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Synthesis, spectroscopic characteristic of novel fluorescent dyes of pyrazoline compounds

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ABSTRACT

Four novel fluorescence dyes of the pyrazoline were synthesized and fully characterized by means of ^{1}H, ^{13}C NMR, and HRMS. The optical, electrochemical properties were also investigated. Solvent effect on the fluorescence characteristics of the four compounds indicates that the emission wavelength was red-shifted with the increase of solvent polarity. As we expected, the results indicated that these compounds exhibited high quantum yields. Quantum chemical calculations were used to obtain optimized ground-state geometry, spatial distributions of the HOMO, LUMO levels of the compounds.

1. Introduction

Pyrazoline derivatives are fluorescent dyes, which are widely used for the optical brightening of textile fiber, plastics and paper [1]. Pyrazoline derivatives have not only excellent hole-transfer performance but also excellent emitting blueness property [2,3]. The fluorescence properties of these pyrazoline derivatives were reported by studying the effect of substituents on the absorption and fluorescence properties [4–8]. In fact, substitution in position 5 is responsible for spiroconjugated charge transfer quenching of pyrazoline fluorescence, whereas aryl units in positions 1 and 3 are essential to enhance fluorescence properties of the most useful products [2c].

Naphthalimide usually exhibits strong fluorescent emission on irradiation, which usually acts as supermolecular moieties for the study of photo-induced electron transfer [9,10], fluorescence switcher [11] or liquid crystal displays [12]. Recently, naphthalimide derivatives utilized as EL materials have been reported [13,14], 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 naphthalimide.

In our previous work, the photoluminescent (PL) properties of several dichromophore pyrazoline derivatives containing naphthalimide group were studied and founded to give green fluorescence [15,16]. Those compounds exhibited strong luminescence in solutions. Carbazole derivatives have been widely used as hole-transporting materials in the fabrication of the organic photoconductors, nonlinear optical materials, and photorefractive materials due to their electron-donating capabilities [17,18]. So if carbazole radical is linked to pyrazoline molecule, it would bring an excelsior efficiency. Herein the new optical materials based on naphthalimide pyrazoline incorporation of carbazole group have been synthesized (Fig. 1) and the optical, electrochemical properties of these compounds were discussed.

2. Experimental

2.1. Chemicals and instruments

All reactants were commercially available and used without further purification. Melting points were recorded on Electrothermal digital melting point apparatus and were uncorrected. ^{1}H and ^{13}C NMR spectra were recorded at 295 K on a Varian INOVA 400 MHz or a Varian NMR System 300 MHz spectrometer using $CDCl_3$ or d_6-DMSO as solvent and TMS as internal standard. UV–vis spectra were recorded on a Shimadzu UV-2501PC spectrometer; fluorescence spectra were obtained on an Hitachi FL-2500 spectrofluorimete; Cyclic voltammetry were carried on a Chi 1200A electrochemical

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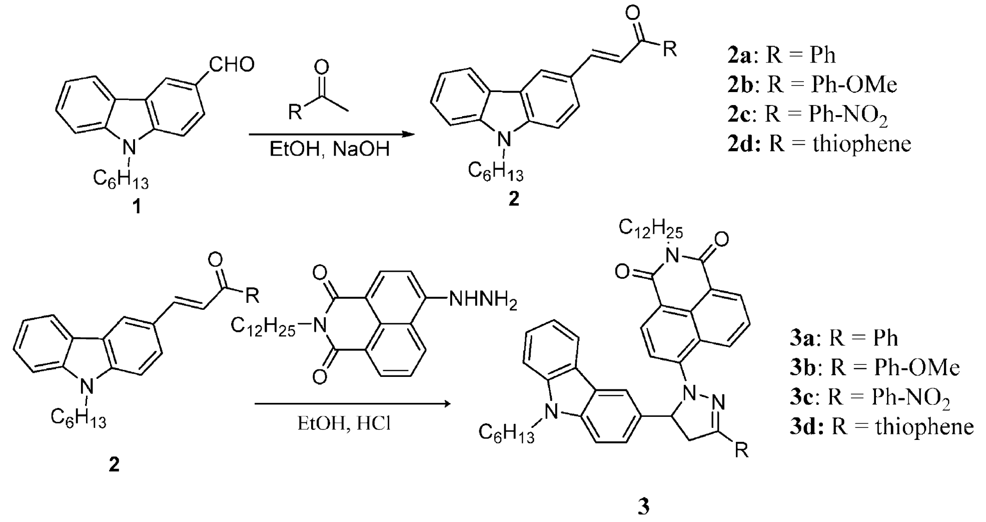

Fig. 1. Synthetic routines for compounds.

analyzer with three-electrode cell (platinum was used as working electrode and as counter electrode, and SCE as reference electrode) at room temperature; HRMS data were measured using TOF-MS(EI$^+$) instrument.

