Synthesis and optical properties of novel compounds containing carbazole and 1, 8-naphthalimide groups

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A series of novel carbazole-naphthalimide compounds with moieties capable of carrier-balance and electroluminescence were synthesised and characterised, and theirs luminescent properties had been studied.

Keywords: carbazole, naphthalimide, fluorescence, Suzuki cross-coupling

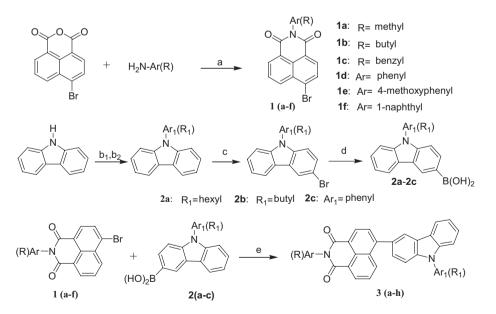
Since Tang and Vanslyke first reported the use of multi-layer organic thin films for light-emitting devices (LEDs),¹ organic light-emitting devices (OLEDs) have gained great interest all over the world. OLEDs have received more attention because of their potential applications in various displays.²⁻⁷ The organic electroluminescent devices have shown several advantages over inorganic ones, such as low cost, high luminous efficiency, wide selection of emission colours via molecular design of organic materials, and easy processing. As an important part of luminescent and fluorescent materials, some two and three chromophores light emitting compounds were synthesised.⁸

During the past several years, the synthesis and application of carbazole derivatives have been of great interest for the chemists and material scientists because these compounds are quite interesting due to their intrinsic photophysical and redox properties: they exhibit relatively intense luminescence and undergo reversible oxidation processes which make them suitable as hole carriers.^{9,10} Moreover, carbazole can be easily functionalised at its 3-, 6-, or 9-position and the carbazole moiety is beneficial for raising the glass-transition temperature and thermal stability. So carbazole derivatives are widely used as building blocks for potential organic semiconductors,¹¹⁻¹⁴ organic light-emitting devices.¹⁵⁻¹⁷ It is also well known that the 1,8-naphthalimide usually exhibits strong fluorescent

moieties for the study of photo-induced electron transfer,^{18,19} fluorescence switcher²⁰ or liquid crystal displays.²¹ Recently, 1,8-naphthalimide derivatives utilised as electroluminescent materials have been reported,²² whose fluorescence emission can be widely tuned (from blue to yellow, green and even red) with amino- and alkoxy-groups at the 4-position of 1,8-naphthalimide. Many multichromophore compounds have been synthesised.^{23,24} Our group has previously reported the synthesis of several 1,8-naphthalimide derivatives containing pyrazoline,²⁵ which have good green fluorescence.

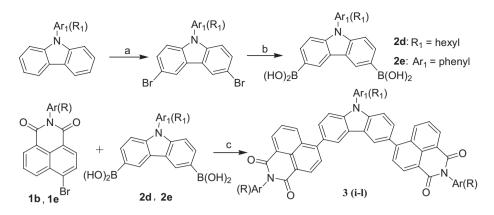
As a continuation of our works, we synthesised a series of novel two chromophores compounds containing carbazole and 1, 8-naphthalimide groups using carbazolboronic acids and 4-bromo-1,8-naphthalimides in this paper (Scheme 1), which had good green light fluorescence. At the same time some three chromophores compounds were obtained by the same method (Scheme 2). These compounds have not been reported previously.

