



# Design of bicarbazole type host materials for long-term stability in blue phosphorescent organic light-emitting diodes



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## ABSTRACT

Two bicarbazole type host materials, 9-(dibenzo [*b,d*]thiophen-4-yl)-9'-phenyl-9*H*,9'*H*-3,3'-bicarbazole (DBTBCz) and 9,9'-bis(dibenzo [*b,d*]thiophen-4-yl)-9*H*,9'*H*-3,3'-bicarbazole (DDBTBCz), were developed as lifetime enhancing host materials for blue phosphorescent organic light-emitting diodes (PhOLEDs). The DBTBCz and DDBTBCz host materials were prepared by substituting one or two dibenzothiophene units to a 3,3'-bicarbazole backbone structure for the purpose of improving thermal stability and rigidity of the host materials for stable operational lifetime. Device characterization of the host materials revealed that the dibenzothiophene modification via 4- position is better than that via 2- position for improved lifetime of blue PhOLEDs.

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## 1. Introduction

Lifetime is one of the most important device performances of organic light-emitting diodes (OLEDs) in addition to quantum efficiency because short lifetime device cannot be applied in practical applications [1–5]. In particular, the lifetime of the blue OLEDs is relatively short compared with that of red and green OLEDs, which encouraged researchers to develop stable blue OLEDs [6]. Through extensive studies and effort, the lifetime of blue fluorescent OLEDs was significantly extended although it is still inferior to that of red and green fluorescent OLEDs [7]. However, the blue fluorescent OLEDs suffer from low quantum efficiency (QE) limited by spin statistics defining singlet and triplet exciton formation, which incited the development of high efficiency blue phosphorescent OLEDs (PhOLEDs) with 100% internal quantum efficiency (QE) theoretically [8].

The theoretical maximum QE of the blue PhOLEDs has already been reached through molecular engineering of hosts, phosphorescent emitters and charge transport materials. Device engineering to confine all excitons and carriers inside an emitting layer also aided the full utilization of singlet and triplet excitons for blue phosphorescent emission [9,10]. However, the lifetime of blue PhOLEDs is still very short in spite of the great advances made in

terms of QE of the blue PhOLEDs [11]. There are many reasons for the poor lifetime of blue PhOLEDs and lack of stable high triplet energy materials is one of critical problems [12–14]. Although many high triplet energy host and charge transport materials were reported, most materials could not work stably due to instability of the high triplet energy organic materials [15,17,18,22–24]. Only several host and charge transport materials were developed as the stable organic materials to obtain improved lifetime. For instance, a CN modified host material, 9-(3''-(carbazol-9-yl)-[1,1',3',1''-terphenyl]-3-yl)-carbazole-3-carbonitrile, was reported as a lifetime boosting host material by strengthened chemical bond [25], and thiophene-carbazole derivatives were also lifetime-extending host materials [20,26]. Therefore, the development of the host materials to fulfil the stability requirement of the blue PhOLEDs is essential to prolong the lifetime of the blue PhOLEDs.

Herein, we describe the synthesis of bicarbazole backbone structure based host materials possessing 4- position modified dibenzothiophene as substituent of the backbone structure. The bicarbazole backbone structure was either substituted with one dibenzothiophene unit or two dibenzothiophene units to yield 9-(dibenzo [*b,d*]thiophen-4-yl)-9'-phenyl-9*H*,9'*H*-3,3'-bicarbazole (DBTBCz) and 9,9'-bis(dibenzo [*b,d*]thiophen-4-yl)-9*H*,9'*H*-3,3'-bicarbazole (DDBTBCz) as the host materials. The two host materials with the dibenzothiophene unit linked via 4- position to the bicarbazole backbone structure outperformed the host material with the same dibenzothiophene unit linked via 2- position to the same backbone structure as the host of blue emitting tris [1-(2,4-

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diisopropylidibenzo [*b,d*]furan-3-yl)-2-phenylimidazole] (Ir (dbi)<sub>3</sub>) triplet emitter.