2.2. *9-hexyl-3-carbaldehyde (**1**)*

9-hexyl-3-carbaldehyde was prepared according to the literature [19]. Phosphorus oxychloride (1.57 mL, 16.82 mmol) was added dropwise to DMF (1.32 mL, 19.50 mmol) at 0 °C, and the mixture was stirred for 1 h at this temperature. Carbazole (3.35 g, 13.33 mmol) was added and the reaction mixture was stirred at 100 °C for 6 h. Then, the mixture was cooled to room temperature, poured into ice water and carefully neutralized with sodium hydroxide. The solution was extracted with dichloromethane (3× 150 mL), then, the organic phase was washed with water (2× 100 mL) and dried over anhydrous sodium sulfate. After filtration, the solvent was removed. The crude product was purified by recrystallization in ethanol.

Compound **1**: yield 90%, yellow. ^{1}H NMR (CDCl$_3$): δ 10.09 (s, 1H), 8.60 (s, 1H), 8.16 (d, J = 8.0 Hz, 1H), 8.01 (d, J = 8.0 Hz, 6H), 7.44–7.56 (m, 3H), 7.33 (t, J = 7.4 Hz, 1H), 4.32 (t, J = 7.2 Hz, 2H), 1.84–1.92 (m, 2H), 1.27–1.41 (m, 6H), 0.87 (t, J = 7.2 Hz, 3H).

2.3. *General procedure for the synthesis of the chalcones (**2**)*

The chalcones **2** were prepared according to the literature [20], as following procedure: 9-hexyl-9H-carbazole-3-carbaldehyde **1** (2.79 g, 10.00 mmol) was dissolved in EtOH (50 mL). 15% NaOH (2 mL) aqueous solution was added dropwise at 0 °C. Substitutional acetophenone (10 mmol) was added and was stirred for 3 h at ambient temperature. The reaction product was filtrated, dried and recrystallized in EtOH.

Compound **2a**: yield 86%, yellow. ^{1}H NMR (CDCl$_3$): δ 8.37 (s, 1H), 8.12–8.15 (m, 1H), 8.03–8.08 (m, 3H), 7.77–7.80 (m, 1H), 7.27–7.61 (m, 8H), 4.30 (t, J = 7.2 Hz, 2H), 1.87 (m, 2H), 1.27–1.39 (m, 6H), 0.86 (t, J = 7.2 Hz, 3H).

Compound **2b**: yield 82%, yellow. ^{1}H NMR (CDCl$_3$): δ 8.36 (s, 1H), 8.08–8.14 (m, 3H), 8.04 (d, J = 15.2 Hz, 1H), 7.76–7.78 (m, 1H), 7.59 (d, J = 15.6 Hz, 1H), 7.28–7.51 (m, 4H), 6.99 (d, J = 8.8 Hz, 2H), 4.28 (t, J = 7.2 Hz, 2H), 3.89 (s, 3H), 1.83–1.90 (m, 2H), 1.26–1.38 (m, 6H), 0.86 (t, J = 7.2 Hz, 3H).

Compound **2c**: yield 92%, yellow. ^{1}H NMR (CDCl$_3$): δ 8.40 (s, 1H), 8.37 (d, J = 8.8 Hz, 2H), 8.14–8.20 (m, 3H), 8.09 (d, J = 15.6 Hz, 1H), 7.80 (d, J = 8.6 Hz, 2H), 7.43–7.55 (m, 4H), 7.31 (d, J = 7.6 Hz, 1H), 4.32 (t, J = 7.0 Hz, 2H), 1.86–1.93 (m, 2H), 1.23–1.40 (m, 6H), 0.87 (t, J = 7.0 Hz, 3H).