To synthesise the targeting two chromophores compounds, our design is to attach carbazole directly to the 1,8naphthalimide via Suzuki cross-coupling. First, the 1,8naphthalimide compounds 1a-f were prepared according to the literature (Scheme 1).²⁶ Second, the boronic acid of carbazole 2a-c) as the key intermediate for the whole procedure was synthesised smoothly via a three-step



Scheme 1 a, Ar(R)NH₂, CH₃COOH(CH₃CH₂OH), reflux; b₁, carbazole: 1-bromohexane = 1: 1.1, KOH, tetrabutyl ammonim bromide, DMSO, 100°C; b₂, carbazole:1-iodobenzene = 1:1, Cul, K₃PO₄, L-proline, DMSO; c, 9-hexyl-9*H*-carbazole: NBS = 1: 1, CHCl₃, DMF, dark, 0°C; d, *n*-BuLi (1.2 eq), THF, -78°C, (CH₃O)₃B(2.4eq), H₂O/HCl; e, Pd(PPh₃)₄, K₂CO₃, toluene, 80°C.

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reaction.^{27,28} 9-Alkylcarbazole was obtained by the reaction of carbazole with 1-bromohexane or 1-bromobutane in DMSO catalysed by KOH and 9-arylcarbazole were synthesised in the presence of L-proline in DMSO, then 9-alkylcarbazole and 9-arylcarbazole with NBS translated into 3-bromocarbazole in CHCl₃, the boronic acid of carbazole was obtained via lithiathion with *n*-BuLi, followed by quenching with trimethyl borate and hydrolysis under acidic conditions. The final step to **3a–h** was also outlined in Scheme 1. As shown in Scheme 1, the carbazole moiety was introduced by C–C bond connection at its 3 position through a Suzuki cross-coupling of **1a–f** with the boronic acid of carbazole (**2a–c**) catalysed by Pd(PPh₃)₄. As we anticipated the products bearing carbazole-3-yl onto the 1,8-naphthalimide were all efficiently achieved with the excellent yields.

In order to synthesise the three chromophores compounds **3i–I**, firstly we obtained 3,6-disbromo-9-hexylcarbazole and 3,6-disbromo-9-phenyllcarbazole as shown in Scheme 2. Following, 3,6-disbromo-9-hexyl carbazole and 3,6-disbromo-9-phenyllcarbazole synthesised the bisboronic acid of carbazole via lithiathion with *n*-BuLi, followed by quenching with trimethyl borate and hydrolysis under acidic conditions. The final step to the three chromophores compounds (**3i–I**) was also outlined in Scheme 2.

Optical properties

The photophysical properties of all compounds **3a–I** were examined in dilute CH_2Cl_2 solution (*ca* 10⁻⁵ M). Photophysical data are summarised in Table 1.

The UV-Vis spectrums of **3a–I** were shown in Table 1. As shown in Table 1, the absorption spectra of the molecules **3a–I** exhibited the characteristic absorption peaks of carbazole unit between 280 and 345 nm and that of the 1,8-naphthalimide at 400 nm. It should be noted that not only does the absorption maximum of N-aryl-1,8-naphthalimide (**3d–f**) shows a small red-shift compared with that of N-alkyl-1,8-naphthalimide (**3a–c**), but also the absorption maximum of 4-(9-phenylcarbazol -3-yl)-1,8-naphthalimide (**3h**, **3k**, **3l**) shows a small blue-shift compared with that of 4-(9-hexyl-carbazol-3-yl)-1,8-naphthalimide (**3g**, **3i**, **3j**).

The emission spectrums of **3a-1** were also presented in Table 1. For two chromophores compounds (**3a** and **3h**),

it can be found that the maximum fluorescence emission wavelengths of 4-(9-phenyl-carbazol-3-yl)-1,8-naphthalimide (**3h**) shows a small blue-shift compared with that of 4-(9alkyl-carbazol-3-yl)-1,8-naphthalimide (**3a–g**). The same phenomenon was found in three chromophores compounds (**3k**, **3l**) compared with (**3i**, **3j**). Mention must be made here that the maximum fluorescence emission wavelengths of three chromophores compounds (**3i**, **3j**, **3k**) has a small blue-shift compared with that of two chromophores compounds (**3b**, **3e**, **3h**), respectively.