## 2. Experimental

### 2.1. General information

All chemicals and reagents were used without further purification. Copper iodide and (±)-trans-1,2-diaminocyclohexane purchased from Sigma Aldrich Co. were used without purification. 3-Bromo-9*H*-carbazole, (9-phenyl-9*H*-carbazol-3-yl)boronic acid, and tetrakis (triphenylphosphine) palladium (0) from P&H Tech Co. were used as received. 4-Iododibenzo [*b,d*]thiophene and 9*H*,9'*H*-3,3'-bicarbazole were purchased from INCO Co.. Potassium phosphate tribasic, potassium carbonate, dimethylformamide, tetrahydrofuran, dichloromethane and magnesium sulfate were products of Duksan Sci. Co.. 9-Phenyl-9*H*,9'*H*-3,3'-bicarbazole was synthesized according to the literature [21].

### 2.2. Synthesis

#### 2.2.1. 9-(dibenzo[*b,d*]thiophen-4-yl)-9'-phenyl-9*H*,9'*H*-3,3'-bicarbazole (DBTBCz)

9*H*,9'*H*-3,3'-bicarbazole (1.50 g, 3.67 mmol), 4-iododibenzo [*b,d*]thiophene (1.48 g, 4.77 mmol), copper iodide (0.21 g, 1.10 mmol), potassium phosphate (2.34 g, 11.02 mmol) and (±)-trans-1,2-diaminocyclohexane (0.13 ml, 1.10 mmol) in 1,4-dioxane (50 ml) were refluxed under nitrogen for 18 h and cooled to room temperature. After filtering the solution, the filtrate was dried and extracted with dichloromethane and distilled water. The organic layer was dehydrated with magnesium sulfate and then dried under vacuum condition. After purification, a white solid was collected by column chromatography (dichloromethane: hexane (1: 1)).

Yield 76%, 1.00 g. <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ 8.75 (s, 1H), 8.70 (s, 1H), 8.63 (d, 1H, *J* = 9.0Hz), 8.48 (d, 2H, *J* = 7.5Hz), 8.44 (d, 2H, *J* = 8.0Hz), 8.34 (d, 2H, *J* = 7.5Hz), 7.94 (d, 2H, *J* = 7.5Hz), 7.93–7.80 (m, 4H), 7.72–7.66 (m, 4H), 7.58–7.32 (m, 9H), 7.20 (d, 1H, *J* = 8.5Hz), 7.13 (d, 1H, *J* = 8.0Hz). <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>): δ 140.6, 140.2, 139.3, 138.8, 138.2, 137.6, 137.0, 136.9, 135.1, 133.6, 133.3, 131.4, 130.2, 127.7, 127.6, 126.8, 126.6, 126.4, 126.3, 125.6, 125.5, 125.1, 123.6, 123.5, 123.2, 123.1, 123.0, 122.6, 122.3, 121.0, 120.8, 120.4, 120.1, 118.8, 118.6, 110.5, 110.2, 110.0, 109.7. LC/MS (*m/z*): found, 590.7 ([*M* + *H*]<sup>+</sup>); Calcd. for C<sub>42</sub>H<sub>26</sub>N<sub>2</sub>S, 590.7.

#### 2.2.2. 9,9'-Bis(dibenzo[*b,d*]thiophen-4-yl)-9*H*,9'*H*-3,3'-bicarbazole (DDBTBCz)

9,9'-Bis(dibenzo [*b,d*]thiophen-4-yl)-9*H*,9'*H*-3,3'-bicarbazole was synthesized in concordance with the synthetic procedure of DBTBCz except for use of 9*H*,9'*H*-3,3'-bicarbazole instead of 9-phenyl-9*H*,9'*H*-3,3'-bicarbazole.

