Songklanakarin J. Sci. Technol.
47 (4), 301–306, Jul. – Aug. 2025

http://www.sjst.psu.ac.th

Original Article

Synthesis and properties of fluorescent blue light emitting materials based on 3,6 and 2,7-carbazole derivatives

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Received: 3 July 2024; Revised: 24 March 2025; Accepted: 4 June 2025

Abstract

The 3,6 and 2,7-carbazole derivatives found in the same molecule as fluorescent blue light emitting materials in organic light-emitting diodes were 3',6'-di-*tert*-butyl-9-dodecyl-7-phenyl-2,9'-bicarbazole (CCP) and 4-(3',6'-di-*tert*-butyl-9-dodecyl-[2,9'-bicarbazol]-7-yl)-*N*,*N*-diphenylaniline (CCT). The target molecules were synthesized based on Ullmann coupling and a Suzuki cross-coupling reactions and characterized using ^{1}H-NMR, ^{13}C-NMR, MALDI-TOF MS, UV-Vis, fluorescence spectroscopy, cyclic voltammetry, and thermogravimetric analysis. The absorption maximum of CCT (359 nm) showed more red-shift than the absorption maximum of CCP (339 nm) due to an increase in conjugation in the molecule. The target molecules displayed emission of blue light in solution with good quantum yields (63-76%) and showed good electrochemical properties. The *n*-dodecyl group substitution at the *N*-position of 2,7-carbazole core led to high solubility and thermal stability (up to 415 ^{o}C).

Keywords: 3,6-carbazole, 2,7-carbazole, fluorescent, blue light emitting materials, organic light-emitting diodes

1. Introduction

Organic light-emitting diodes (OLEDs) were introduced in 1987 (Tang & VanSlyke, 1987). The advantages of OLEDs include their simplicity and low cost of fabrication, full color flat display, and brightness (Khanasa *et al.*, 2013). The most important point in OLEDs evolution was the discovery of RGB three-color light-emitting materials (red, green, and blue) with good emission efficiency and stability (Grinevich & Novikov, 2013; Kotchapradist *et al.*, 2013). Many scientists have designed and synthesized fluorescent blue light emitting materials using carbazole (Haijuan, & Wenmu, 2015; Hongji, Jian & Jinlong, 2012; Lee *et al.*, 2008; Li *et al.*, 2006; Reig *et al.*, 2017; Zhang *et al.*, 2020; Zhou, Guo, & Shao, 2022) in OLEDs. The advantages of the carbazole moiety are good aromatic, chemical, and environmental stability (Data *et al.*, 2014). The 3,6, 2,7, 1,8, and *N* positions of carbazole can be replaced by other substitution groups to make new compounds (Chara, 2018; Huang *et al.*, 2012; Konidena & Thomas, 2017).

This work reports on the synthesis and properties of fluorescent blue light emitting materials based on 3,6 and 2,7-carbazole derivatives with different types of conjugation units (phenyl and triphenylamine moieties) (Kotchapradist *et al.*, 2013; Zeng *et al.*, 2002), namely 3',6'-di-*tert*-butyl-9-dodecyl-7-phenyl-2,9'-bicarbazole (CCP) and 4-(3',6'-di-*tert*-butyl-9-dodecyl-[2,9'-bicarbazol]-7-yl)-*N*,*N*-diphenylaniline (CCT), respectively (Li *et al.*, 2007; Ye *et al.*, 2013). In this design, the nature of the *N*-substitution of the 2,7-carbazole core was modified by the *N*-dodecyl chains for thermal stability and solubility of the molecule and the 3,6 position of carbazole was modified by di-*tert*-butyl groups to prevent this type of electrochemical coupling reaction (Prachumrak *et al.*, 2014).