Figure 1 presented the emission spectrums of **3b** at different excitation wavelengths 280, 345 and 401 nm in CH_2Cl_2 solution (1 × 10⁻⁵ M). We can find their intensity of fluorescence all at 534 nm. It indicated that excitation wavelengths do not affect the emission wavelengths. The maximum fluorescence emission wavelengths of **3b** in different solvent were presented in Fig. 2. As shown in Fig. 2, the emission band strongly occurs blue-shift on decreasing the polar nature the solvent, reaching a maximum at 476 nm in toluene.

In conclusion, a series of novel two chromophores and three chromophores compounds containing carbazolyl and

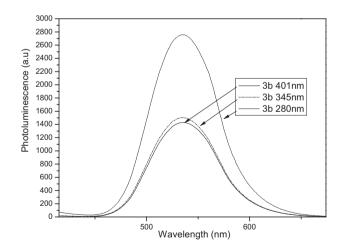


Fig. 1 The fluorescence emission spectra of **3b** at different excitation wavelengths in CH_2CI_2 (1 × 10⁻⁵ M).

Table 1 Absorption and fluorescence spectra of 3a-I in CH₂Cl₂ solution

Compound	3a	3b	3c	3d	3e	3f	3g	3h	3i	Зј	3k	31
λ ^{abs} (nm)	280	280	282	285	281	282	280	286	284	282	285	282
	345	345	345	344	344	344	345	345	345	343	345	344
	401	401	401	407	406	408	401	395	401	401	394	396
λ ^{em} _{max (} nm)	536	534	539	539	539	542	534	522	522	527	514	516

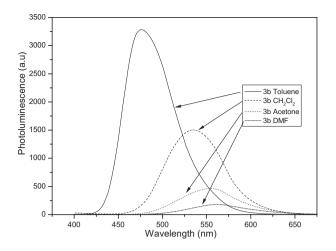


Fig. 2 The fluorescence emission spectra of **3b** in solvents of different polarities.

1,8-naphthalimide groups were synthesised via Suzuki cross-coupling. Their absorption and photoluminescence in dicholomethane solution were studied. They have different emitting fluorescence wavelengths ranging from 514 to 542 nm that may be served as good green light emitting materials. The application of novel compounds is under way in our laboratory.

Experimental

All chemicals were obtained commercially and used without further purification unless otherwise noted. THF was distilled over sodium and diphenylketone before use. Carbazole was purchased from Aldrich. Melting points were determined by an XT-5A micromelting point apparatus and are uncorrected. ¹H NMR spectra were determined on a Varian VXP-400s spectrometer using CDCl₃ as solvent and tetramethylsilane (TMS) as internal reference. IR Spectra was obtained on a Nicolet FT-IR500 spectrophotometer using KBr pellets. Elementary analyses were performed by a Carlo-Erba EA1110 CNNO–S analyser. Absorption spectra were determined on a Shimadzu UV-240 Spectrometer. Photoluminescence(PL) spectra were measured using a Shimadzu RF-5301PC spectrofluorometer at a concentration of 1.0×10^{-5} M in dichloromethane.

Experimental procedure (3a-f)

To a degassed (N₂) solution of the 4-bromo-1, 8-naphthalimide **1a–f** (2 mmol) and Pd(PPh₃)₄ catalyst (115 mg, 0.1 mmol, 5 mol.% per C–Br bond) in toluene (30 ml), a solution of carbazole-boronic acid (2.3 mmol) in toluene (10 ml) and 2M aqueous K₂CO₃ solution (4 ml) was added via syringe. The reaction mixture was stirred at 80°C for 10 h. After cooling, the product was extracted with DCM, washed with water and dried over MgSO₄. The solvent was evaporated to afford the crude mixture. After column chromatography on silica gel eluting with petroleum ester:ethyl acetate(8:1), pure compounds **3a–f** were obtained.