Yield 48%, 1.50 g. <sup>1</sup>H NMR (500 MHz, DMSO-*d*<sub>6</sub>): δ 8.74 (s, 1H), 8.60 (d, 1H, *J* = 7.5Hz) 8.49 (d, 1H, *J* = 8.0Hz), 8.42 (d, 1H, *J* = 7.5Hz), 7.90 (d, 1H, *J* = 8.0Hz), 7.80–7.75 (m, 3H), 7.56–7.49 (m, 2H), 7.39 (t, 1H, *J* = 15.0Hz), 7.33 (t, 1H, *J* = 14.5Hz), 7.16 (d, 1H, *J* = 8.5Hz), 7.11 (d, 1H, *J* = 8.0Hz). <sup>13</sup>C NMR (125 MHz, DMSO-*d*<sub>6</sub>): δ 140.6, 138.8, 138.2, 137.6, 137.0, 135.1, 133.6, 131.4, 127.7, 126.7, 126.4, 126.3, 125.5, 125.1, 123.6, 123.1, 122.5, 122.3, 121.0, 120.3, 118.9, 110.5, 110.1. LC/MS (*m/z*): found, 696.9 ([*M* + *H*]<sup>+</sup>); Calcd. for C<sub>48</sub>H<sub>28</sub>N<sub>2</sub>S<sub>2</sub>, 696.9.

### 2.3. Device fabrication and measurements

Device fabrication process, device structure and device performance measurement of the blue PHOLEDs with the DBTBCz and DDBTBCz hosts were the same as those described in other work.<sup>20</sup>

Lifetime devices had the following stack structure.

ITO/DNTPD (60 nm)/BPBPA (30 nm)/host:Ir (dbi)<sub>3</sub> (25 nm, 5–15% doping)/LG201 (35 nm)/LiF (1 nm)/Al (200 nm).

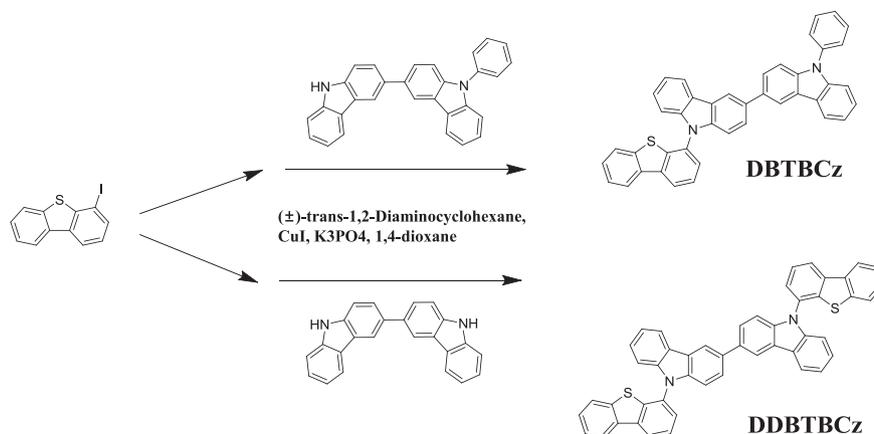
ITO represents indium tin oxide, DNTPD is *N,N'*-diphenyl-*N,N'*-bis-[4-(phenyl-*m*-tolyl-amino)-phenyl]-biphenyl-4,4'-diamine, and BPBPA is *N,N,N',N'*-tetra [(1,1'-biphenyl)-4-yl]-(1,1'-biphenyl)-4,4'-diamine. Lifetime data of the blue PHOLEDs were gathered at an interval of 0.07 h at a constant current mode.

## 3. Results and discussion

Several bicarbazole backbone derived host materials have been known as the host materials of blue PHOLEDs [19,20] and the merits of the bicarbazole backbone structure are rigidity and adjusted highest occupied molecular orbital (HOMO) level for good hole injection. The adjusted HOMO level for hole injection from an aromatic amine type hole transport material or carbazole type hole transport material may prevent charge accumulation at the interface between hole transport layer and emitting layer. The rigidity of the backbone structure may allow the host material to have thermal stability and amorphous character for suppressed crystallization. Moreover, the two carbazole units in the backbone structure are linked by stable sp<sup>2</sup> C–C bond which can be stable under long-term electrical operation. These benefits of the bicarbazole backbone based host materials can be strengthened by modifying the bicarbazole moiety with a dibenzothiophene moiety as reported in our previous work [20]. The bicarbazole type host material modified with 2- position activated dibenzothiophene (BCzDBT) was better than other bicarbazole host materials substituted with dibenzofuran or biphenyl. Although it was found that the 2- position activated dibenzothiophene modification worked effectively, the lifetime was rather short. In order to resolve the shortcoming of the 2- position modification of dibenzothiophene, 4- position activated dibenzothiophene was adopted to construct the bicarbazole type host materials. Main reason of designing the hosts using 4- position substituted dibenzothiophene was to stabilize the molecule by added rigidity as reported in other works [26] and also to improve hole transport properties to properly manage the carrier recombination zone in the emitting layer.