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2. Materials and Methods

2.1 Materials and characterization

All chemicals were procured from commercial sources. Melting points were measured using a melting point apparatus with the open capillary method and are reported in degrees Celsius. All NMR spectra were recorded on a 600 MHz Bruker Spectrometer, using $CDCl_3$ as a solvent. MALDI-TOF MS mass analysis was run on a Bruker Mass Spectrometer, using HCCA as a matrix. UV-Vis spectra were recorded in solutions using a UV-Vis spectrometer. Fluorescence emission spectra were measured using a fluorescence spectrometer. Φ_{PL} was measured using an integrating sphere. The electrochemistry was performed using an Autolab Spectrophotometer. All measurements were taken at room temperature on samples dissolved in freshly distilled CH_2Cl_2, using 0.1 M n-Bu_4NPF_6 as the electrolyte. A glassy carbon working electrode, platinum wire counter electrode, and an Ag/AgCl/NaCl (Sat.) reference electrode were used. The solutions were degassed by bubbling with argon. CH_2Cl_2 was washed with concentrated H_2SO_4 and distilled from CaH_2. The Fc^+/Fc redox couple was used as a standard and Fc was purified based on recrystallization from C_2H_5OH and then dried under a high vacuum and stored over P_2O_5. TGA was performed on a Mettler Toledo thermogravimetric analyzer. Samples were scanned from 25 °C to 800 °C using a heating rate of 10 °C/min and a cooling rate of 70 °C/min with N_2 flushing.

2.2 General procedure for synthesis of compounds

2.2.1 Synthesis of 7-bromo-3',6'-di-*tert*-butyl--9 dodecyl-2,9'-bicarbazole (3)

To a mixture of CuI (0.03 g, 0.14 mmol), KO^tBu (0.08 g, 0.72 mmol), and 2,7-dibromo-9-dodecylcarbazole (1) (0.14 g, 0.29 mmol) in toluene (20 mL), 3,6-di-*tert*-butylcarbazole (2) (0.20 g, 0.72 mmol) and (±)-*trans*-1,2-diaminocyclohexane (0.02 g, 0.14 mmol) were added. The mixture was stirred at 110 °C under N_2 for 24 h. The solution was extracted with CH_2Cl_2, H_2O, and NaCl and dried with Na_2SO_4, filtered, and the solvent was evaporated to dryness. Purification was done using column chromatography over silica gel eluted with hexane to yield 3 (0.29 g, 70%) as a light-yellow viscous liquid; R_F 0.25; $C_{44}H_{55}BrN_2$; ^1H-NMR (600 MHz, $CDCl_3$) δ8.31 (2H, s), 8.26 (1H, d, J = 8.4 Hz), 8.03 (1H, d, J = 8.2 Hz), 7.66 (2H, d, J = 9.6 Hz), 7.57 (2H, d, J = 8.4 Hz), 7.52-7.49 (3H, m), 7.46 (1H, d, J = 7.8 Hz), 4.29 (3H, t, J = 6.6 Hz), 1.95-1.90 (2H, m), 1.59 (18H, s), 1.49-1.32 (18H, m), and 0.97 (3H, t, J = 6.6 Hz) ppm; ^{13}C-NMR (150 MHz, $CDCl_3$) δ142.90, 141.97, 141.45, 139.76, 136.32, 123.71, 123.66, 123.46, 122.56, 121.66, 121.57, 121.40, 121.30, 119.62, 118.48, 116.38, 112.02, 109.42, 109.35, 107.45, 43.51, 34.84, 32.15, 31.98, 29.72, 29.68, 29.66, 29.63, 29.51, 29.41, 29.07, 27.48, 22.77, and 14.21 ppm; MALDI-TOF MS m/z = 690.5840 $[M]^{+\cdot}$, 690.3549 calculated for $C_{44}H_{55}BrN_2$.