4-(9-hexyl-carbazol-3-yl)-N-methyl-1,8-naphthalimide (**3a**): Green powder(yield:87.6%). M.p. 167.5–168.5°C. ¹HNMR(CDCl₃,400MHz) δ : 0.90 (t, J = 7.4 Hz, 3H), 1.35–1.96 (m, 8H), 4.39 (t, J = 7.8 Hz, 2H), 3.63 (s, 2H), 7.51–7.62 (m, 5H), 7.72 (t, J = 8.0 Hz, 1H), 7.84 (d, J = 7.6 Hz, 1H), 8.14 (d, J = 7.6 Hz, 1H), 8.26 (s, 1H), 8.44 (d, J = 8.0 Hz, 1H), 8.66–8.71 (m, 2H). IR (KBr, cm⁻¹): 3056, 2956, 2933, 2856, 1698, 1659, 1590, 1490, 1466, 1405, 1366, 1289, 1235, 1158, 1042, 926, 818, 799, 749. Anal. Calcd. for C₃₁H₂₈N₂O₂: C, 80.84; H, 6.13; N, 6.08; Found: C, 80.93; H, 6.01; N, 5.89.

4-(9-hexyl-carbazol-3-yl)-N-butyl-1,8-naphthalimide (**3b**): Green powder (yield: 89%). M.p. 127–128°C. ¹H NMR (CDCl₃, 400 MHz) δ : 0.90 (t, J = 7.4 Hz, 3H), 1.01 (t, J = 7.4 Hz, 3H), 1.35–1.97 (m, 12H), 4.24 (t, J = 7.2 Hz, 2H), 4.40 (t, J = 7.8 Hz, 2H), 7.47–7.62 (m, 5H), 7.70 (t, J = 7.8 Hz, 1H), 7.82 (d, J = 7.6 Hz, 1H), 8.13 (d, J = 7.6 Hz, 1H), 8.24 (s, 1H), 8.41 (d, J = 8.4 Hz, 1H), 8.64–8.69 (m, 2H). IR (KBr, cm⁻¹): 3064, 2956, 2933, 2863, 1698, 1659, 1590, 1490, 1466, 1389, 1358, 1266, 1235, 1150, 1073, 949, 810, 780, 741, Anal. Calcd. for C₃₄H₃₄N₂O₂: C, 81.24; H, 6.82; N, 5.57; Found: C, 81.30; H, 6.75; N, 5.64.

4-(9-hexyl-carbazol-3-yl)-N-benzyl-1,8-naphthalimide (**3c**): Green powder (yield: 82%). M.p. 174–175°C. ¹H NMR (CDCl₃, 400 MHz) δ : 0.90 (t, J = 7.4 Hz, 3H), 1.35–1.95 (m, 8H), 4.39 (t, J = 7.0 Hz, 2H), 5.44 (s, 2H), 7.32 (d, J = 8.0 Hz, 2H), 7.50–7.59 (m, 8H), 7.70 (t, J = 8.0 Hz, 1H), 7.82 (d, J = 7.6 Hz, 1H), 8.12 (d, J = 7.2 Hz, 1H), 8.23 (s, 1H), 8.42 (d, J = 7.6 Hz, 1H), 8.65–8.71 (m, 2H). IR (KBr, cm⁻¹): 3064, 2956, 2933, 2856, 1690, 1659, 1590, 1520, 1466, 1381, 1343, 1235, 1150, 1026, 965, 810, 779, 746, 695. Anal. Calcd. for C₃₇H₃₂N₂O₂: C, 82.81; H, 6.01; N, 5.22; Found: C, 82.93; H, 5.95; N, 5.04.