Synthesis procedure of DDBTBCz followed the synthetic method of other bicarbazole type compounds as depicted in Scheme 1. The only difference was that 9-phenyl-9*H*,9'*H*-3,3'-bicarbazole was replaced with 9*H*,9'*H*-3,3'-bicarbazole. DBTBCz was produced from 9*H*,9'*H*-3,3'-bicarbazole intermediate with only one active carbazole for substitution.

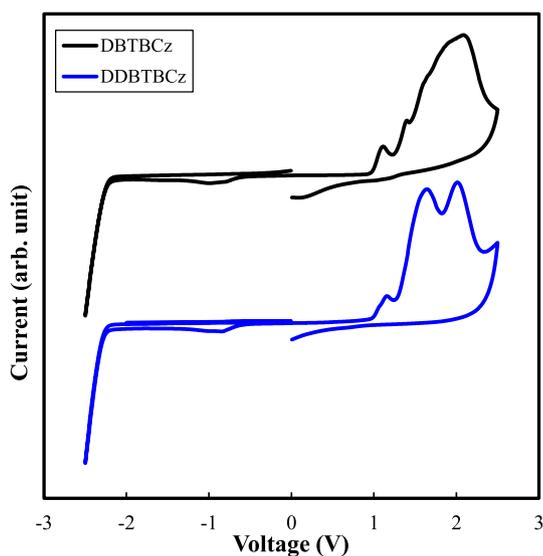
The synthesized compounds were electrochemically and photophysically characterized after sublimation to measure the HOMO, lowest unoccupied molecular orbital (LUMO), singlet energy, and triplet energy. Electrochemical characterization of DBTBCz and DDBTBCz by cyclic voltammetry (CV) estimated the HOMO and LUMO from oxidation and reduction onset of the voltage scan in Fig. 1. The HOMO of the two host materials was –5.85 eV, and the LUMO values of DBTBCz and DDBTBCz were –2.65 eV and –2.60 eV, respectively, by calibrating the oxidation and reduction onset voltages using Ferrocene as the standard material. In order to understand the electrochemical characterization data, the HOMO and LUMO calculation results were obtained after optimizing the geometry of the host material using Gaussian 09 program providing B3LYP 6-31G\* basis set. The HOMO calculation results of DBTBCz and DDBTBCz in Fig. 2 were of little difference and the HOMO was evenly delocalized over the bicarbazole backbone structure. The LUMO calculation results were also similar in the two host materials because the LUMO was localized on the dibenzothiophene unit. These similar orbital calculation results in the two compounds



**Scheme 1.** Synthetic scheme of DBTBCz and DDBTBCz.

can explain the similar HOMO and LUMO level of DBTBCz and DDBTBCz.

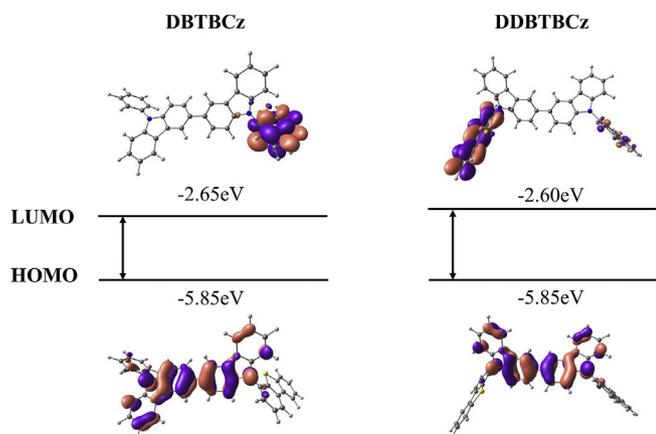
Photophysical characterization results of DBTBCz and DDBTBCz are presented in Fig. 3. Singlet energy and triplet energy were