2.2.2 Synthesis of 3',6'-di-*tert*-butyl-9-dodecyl-7-phenyl-2,9'-bicarbazole (CCP)

A mixture of 3 (0.30 g, 0.44 mmol), phenylboronic acid (4a) (0.06 g, 0.47 mmol), $Pd(PPh_3)_4$ (0.01 g, 0.01 mmol), and 2M Na_2CO_3 (1.02 g, 9.63 mmol) in THF (20 ml) was degassed with N_2 for 5 min. The mixture was stirred at reflux under N_2 for 24 h. The solution was extracted with CH_2Cl_2, H_2O, and NaCl and dried with Na_2SO_4, filtered, and the solvent was evaporated to dryness. Purification was performed using column chromatography over silica gel eluted with a mixture of CH_2Cl_2 and hexane (1:4) to form CCP (0.23 g, 75%) as a white solid; R_F 0.59; $C_{51}H_{60}N_2O$; m.p. 80-82°C; ^1H-NMR (600 MHz, $CDCl_3$) δ8.28 (1H, d, J = 7.8 Hz), 8.23-8.21 (3H, m), 7.78 (2H, d, J = 7.2 Hz), 7.65 (1H, s), 7.60 (1H, s), 7.57-7.50 (5H, m), 7.46-7.41 (4H, m), 4.36 (2H, t, J = 6.6 Hz), 1.95-1.90 (2H, m), 1.52 (18H, s), 1.45-1.23 (18H, m), and 0.88 (3H, t, J = 7.2 Hz) ppm; ^{13}C-NMR (150 MHz, $CDCl_3$) δ142.71, 142.18, 141.74, 141.67.139.78, 139.41, 128.83, 127.65, 127.17, 123.58, 123.30, 121.88, 121.59, 121.28, 120.61, 119.08, 118.05, 116.25, 109.32, 107.42, 107.26, 43.35, 34.76, 32.06, 31.88, 29.63, 29.59, 29.57, 29.48, 29.31, 29.11, 27.49, 22.66, and 14.09 ppm; MALDI-TOF MS m/z = 688.5699 $[M]^{+\cdot}$, 688.4756 calculated for $C_{50}H_{60}N_2$.

2.2.3 Synthesis of 4-(3',6'-di-*tert*-butyl-9-dodecyl-[2,9'-bicarbazol]-7-yl)-*N,N*-diphenylaniline (CCT)

The synthesis of CCT from *3* (0.32 g, 0.46 mmol) and (diphenylamino)phenyl)boronic acid (4b) (0.14 g, 0.49 mmol), was performed similar to that of CCP, producing CCT (0.21 g, 55%) as a white solid; R_F 0.48; $C_{62}H_{69}N_3$; m.p. 108-110°C; ^1H-NMR (600 MHz, $CDCl_3$) δ8.27 (1H, d, J = 7.2 Hz), 8.21-8.19 (3H, m), 7.67-7.42 (10H, m), 7.33-7.20 (10H, m), 7.09-7.07 (2H, m), 4.35 (2H, t, J = 7.2 Hz), 1.95-1.90 (2H, m), 1.52 (18H, s), 1.46-1.23 (18H, m), 0.88 (3H, t, J = 7.2 Hz); ^{13}C-NMR (150 MHz, $CDCl_3$) δ147.74, 147.15, 142.69, 141.74, 141.71, 139.79, 138.87, 136.09, 135.70, 129.31, 128.23, 124.43, 124.07, 123.57, 123.29, 122.96, 121.66, 121.55, 121.18, 120.60, 118.72, 118.03, 116.25, 109.33, 107.24, 106.80, 43.34, 34.76, 32.06, 31.87, 29.63, 29.58, 29.49, 20.30, 29.13, 27.51, 22.66 and 14.09 ppm; MALDI-TOF MS m/z = 855.9538 $[M]^{+\cdot}$, 855.5491 calculated for $C_{62}H_{69}N_3$.