4-(9-hexyl-carbazol-3-yl)-N-phenyl-1,8-naphthalimide (**3d**): Green powder (yield: 83.4%). M.p. 196.5-198°C. ¹H NMR (CDCl₃, 400 MHz) δ : 0.90 (t, J = 7.4 Hz, 3H), 1.25–2.17 (m, 8H), 4.40 (t, J = 7.0 Hz, 2H), 7.37 (d, J = 6.8 Hz, 2H), 7.50–7.62 (m, 8H), 7.74 (t, J = 7.8 Hz, 1H), 7.87 (d, J = 7.6 Hz, 1H), 8.14 (d, J = 7.2 Hz, 1H), 8.26 (s, 1H), 8.48 (d, J = 7.6 Hz, 1H), 8.68–8.74 (m, 2H). IR (KBr, cm⁻¹): 3056, 2956, 2933, 2856, 1706, 1667, 1590, 1489, 1466, 1366, 1243, 1188, 1026, 810, 790, 756. Anal. Calcd. for C₃₆H₃₀N₂O₂: C, 82.73; H, 5.79; N, 5.36; Found; C, 82.86; H, 5.90; N, 5.47.

4-(9-hexyl-carbazol-3-yl)-N-(4-methoxyphenyl)-1,8-naphthalimide (3e): Green powder (yield: 87%). M.p. 248–249°C. ¹H NMR (CDCl₃, 400 MHz) δ : 0.90 (t, J = 7.4 Hz, 3H), 1.32–1.97 (m, 8H), 3.89 (s, 3H), 4.40 (t, J = 7.0 Hz, 2H), 7.09 (d, J = 8.0 Hz, 2H), 7.26– 7.29 (m, 3H), 7.48–7.64 (m, 4H), 7.73 (t, J = 8.0 Hz, 1H), 7.85 (d, J = 7.2 Hz, 1H), 8.14 (d, J = 7.6 Hz, 1H), 8.26 (s, 1H), 8.47 (d, J = 8.0 Hz, 1H), 8.68-8.73 (m, 2H). IR (KBr, cm⁻¹): 3064, 2956, 2933, 2863, 1706, 1659, 1590, 1513, 1466, 1366, 1297, 1250, 1188, 1027, 810, 787, 741. Anal. Calcd. for C₃₇H₃₂N₂O₃: C, 80.41; H, 5.84; N, 5.07; Found; C, 80.53; H, 5.81; N, 5.23.

4-(9-hexyl-carbazol-3-yl)-N-[1]naphthyl-1,8-naphthalimide (3f): Green powder (yield: 85.5%). M.p. 214.5–216°C. ¹H NMR (CDCl₃, 400 MHz) δ : 0.91 (t, J = 7.4 Hz, 3H), 1.36–1.56 (m, 8H), 4.41 (t, J = 6.4 Hz, 2H), 7.29 (d, J = 7.0 Hz, 1H), 7.45–7.78 (m, 9H), 7.76 (t, J = 7.8 Hz, 1H), 7.90 (d, J = 7.2 Hz, 1H), 7.96–8.03 (m, 2H), 8.16 (d, J = 7.6 Hz, 1H), 8.29 (s, 1H), 8.53 (d, J = 8.4 Hz, 1H), 8.71–8.76 (m, 2H). IR (KBr, cm⁻¹): 3056, 2956, 2933, 2856, 1705, 1667, 1590, 1466, 1358, 1243, 1196, 1026, 872, 787, 749. Anal. Calcd. for C₄₀H₃₂N₂O₂: C, 83.89; H, 5.63; N, 4.89; Found: C, 84.03; H, 5.51; N, 4.72.

Experimental procedure (3g-h): To a degassed (N_2) solution of the 4-bromo-1,8-naphthalimide **1b** (2 mmol) and Pd(PPh₃)₄ catalyst (115 mg, 0.1 mmol, 5 mol.% per C–Br bond) in toluene (30 ml), a solution of carbazole-boronic acid **2b–c** (2.3 mmol) in toluene (10 ml) and 2M aqueous K₂CO₃ solution (4 ml) was added via syringe. The reaction mixture was stirred at 80°C for 10 h. After cooling, the product was extracted with DCM, washed with water and dried over MgSO₄. The solvent was evaporated to afford the crude mixture. After column chromatography on silica gel eluting with petroleum ester:ethyl acetate (8:1), pure compounds **3g–h** were obtained.