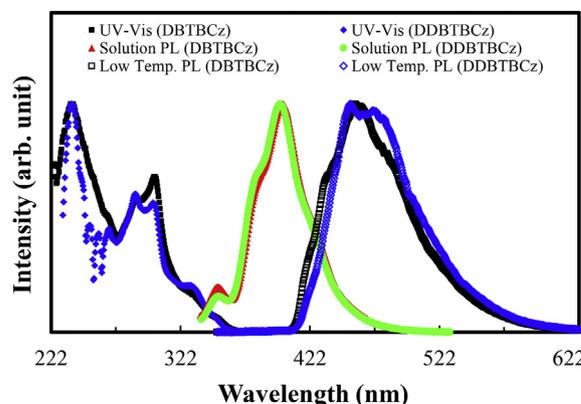
**Fig. 1.** CV curves of DBTBCz and DDBTBCz.

calculated from the photophysical measurement results by fluorescence spectrometer. The singlet energy from the solution photoluminescence (PL) peak position was 3.09 eV in the two compounds and the triplet energies from the first phosphorescent emission peak of low temperature PL were 2.92 eV and 2.94 eV in the DBTBCz and DDBTBCz, respectively. The introduction of the dibenzothiophene moiety through 4- position had little influence on the singlet and triplet energy of the host materials judging from the similar singlet and triplet energy of DBTBCz and DDBTBCz. Comparing the PL emission spectra of the two host materials with ultraviolet–visible (UV–vis) absorption spectrum of Ir (dbi)<sub>3</sub> [16], it can be projected that they may be good energy transferring host material of Ir (dbi)<sub>3</sub>. High singlet energy (>3.0 eV) and triplet energy (>2.9 eV) of DBTBCz and DDBTBCz may activate both Forster and Dexter energy transfer processes between the host materials and Ir (dbi)<sub>3</sub>, and can lead to full harvesting of singlet and triplet excitons in the phosphorescent emission process of Ir (dbi)<sub>3</sub>. UV–vis absorption spectra were also quite similar in the DBTBCz and DDBTBCz.

Electrochemical and photophysical measurements of DBTBCz and DDBTBCz substantiated that the two host materials are electrochemically and photophysically similar. However, the two hosts were dissimilar in terms of thermal properties represented by glass transition temperature ( $T_g$ ) and thermal decomposition temperature ( $T_d$ ). The  $T_g$  were extracted from second heating scan data of differential scanning calorimeter (DSC) under nitrogen in Fig. 4 and the  $T_d$  was estimated from heating scan data of thermogravimetric analyser (TGA) under nitrogen in Fig. 5. The 4- position



**Fig. 2.** HOMO and LUMO distribution and energy levels of DBTBCz and DDBTBCz.



**Fig. 3.** UV–Vis, solution PL and low temperature PL spectra of DBTBCz and DDBTBCz.

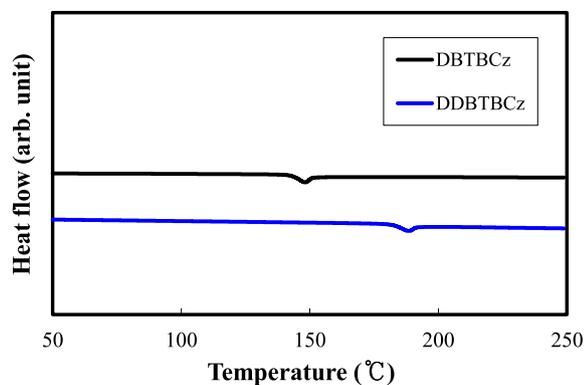


Fig. 4. DSC curves of DBTBCz and DDBTBCz.