3. Results and Discussion

3.1 Synthesis

The synthesis of the target compounds is outlined in Figure 1. The key intermediate 3 prepared from Ullmann coupling of 1 with 2 catalyzed by CuI, KO^tBu, and (±)-*trans*-1,2-diaminocyclohexane in toluene gave 3, which was first Suzuki cross-coupled with 4a catalyzed by $Pd(PPh_3)_4$ and 2M Na_2CO_3 in THF to give the target CCP with a good yield of 75% (Sudyoadsuk *et al.*, 2014). Further condensation of 3 with 4b under the same conditions gave the target CCT with a

55% yield. The two products were fully characterized using ^{1}H-NMR, ^{13}C-NMR, and MALDI-TOF MS, with the results matching well with their expected structures (Konidena *et al.*, 2015; Therdkatanyuphong *et al.*, 2020). All compounds showed high solubility in most organic solvents, due to the presence of *n*-dodecyl substituent in the molecule.

3.2 Optical properties

The optical properties of CCP and CCT were investigated in CH_2Cl_2 solution. The results are presented Figure 2 and summarized in Table 1. Their UV-Vis spectra showed two absorption bands, which were assigned in terms of the absorption band at 298 nm for CCP and 299 nm for CCT corresponding to the π-π* local electron transition of the carbazole ring, and the absorption band at longer wavelengths (339 nm for CCP and 359 nm for CCT) attributed to the π-π* electron transition of the phenyl-carbazole and triphenylamine-carbazole, respectively (Kotchapradist *et al.*, 2013). Fluorescent blue compounds with featureless PL spectra measured in dilute CH_2Cl_2 solutions exhibited structureless emissions that peaked at 392 nm for CCP and 422 nm for CCT (Therdkatanyuphong *et al.*, 2020). The spectrum of CCT showed more red-shift than the spectrum of CCP due to an increase in conjugation in the molecule (Kotchapradist *et al.*, 2013; Thongkasee *et al.*, 2014). CCT was a deeper blue than CCP according to the CIE coordinates of CCT (0.16, 0.05) and CCP (0.18, 0.07). Utilizing them as emitter, doped OLED devices of CCP and CCT displayed blue emission. The solution PL quantum yields (Φ_{PL}) of CCP and CCT were 63% and 76%, respectively.

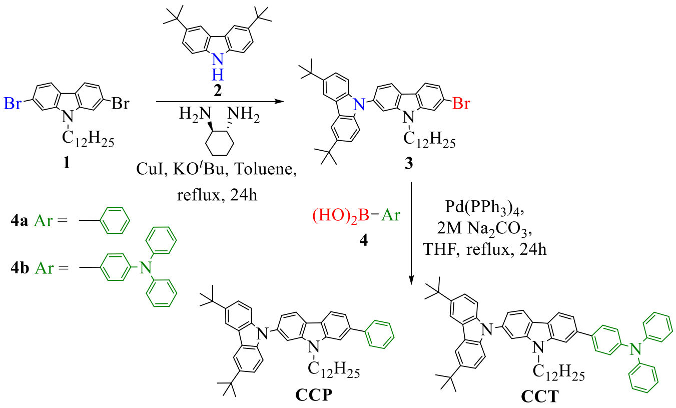
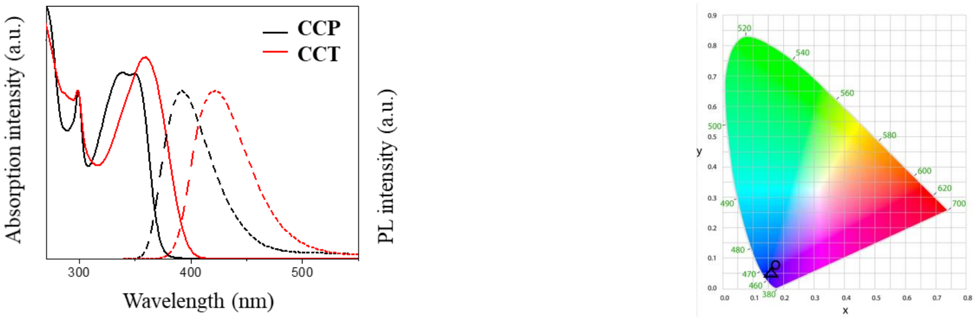