Compound **2d**: Yield 94%, yellow. ^{1}H NMR (400 MHz, CDCl$_3$): δ 8.34 (s, 1H), 8.08–8.16 (m, 2H), 7.92–7.93 (d, J = 3.6 Hz, 1H), 7.78–7.81 (d, J = 8.4 Hz, 1H), 7.67–7.68 (d, J = 4.8 Hz, 1H), 7.41–7.53 (m, 4H), 7.28–7.32 (t, J = 7.4 Hz, 1H), 7.20–7.22 (t, J = 4.2 Hz, 1H), 4.29–4.33 (t, J = 7.2 Hz, 2H), 1.85–1.92 (m, 2H), 1.25–1.40 (m, 6H), 0.85–0.89 (t, J = 6.8 Hz, 3H).

2.4. *General procedure for the synthesis of the compounds (**3**)*

Compound **3** were synthesizsd by 2-hydrazinylnaphthalimide with chalcones **2** as following procedure: a mixture of 2-hydrazinylbenzothiazole (1.00 mmol) and chalcones **2** (1.00 mmol) in EtOH (5.00 mL) and 37% HCl (0.20 mL) was refluxed for 6–12 h. The resulting mixture was cooled down and the precipitates were filtered to afford the crude products, which can be purified by recrystallization in EtOH/THF (v/v = 1:1).

Compound **3a**: M.p.: 124–125 °C. yield 76%, red powder. ^{1}H NMR (400 MHz, CDCl$_3$): δ 9.76 (d, J = 9.2 Hz, 1H), 8.63 (d, J = 7.2 Hz, 1H), 8.23 (d, J = 8.4 Hz, 1H), 7.98–8.07 (m, 2H), 7.71–7.83 (m, 3H), 7.28–7.52 (m, 7H), 7.16–7.20 (m, 1H), 6.91 (d, J = 8.4 Hz, 1H), 5.84–5.89 (m, 1H), 4.22 (t, J = 7.2 Hz, 2H), 4.09 (t, J = 7.6 Hz, 2H), 3.60–3.93 (m, 1H), 3.36–3.42 (m, 1H), 1.64–1.82 (m, 4H), 1.21–1.36 (m, 24H), 0.81–0.87 (m, 6H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 165.0, 164.1, 152.1, 146.0, 141.0, 140.3, 134.7, 132.9, 132.6, 132.0, 131.5, 131.1, 130.9, 130.7, 130.0, 129.1, 126.5, 126.3, 124.9, 123.6, 123.5, 123.3, 122.6, 122.4, 120.6, 119.2, 118.0, 113.9, 111.0, 109.7, 109.1, 66.8, 43.3, 40.5, 32.1, 31.7, 29.8, 29.7, 29.6, 29.5, 29.1, 28.4, 27.4, 27.1, 22.9, 22.7, 14.4, 14.2;

HRMS [Found: m/z 758.4553 (M$^+$), Calcd for $C_{51}H_{58}N_4O_2$: M, 758.4560].

Compound **3b**: M.p.: 164–165 °C. yield 63%, red powder. ^{1}H NMR (400 MHz, CDCl$_3$): δ 8.62 (d, J = 7.2 Hz, 1H), 8.22 (d, J = 8.4 Hz, 1H), 8.06 (s, 1H), 8.02 (d, J = 7.6 Hz, 1H), 7.70–7.77 (m, 3H), 7.30–7.53 (m, 4H), 7.17–7.20 (m, 1H), 6.99 (d, J = 8.0 Hz, 2H), 6.87 (d, J = 8.8 Hz, 1H), 5.81–5.85 (m, 1H), 4.29–4.32 (m, 1H), 4.23 (t, J = 6.8 Hz, 2H), 4.10 (t, J = 7.0 Hz, 2H), 3.92–3.97 (m, 1H), 3.87 (s, 3H), 3.33–3.39 (m, 1H), 1.65–1.83 (m, 4H), 1.25–1.47 (m, 24H), 0.84–1.00 (m, 6H); ^{13}C NMR (75 MHz, CDCl$_3$): δ 160.4, 160.3, 159.5, 156.5, 147.5, 141.5, 136.3, 135.6, 130.3, 128.1, 126.8, 126.3, 126.2, 123.4, 121.6, 112.0, 118.8, 117.9, 117.7, 116.0, 114.5, 113.3, 112.4, 109.8, 108.7, 106.0,

Table 1
Optical and electrochemical properties of the compounds 3.

