) and is shown in Figure 6.

Secondly, the electroluminescence (EL) spectrum and photoluminescence (PL) spectrum of ITO/NPB (40 nm)/BACB (20 nm)/TPBi (20 nm)/LiF (0.5 nm)/Al (95 nm) device were researched and the results are shown in Figure 7.

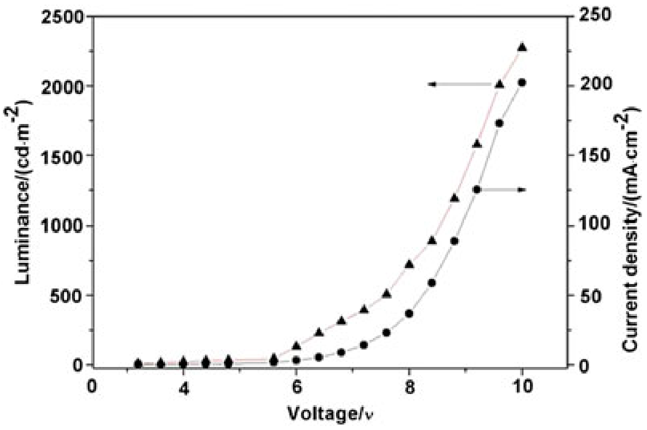


FIGURE 8 The luminance–voltage–current density curves of the device

As shown in Figure 7, the device emits dark blue light with λ_{max} at 476 nm accompanied by a weak shoulder peak at 487 nm at a voltage of 10 V. By contrast, the electroluminescent spectra (EL) of the device are similar to the photoluminescence spectra (PL), and the maximum wavelength (476 nm) of electroluminescence is only 3 nm less than the maximum wavelength (479 nm) of photoluminescence, indicating that the light from the device is mainly produced from the compound BACB.

Finally, the luminance–voltage–current density curves of the ITO/NPB/BACB/TPBi/LiF/Al device were recorded and displayed in Figure 8.

As shown in Figure 8, the turn-on voltage, which is defined as the voltage at which the luminance is 1 cd/m^2, was measured and found to be 5 V. At the same time, it was found that the luminance increased with increasing injection current as well as bias voltage. At the driven voltage of 10 V, the maximum luminance of 2300 cd/m^2, the current densities of 200 mA/cm^2 and the maximum luminance efficiency of 6.55 cd/A were obtained, respectively. The device emits dark blue light with Commission Internationale de L'Eclairage (CIE) coordinates of (0.157, 0.079).

4 | CONCLUSION

With carbazole as the raw material, DACB and BACB were synthesized through the Suzuki coupling and Sonogashira coupling reactions, with thermal decomposition temperatures at 410°C and 260°C and melting points at 357–359°C and 328–330°C, respectively, indicating that these two compounds had excellent thermal stability. Meanwhile, both DACB and BACB could emit strong blue light with quantum yields of 0.61 and 0.87, respectively. Their electrochemical performance showed that, through the introduction of the appropriate electron withdrawing groups and conjugated systems to modify the carbazole structure, the ability of the electronic transmission of carbazole could be improved to turn carbazole with typical hole transmission capability into an ambipolar blue light material. Finally, the electroluminescence (EL) device of ITO/NPB (40 nm)/BACB (20 nm)/TPBi (20 nm)/LiF (0.5 nm)/Al (95 nm) with BACB as the emitting layer was fabricated, and the EL device was shown to emit dark blue light.

ACKNOWLEDGEMENTS

Authors would like to thank Guangzhou City Education System Innovative Academic Team Foundation (Project No. 1201610020) and key project (Project No. ZSH201609) of Guangzhou Institute of Technology for financial support.

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How to cite this article: Xie P, Yuan N, Li S, Ouyang Y, Zhu Y, Liang H. Synthesis and properties of blue luminescent bipolar materials constructed with carbazole and anthracene units with 4-cyanophenyl substitute at the 9-position of the carbazole unit. *Luminescence*. 2018;1–7. https://doi.org/10.1002/bio.3452