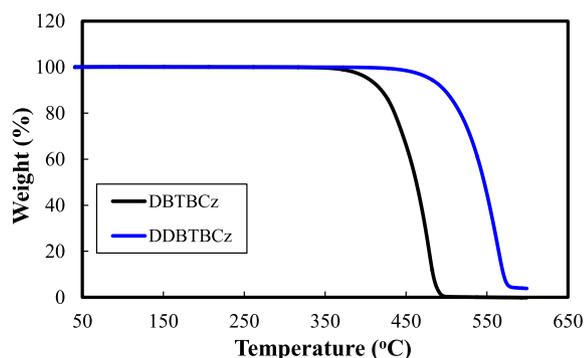


Fig. 5. TGA curves of DBTBCz and DDBTBCz.

modification of dibenzothiophene increased the  $T_g$  up to 148 °C and an additional dibenzothiophene modification further increased the  $T_g$  up to 189 °C due to restricted molecular motion by large molecular size and rigidity by additional 4- position modification. In previous work, the  $T_g$  of the bicarbazole host material with two dibenzothiophene units was 172 °C, but it was increased by 17 °C just by changing the interconnect position of dibenzothiophene owing to steric hindrance by 4- position connection [20]. This also affected the  $T_d$  at 5 wt% loss of the DBTBCz and DDBTBCz, resulting in high  $T_d$  of 478 °C and 564 °C in the DBTBCz and DDBTBCz, respectively. The thermal analysis results of DBTBCz and DDBTBCz suggest that the 4- position interconnection of dibenzothiophene differentiates the DBTBCz and DDBTBCz host materials from previous host materials in terms of thermal stability [20]. All material characterization results are summarized in Table 1.

The material characterization data described above proposed that the DBTBCz and DDBTBCz host materials are appropriate as the hosts of blue phosphorescent Ir (dbi)<sub>3</sub> emitter. Therefore, the two hosts were evaluated in the device structure optimized for blue PhOLEDs. Fig. 6 shows the device structure along with energy levels of each layer comprising the blue PhOLEDs. The blue PhOLEDs were grown by doping Ir (dbi)<sub>3</sub> at an optimized doping concentration of 15% in the DBTBCz and DDBTBCz hosts. Electrical measurement data of the DBTBCz and DDBTBCz devices obtained by voltage

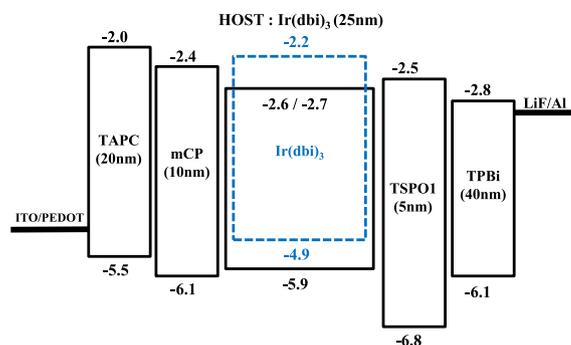


Fig. 6. Energy level diagram of the blue PhOLEDs.

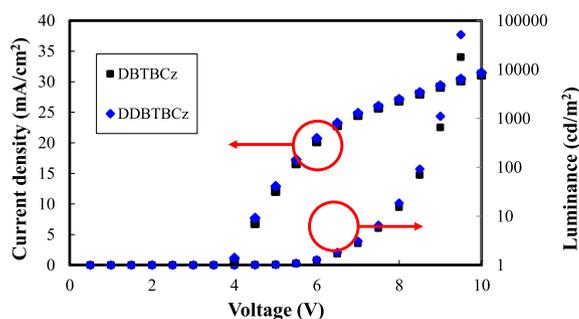


Fig. 7. Current density–voltage and luminance–voltage curves of DBTBCz and DDBTBCz devices doped with Ir (dbi)<sub>3</sub>.

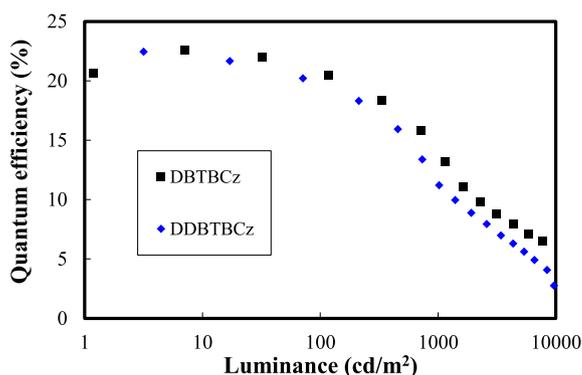


Fig. 8. Quantum efficiency–luminance plots of the DBTBCz and DDBTBCz devices doped with Ir (dbi)<sub>3</sub>.