Compound	Abs. (nm)	E_m (nm)	Φ^a	Band gap[b]	HOMO/LUMO[b] (eV)	E_g (eV)	E_{ox}[c] (V)	E_{HOMO}/E_{LUMO}[d] (eV)
3a	465	505	0.15	3.18	−5.31/−2.13	3.26	1.45	−5.85/−2.59
3b	475	516	0.026	3.07	−5.12/−2.05	3.23	1.48	−5.88/−2.65
3c	476	548	0.021	2.97	−5.62/−2.65	3.38	1.39	−5.79/−2.41
3d	472	513	0.040	3.09	−5.23/−2.14	3.16	1.40	−5.80/−2.62

[a] Quantum yields (Φ) in $CHCl_3$ solutions were determined using quinine sulfate ($\Phi = 0.55$) as standard.
[b] DFT/B3LYP calculated values.
[c] Oxidation potential in DMF (10^{-3} M) containing 0.1 M $(n\text{-}C_4H_9)_4NPF_6$ with a scan rate of 100 mV s^{-1}.
[d] E_{HOMO} was calculated by E_{ox} + 4.4 V (vs NHE), and $E_{LUMO} = E_{HOMO} - E_g$.

105.1, 104.4, 62.0, 51.0, 38.7, 35.8, 27.5, 27.1, 25.2, 25.0, 24.9, 24.8, 24.7, 24.5, 23.7, 22.8, 22.5, 18.3, 18.1, 9.7, 9.6;

HRMS [Found: *m*/*z* 788.4654 (M^+), Calcd for $C_{52}H_{40}N_4O_3$: M,788.4665].

Compound **3c**: M.p.: 98–99 °C. yield 52%, red powder. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.58 (d, J = 8.8 Hz, 1H), 8.66 (d, J = 7.2 Hz, 1H), 8.31 (d, J = 8.8 Hz, 2H), 8.25–8.28 (m, 1H), 8.00–8.10 (m, 2H), 7.91–7.94 (m, 2H), 7.76–7.80 (m, 1H), 7.32–7.47 (m, 4H), 7.16–7.21 (m, 1H), 7.02 (d, J = 8.4 Hz, 1H), 5.95–6.00 (m, 1H), 4.23 (t, J = 7.2 Hz, 2H), 4.10 (t, J = 7.6 Hz, 2H), 3.95–4.02 (m, 1H), 3.40–3.46 (m, 1H), 1.61–1.86 (m, 4H), 1.22–1.36 (m, 24H), 0.79–0.88 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.1, 159.3, 152.6, 151.8, 144.6, 143.3, 140.5, 136.4, 135.7, 133.6, 129.1, 127.6, 126.9, 126.0, 125.4, 124.7, 122.1, 121.8, 120.8, 119.8, 118.9, 118.2, 117.6, 115.9, 114.6, 113.4, 110.7, 107.5, 105.2, 104.5, 62.9, 38.7, 38.2, 38.1, 35.9, 35.8, 27.5, 27.1, 25.2, 25.1, 25.0, 24.9, 24.5, 23.7, 22.7, 22.5, 18.3, 18.1, 9.7, 9.6;

HRMS [Found: *m*/*z* 803.4427 (M^+), Calcd for $C_{51}H_{57}N_5O_4$: M, 803.4411].

Compound **3d**: M.p.: 113–115 °C. yield 64%, red powder. ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.70 (d, J = 8.8 Hz, 1H), 8.62 (d, J = 7.2 Hz, 1H), 8.23 (d, J = 8.4 Hz, 1H), 8.06 (s, 1H), 8.02 (d, J = 7.6 Hz, 1H), 7.71–7.75 (m, 1H), 7.31–7.44 (m, 5H), 7.17–7.23 (m, 2H), 7.09–7.11 (m, 1H), 6.90 (d, J = 8.8 Hz, 1H), 5.85–5.90 (m, 1H), 4.23 (t, J = 7.2 Hz, 2H), 4.10 (t, J = 7.6 Hz, 2H), 3.93–4.00 (m, 1H), 3.36–3.43 (m, 1H), 1.57–1.85 (m, 4H), 1.22–1.36 (m, 24H), 0.82–0.88 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 160.3, 159.4, 143.2, 141.1, 136.4, 135.6, 134.2, 131.2, 123.0, 128.1, 127.9, 126.8, 126.2, 125.8, 123.6, 123.4, 123.2, 121.7, 120.3, 118.9, 118.8, 118.7, 117.9, 116.0, 114.6, 113.4, 109.2, 106.4, 105.1, 104.4, 62.4, 39.2, 38.7, 35.8, 27.3, 27.1, 25.2, 25.1, 25.0, 24.9, 24.5, 23.7, 22.8, 22.5, 18.3, 18.1, 9.7, 9.6;

HRMS [Found: *m*/*z* 764.4124 (M^+), Calcd for $C_{49}H_{56}N_4O_2S$: M, 764.4124].