4-(9-butyl-carbazol-3-yl)-N-butyl-1,8-naphthalimide (3g): Green powder (yield: 85%). M.p. 167–168.5°C. ¹H NMR (400 MHz, CDCl₃) δ : 1.00 (t, J = 7.2 Hz, 6H), 1.45–1.50 (m, 4H), 1.77 (t, J = 7.6 Hz, 2H), 1.94 (t, J = 7.2 Hz, 2H), 4.24 (t, J = 7.6 Hz, 2H), 4.40 (t, J = 7.2 Hz, 2H), 7.26–7.27 (m, 2H), 7.48–7.62 (m, 3H), 7.71 (t, J = 8 Hz, 1H), 7.83 (d, J = 7.6 Hz, 1H), 8.12 (d, J = 8 Hz, 1H), 8.24 (s, 1H), 8.42 (d, J = 8.4 Hz, 1H), 8.64–8.69 (m, 2H). IR (KBr, cm⁻¹): 3056, 2948, 2870, 1697, 1659, 1589, 1466, 1381, 1358, 1234, 1149, 1072, 949, 872, 810, 802, 748. Anal. Calcd. for $C_{32}H_{30}N_2O_2$: C, 80.98; H, 6.37; N, 5.90; Found: C, 80.79; H, 6.32; N, 5.97.

4-(9-phenyl-carbazol-3-yl)-N-butyl-1,8-naphthalimide (**3h**): Green powder (yield: 82%). M.p. 169–170°C. ¹H NMR (400 MHz, CDCl₃) δ : 1.01 (t, J = 7.2 Hz, 3H), 1.45–1.52 (m, 2H), 1.75–1.77 (m, 2H), 4.24 (t, J = 7.2 Hz, 2H), 7.26 (s, 1H), 7.30–7.40 (m, 1H), 7.47–7.49 (m, 2H), 7.53–7.56 (m, 3H), 7.65–7.76 (m, 4H), 7.84 (d, J = 7.6 Hz, 1H), 8.18 (d, J = 7.6 Hz, 1H), 8.29 (s, 1H), 8.41 (d, J = 8.4 Hz, 1H), 8.65-8.70 (m, 2H). IR (KBr, cm⁻¹): 3056, 2955, 2871, 1697, 1589, 1651, 1505, 1458, 1358, 1234, 1188, 1072, 1026, 995, 941, 846, 810, 756, 694. Anal. Calcd. for C₃₄H₂₆N₂O₂: C, 82.57; H, 5.30; N, 5.66; Found: C, 82.49; H, 5.32; N, 5.77.

Experimental procedure (3i–l): To a degassed (N₂) solution of the 4-bromo-1, 8-naphthalimide **1b** or **1e** (2 mmol) and Pd(PPh₃)₄ catalyst (115 mg, 0.1 mmol, 5 mol.% per C–Br bond) in toluene (30 ml), a solution of carbazole–boronic acid **2d** or **2e** (0.67 mmol) in toluene (10 ml) and 2M aqueous K₂CO₃ solution (4 ml) was added via syringe. The reaction mixture was stirred at 80°C for 24 h. After cooling, the product was extracted with DCM, washed with water and dried over MgSO₄. The solvent was evaporated to afford the crude mixture. After column chromatography on silica gel eluting with petroleum ester:dichloromethane (1:1), pure compounds **3i–l** were obtained.

4,4'-(9-hexyl-carbazol-3,6-yl)-N-butyl-1,8-naphthalimide (3i):Green powder (yield: 69%). M.p. 226–227.5°C. ¹H NMR (400 MHz, CDCl₃) δ: 0.90-1.02 (m, 9H), 1.35-1.57 (m, 10H), 1.70-1.78 (m, 4H), 2.01-2.05 (m, 2H), 4.24 (t, J = 9.6 Hz, 4H), 4.49 (t, J = 9.2 Hz, 2H), 7.63–7.72 (m, 6H), 7.82 (d, J = 10.4 Hz, 2H), 8.27 ((s, 2H), 8.40 $(d, J = 11.2 \text{ Hz}, 2\text{H}), 8.62 - 8.68 \text{ (m, 4H)}; \text{ IR (KBr, cm}^{-1}): 3072, 2956,$ 2863, 1697, 1659, 1589, 1481, 1466, 1358, 1281, 1234, 1188, 1149, 1072, 1026, 957, 864, 810, 756, 617. Anal. Calcd. for C₅₀H₄₇N₃O₄: C, 79.65; H, 6.28; N, 5.57; Found: C, 79.56; H, 6.23; N, 5.64.