sweep at an interval of 0.5 V are summarized in Fig. 7. The current density obtained from the voltage scan was of little difference in the two devices because of the similar HOMO and LUMO levels from CV measurements. Luminance obtained from the voltage scan was also plotted according to voltage in Fig. 7 in addition to the current density. The luminance were quite similar in the DBTBCz and DDBTBCz devices and only slight difference of the luminance was observed. As the current density at the same voltage was similar in the two devices, the similar luminance forecasts similar efficiency in the two devices.

Table 1

Photophysical, electrochemical and thermal properties of the materials.

	Singlet energy (eV)	Triplet energy (eV)	HOMO (eV)	LUMO (eV)	$T_g$ (°C)	$T_d$ (°C)
DBTBCz	3.09	2.92	−5.85	−2.65	148	478
DDBTBCz	3.09	2.94	−5.85	−2.60	189	564

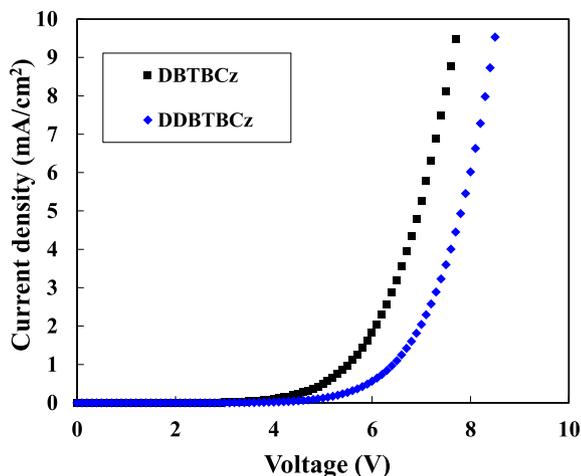


Fig. 9. Current density-voltage curves of hole only devices of DBTBCz and DDBTBCz.

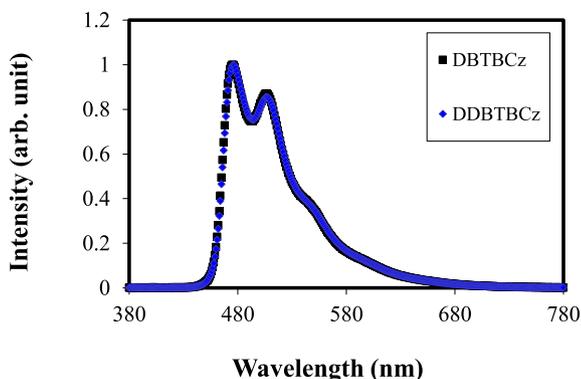


Fig. 10. EL spectra of the DBTBCz and DDBTBCz devices doped with Ir (dbi)<sub>3</sub>.

The combined electrical and optical measurements provided quantum efficiency (QE) of the DBTBCz and DDBTBCz devices in Fig. 8. As projected from the current density and luminance data, there was similarity of the QE in the DBTBCz and DDBTBCz devices by providing maximum QE of 22.6% and 22.5%, respectively. High QE was achieved in the two devices by good hole transport properties of the two hosts overcoming the strong hole trapping effect by Ir (dbi)<sub>3</sub> as predicted from the energy levels of the host and dopant materials. The bicarbazole backbone structure is responsible for the good hole transport character of the DBTBCz and DDBTBCz hosts. DBTBCz and DDBTBCz behaved quite similarly as the host of Ir (dbi)<sub>3</sub> judging from the current density, luminance and QE data. However, the efficiency roll-off was rather significant in the two devices because of strong hole trapping effect by Ir (dbi)<sub>3</sub> which narrows the emission zone of the Ir (dbi)<sub>3</sub> devices [27]. It was relatively insignificant in the DBTBCz device because of better hole transport properties of DBTBCz than that of DDBTBCz as presented in Fig. 9.