3. Results and discussion

3.1. Absorption and fluorescence spectra

The UV–vis absorption and photoluminescent (PL) properties of compounds 3 in dilute solutions are presented in Table 1. As shown in Fig. 2, these compounds exhibit two prominent bands in the solution, appearing at 336–352 nm and 464–476 nm, respectively. The absorption peaks at about 336–352 nm are attributed to the absorption of the carbazole moiety; the absorption peak at 464–473 nm is attributed to naphthalimide unit [9c]. The maximum absorption of **3c** was located at 476 nm and the others appeared at 465–472 nm. The red-shift phenomenon of **3c** was due to the electron affinity. There are slightly differences among these compounds, although they are similar in the structure. Thus, these differences might be result from different conjugation degree and electron effect in these compounds [21].

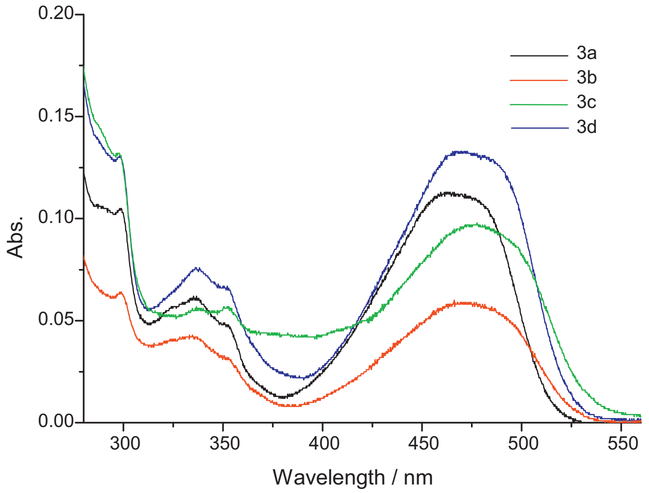

Fig. 2. The absorption spectra of Compound **3a–d** (1×10^{-5} mol L^{-1}) in CH_2Cl_3.

Fig. 3 shows the fluorescence spectra for these compounds in diluted toluene solutions. Compounds **3** present a blue emission with the peaks varying from 505 nm to 548 nm. The difference in the fluorescence spectra of 3 and 1,3,5-triphenyl-2-pyrazoline (TPP) may be possibly due to the introduction of naphthalimide unit at the C-3 position in these compounds. As shown in Fig. 3, the emission peaks of **3a**, **3b** and **3d** were at 505 nm, 516 nm, and 513 nm, respectively, which may be possibly due to the different conjugation degree and different electron effect in these compounds. In the case of **3c**, the NO_2 functional group with the electron-accepting ability is introduced. As a result, the position of the maximum emission peaks was red-shifted at 530 nm [2c].

The fluorescence quantum yields (Φ) were measured in $CHCl_3$ using quinine sulfate ($\Phi = 0.55$) as standard [22]. The Φ value of 0.15 is observed for **3a**, which is higher than that of other compounds. The PL quantum yields of the other compounds are in the range of 0.021–0.040. This difference of quantum yields might be due to the

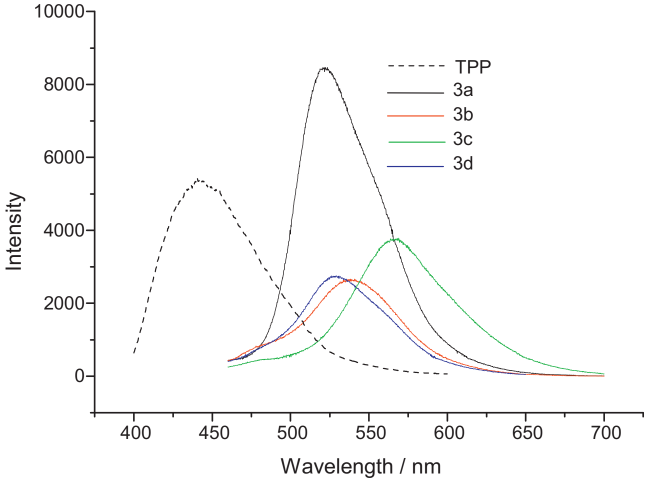

Fig. 3. Fluorescence emission spectra of compounds **3a–d** (1×10^{-5} mol L^{-1}) in THF.