4,4'-(9-hexyl-carbazol-3,6-yl)-N-(4-methoxyphenyl)-1,8-naphthalimide (3j): Green powder (yield: 65%). M.p. >300°C. ¹H NMR (CDCl₃, 400 MHz) δ : 0.92 (t, J = 6.8 Hz, 3H), 1.34–1.44 (m, 6H), 2.01–2.05 (m, 2H), 3.89 (s, 6H), 4.49 (t, J = 9.2 Hz, 2H), 7.06–7.08 (m, 4H), 7.24-7.26 (m, 4H), 7.65-7.76 (m, 6H), 7.85 (d, J = 7.2 Hz, 2H), 8.31 (s, 2H), 8.45–8.47 (m, 2H), 8.68 (d, J = 7.2 Hz, 2H), 8.71 (d, J = 7.6 Hz, 2H). IR (KBr, cm⁻¹): 3072, 2933, 2863, 1706, 1667, 1590, 1513, 1466, 1366, 1281, 1242, 1188, 1134, 1034, 864, 818, 779, 756. Anal. Calcd. for C₅₆H₄₃N₃O₆: C, 78.76; H, 5.08; N, 4.92; Found: C, 79.02; H, 5.21; N, 5.04

4,4'-(9-phenyl-carbazol-3,6-yl)-N-butyl-1,8-naphthalimide (3k): Green powder (yield: 66%). M.p. 281-283°C. ¹H NMR (CDCl₃, 400 MHz) δ: 0.97 (t, J = 7.2 Hz, 6H), 1.14–1.49 (m, 4H), 1.69–1.76 (m, 4H), 4.22 (t, J = 9.6 Hz, 4H), 7.60–7.70 (m, 12H), 7.81 (d, J = 7.6 Hz, 2H), 8.30 (s, 1H), 8.38 (d, J = 8.4 Hz, 2H), 8.62 (d, J = 7.2 Hz, 2H), 8.66 (d, J = 7.6 Hz, 2H). IR (KBr, cm⁻¹): 3064, 2955, 2871, 1698, 1659, 1590, 1497, 1458, 1358, 1281, 1234, 1188, 1072, 949, 872, 818, 787, 756, 702. Anal. Calcd. for C₅₀H₃₉N₃O₄: C, 80.52; H, 5.27; N, 5.63; Found: C, 80.40; H, 5.21; N, 5.74.

4,4'-(9-phenyl-carbazol-3,6-yl)-N-(4-methoxyphenyl)-1,8-naphthalimide (31): Green powder (yield: 63%). M.p. >300°C. ¹H NMR (CDCl₃, 400 MHz) δ : 3.89 (s, 6H), 7.07–7.09 (m, 4H), 7.27–7.29 (m, 2H), 7.60–7.77 (m, 14H), 7.87 (d, J = 8.0 Hz, 2H), 8.36 (s, 1H), 8.46 (d, J = 8.4 Hz, 2H), 8.69 (d, J = 7.6 Hz, 2H), 8.73 (d, J = 7.6 Hz, 2H). IR (KBr, cm⁻¹): 3056, 2932, 2840, 1706, 1667, 1590, 1512, 1488, 1366, 1242, 1188, 1134, 1034, 818, 779, 756, 702. Anal. Calcd. for C₅₆H₃₅N₃O₆: C, 79.51; H, 4.17; N, 4.97; Found: C, 79.43; H, 4.28; N, 5.14.

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