Figure 1. Synthesis of CCP and CCT



(a)

(b)

Figure 2. (a) UV–vis absorption and PL spectra in CH_2Cl_2 solution, and (b) CIE chromaticity coordinate diagrams of CCP (○) and CCT (△)

Table 1. Physical data for CCP and CCT

Com.	$\lambda_{abs}{}^{a}$ (nm)	$\lambda_{onset}{}^{a}$ (nm)	$\lambda_{em}{}^{a}$ (nm)	Conc.^{a} (M)	$E_g{}^{b}$ (eV)	$E^{ox}{}_{onset}{}^{c}$ (eV)	HOMO^{d} (eV)	LUMO^{d} (eV)	$T_{5d}{}^{e}$ (°C)
CCP	299, 339	370	392	6.39×10^{-5}	3.35	1.01	-5.45	-2.10	415
CCT	298, 359	395	422	2.99×10^{-5}	3.14	0.74	-5.18	-2.04	466

[a] Measured in CH_2Cl_2
[b] Calculated from the absorption edge, $E_g = 1240$ per λ_{onset}
[c] Obtained from CV in CH_2Cl_2/n-Bu_4NPF_6 (0.1 M) at a scan rate of 50 mV s^{-1}
[d] Estimated from HOMO = $-(4.44 + E^{ox}{}_{onset})$; LUMO = HOMO + E_g
[e] Obtained from TGA measured at 10 °C min^{-1} under N_2

3.3 Electrochemical properties

The electrochemical properties of CCP and CCT were investigated using cyclic voltammetry (CV) in CH_2Cl_2 (Figure 3) (Kotchapadist *et al.*, 2015). The CV curve of CCP exhibited two quasi-reversible oxidation processes, with the first and second oxidation peaks ($E_{1/2}^{ox}$ = 1.09 and 1.49 V, respectively) attributed to the oxidation of two carbazole moieties to form a radical cation. Additionally, its emphasized CV scans indicated unchanged CV curves, implying that no electrochemical oxidative coupling reaction occurred, resulting in no electro-polymerization on the peripheral carbazole at the 3,6 positions, due to the presence of di-*tert*-butyl groups that prevented this type of electrochemical coupling reaction (Figure 4) (Kotchapradist *et al.*, 2013; Sudyoadsuk *et al.*, 2014; Therdkatanyuphong *et al.*, 2020). On the other hand, the CV curve of CCT indicated three quasi-reversible oxidation processes. The first oxidation peak ($E_{1/2}^{ox}$ = 0.82 V) was attributed to the removal of electrons from the peripheral triphenylamine group to form the triphenylamine radical cation (TPA$^+$) with electrochemical stability. Its multiple CV scans showed no electrochemical oxidative coupling reaction (Figure 5). A proposed oxidation reaction of the CCT compound is outlined in Figure 6 (Kotchapadist *et al.*, 2015). In addition, the second and third oxidation peaks ($E_{1/2}^{ox}$ = 1.01 and 1.49 V, respectively) were assigned to the oxidation of two carbazole moieties to form a radical cation (Therdkatanyuphong *et al.*, 2020).

The HOMO and LUMO energy levels of CCP and CCT were enumerated from the oxidation onset potentials (E^{ox}_{onset}) and energy gaps (E_g) and the results are summarized in Table 1. The HOMO levels of these materials were nearly identical (-5.18–5.45 eV) and lower than the work functions of the ITO (-4.80 eV) electrodes, hence favoring the hole injection and transport. Their LUMO levels ranged from -2.04 eV to -2.10 eV (Khanasa *et al.*, 2013; Khunchalee *et al.*, 2012).