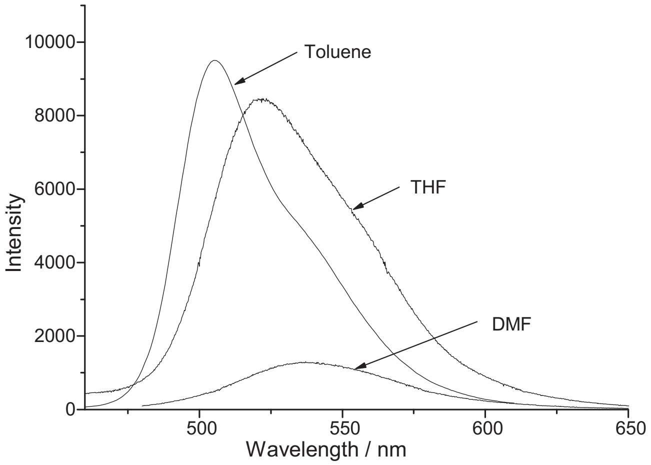

Fig. 4. The emission spectra of compound **3a** in different solvents (1×10^{-5} mol L^{-1}).

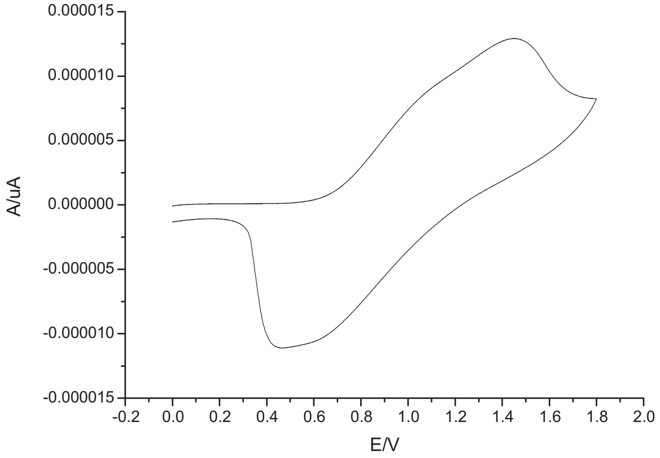

Fig. 5. Cyclic voltammetry of the compound **3a** in 0.1 mol L^{-1} Bu_4NPF_6–$CHCl_3$.

change of the electronic push–pull substitution of the conjugated part in the molecules [4].

Moreover, the solvent effect on the fluorescence characteristics of these compounds was studied, which indicated that the emission wavelength of the compound was red-shifted with the increase of solvent polarity (Fig. 4) [6,7].

3.2. Electrochemical properties

The electrochemical properties of compounds **3** were analyzed by cyclic voltammetry in $CHCl_3$ in the presence of tetrabutylammonium hexafluorophosphate (0.10 mol L^{-1}) as supporting electrolyte (Fig. 5) and the results are listed in Table 1. All CV measurements were recorded at room temperature with a conventional three electrode configuration consisting of a platinum wire working electrode, a platinum counter electrode, and a SCE (saturated calomel electrode) reference electrode under argon. Electrochemical band gaps were calculated from onset potentials of the anodic and cathodic waves [23]. The cyclic voltammetry (CV) of compounds **3** exhibit an reversible oxidation process which shift positively from −1.48 to −1.39 V. As shown in Table 1, the HOMO ranges are from −5.88 to −5.79 eV, while the LUMO ranges are from −2.65 to −2.41 eV, which are in agreement with the calculated values (−5.62 to −5.12 eV for the HOMO, and −2.65 to −2.05 eV for the LUMO of compounds), which were near the most widely used hole-transport material 4,4′-bis(1-naphthylphenylamino)biphenyl (NPB) (HOMO = −5.5 eV, LUMO = −2.4 eV) and therefore, these compounds might be used as hole-transporting materials in OLEDs [24]. Therefore, these compounds might be used for hole-transporting and electron-transporting materials for OLEDs [4].