Table 2  
Summarized device performances of the DBTBCz and DDBTBCz devices.

	CIE (x,y)	EL $\lambda_{\max}$ (nm)	Quantum efficiency (%)		Current efficiency (cd/A)	
			[1000 cd/m <sup>2</sup> ]	[Max]	[1000 cd/m <sup>2</sup> ]	[Max]
DBTBCz	(0.21, 0.43)	475	14.13	22.61	34.41	53.55
DDBTBCz	(0.21, 0.43)	475	14.93	22.46	36.50	53.77
DBTBCz (lifetime)	(0.20, 0.41)	475	4.59	4.91	11.14	12.05
DDBTBCz (lifetime)	(0.20, 0.43)	475	5.05	6.83	12.53	17.42

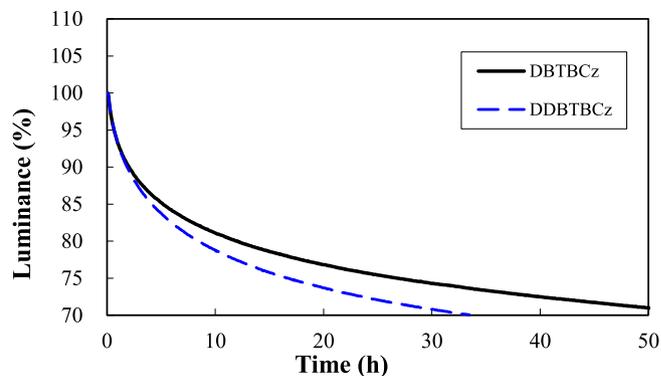


Fig. 11. Lifetime plots of the DBTBCz and DDBTBCz devices doped with Ir (dbi)<sub>3</sub> at an initial luminance of 1000 cd/m<sup>2</sup>.

Emission spectra of the DBTBCz and DDBTBCz devices in Fig. 10 indicate that only Ir (dbi)<sub>3</sub> emission was activated in the blue device by energy transfer process from the hosts to Ir (dbi)<sub>3</sub>. As the device structure was designed to block charge or exciton leakage from the device, pure Ir (dbi)<sub>3</sub> emission peak appeared in the device emission spectra. All device performances are listed in Table 2.

One important objective of designing the DBTBCz and DDBTBCz host materials was to develop stably operating host for extended lifetime. Therefore, the long-term operation behaviour of the blue PhOLEDs was investigated by driving the DBTBCz and DDBTBCz devices at a fixed luminance of 1000 cd/m<sup>2</sup>. Fig. 11 plots the luminance change of the DBTBCz and DDBTBCz blue PhOLEDs. It is clear from the luminance-operation time data that DBTBCz performed much better than DDBTBCz as the lifetime extending host material. In particular, the DBTBCz was even better than the host with 2- position modified dibenzothiophene in the bicarbazole backbone structure. At the same driving condition, the lifetime up to 70% of initial luminance was 55 h in our work, but it was 25 h in the blue PhOLEDs with the 2- position modified dibenzothiophene based host material [20]. This proves that 4- position modifying approach is better than 2- position modifying approach in the development of dibenzothiophene modified bicarbazole host materials. The improved lifetime of the DBTBCz device can be understood by two main factors related with material parameters. Rigidity of the molecular structure by 4- position modification seems to be one key reason for the improved lifetime of the DBTBCz and DDBTBCz devices compared to the BCzDBT device with a 2- position modified dibenzothiophene. The other key factor is good hole transport character of the host materials because facile hole transport would widen the exciton formation zone and stabilize the device by reduced triplet-triplet and triplet-polaron annihilation [2].

#### 4. Conclusions

The synthesis and device evaluation of high triplet energy hosts having dibenzothiophene unit linked to a bicarbazole backbone

structure via 4- position revealed that the host design approach linking the dibenzothiophene via 4- position is better than that via 2- position in terms of long-term stability of the devices. The DBTBCz host with the bicarbazole backbone structure and one dibenzothiophene unit exhibited both high efficiency and improved lifetime. Although absolute lifetime of the blue PhOLEDs is still short, but the design approach used in this work can open a way of improving the stability of the blue PhOLEDs.

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