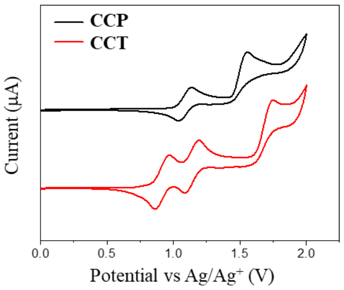


Figure 3. Cyclic voltammograms of CCP and CCT in dry CH_2Cl_2 with scan rate of 0.05 V/s and 0.1 M *n*-Bu_4NPF_6 as electrolyte

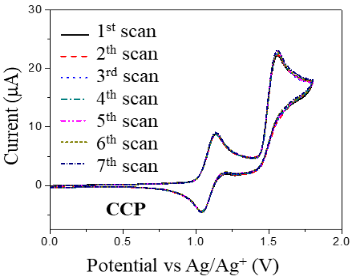


Figure 4. Cyclic voltammograms with multiple scans of CCP in dry CH_2Cl_2 with scan rate of 0.05 V/s and 0.1 M *n*-Bu_4NPF_6 as electrolyte

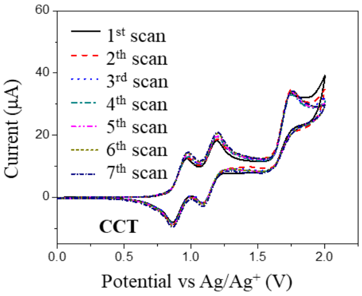


Figure 5. Cyclic voltammograms with multiple scans of CCT in dry CH_2Cl_2 with scan rate of 0.05 V/s and 0.1 M *n*-Bu_4NPF_6 as electrolyte

3.4 Thermal properties

The thermal stability of the two emitters was analyzed using thermogravimetric analysis (TGA) under nitrogen flushing (Figure 7 and Table 1) (Konidena *et al.*, 2017). Both CCP and CCT showed excellent thermal stability with decomposition temperatures at 5% weight loss (T_{5d}) of 415 and 466 °C, respectively (Kumsampao *et al.*, 2020; Therdkatanyuphong *et al.*, 2020). The thermal stability of organic materials is crucial for the lifetime of OLED displays (Huang *et al.*, 2013; Najare *et al.*, 2021).

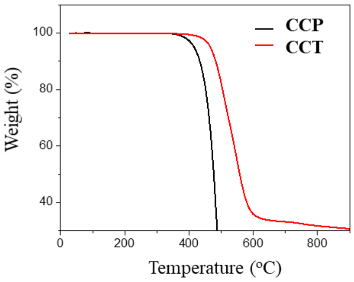


Figure 7. TGA traces measured at heating rate of 10 °C min^{-1} under N_2 flushing

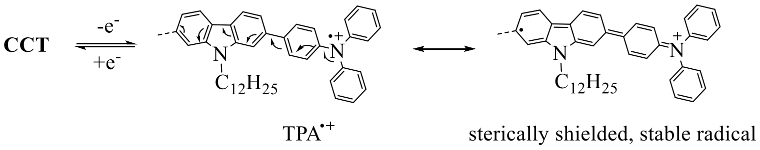


Figure 6. *π*-Electron delocalization in triphenylamine radical cation and its oxidation reaction

4. Conclusions

This research demonstrated the synthesis of 3,6 and 2,7-carbazole derivatives as fluorescent blue light emitting materials. The absorption maximum of CCT at 359 nm showed more red-shift than the absorption maximum of CCP at 339 nm, due to an increase in conjugation in the molecule. The target molecules displayed emission of blue light in solution with good quantum yields (63-76%) and good electrochemical properties. The *n*-dodecyl group substituted at the *N*-position of the 2,7-carbazole core led to high solubility and thermal stability (up to 415°C).

Acknowledgements

The Thailand Research Fund (MRG6080117), National Research Council of Thailand and Rajamangala University of Technology Isan Sakonnakhon Campus provided financial support.

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