3.3. Theoretical calculation

The ground-state geometry of compounds were optimized by hybrid densityfunctional theory (B3LYP) with 6-31G* basis set in the Gaussian 03 program package [25] (Fig. 6). The dihedral angle of 3 formed between the naphthalimide ring and a benzene ring of carbazole is about 30 degree and the whole molecule takes a planar configuration, which helps to impede the π–π* stacking interaction in solid state to some extent. Fig. 7 illustrates the calculated spatial distributions of the HOMO (the highest occupied molecular orbital), LUMO (the lowest unoccupied molecular orbital) levels with

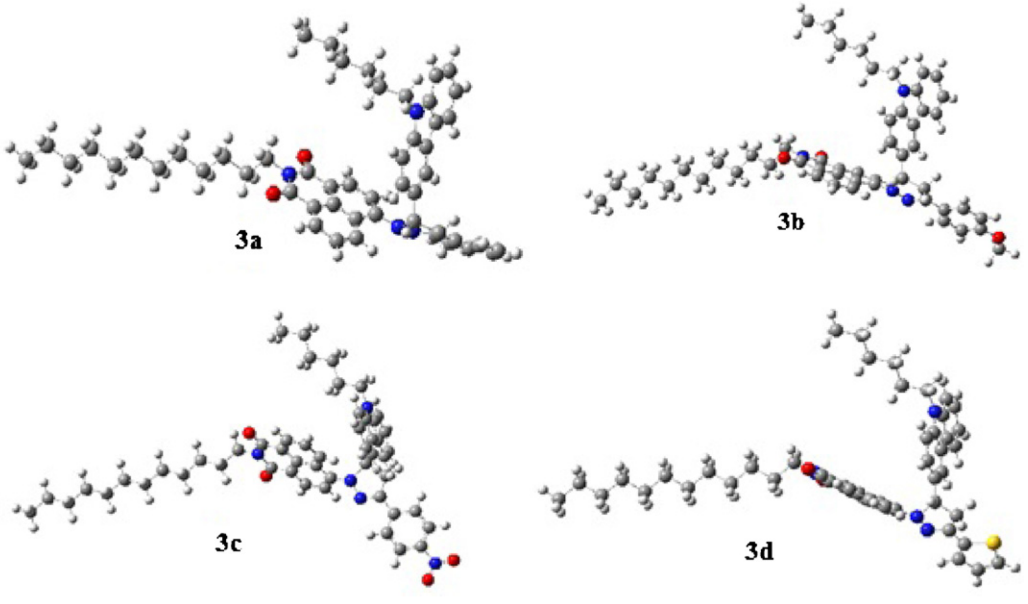

Fig. 6. Optimized ground-state geometry of compounds with B3LYP/6-31G* in gas phase.

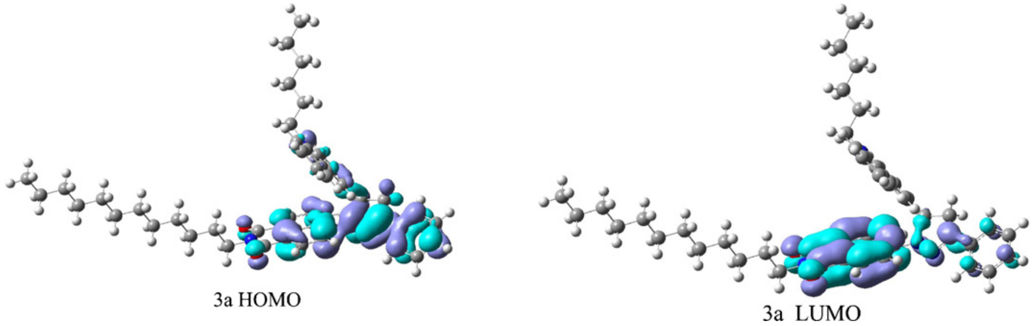

Fig. 7. Calculated spatial distributions of the HOMO, LUMO levels of compound **3a**.

compound **3a** as sample. The theoretically predicted energy levels are a little lower. The lower values than those estimated experimentally may be related with various effects such as conformation and salvation which were not taken into account. The HOMO/LUMO energy levels and band gaps of the compounds are listed in Table 1.

4. Conclusions

A series of new organic fluorescent dyes of pyrazoline compounds have been synthesized. Their optical, electrochemical properties are studied. As believed, these compounds showed efficient emission from blue to green. Moreover, all of the compounds exhibit suited HOMO ranges (−5.62 to −5.12 eV), which have promising potential for application in OLEDs.

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