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The synthesis, characterization and optical properties of novel 5-(3-aryl-1*H*-pyrazol-5-yl)-2-(3-butyl-1-chloroimidazo[1,5-a] pyridin-7-yl)-1,3,4-oxadiazole

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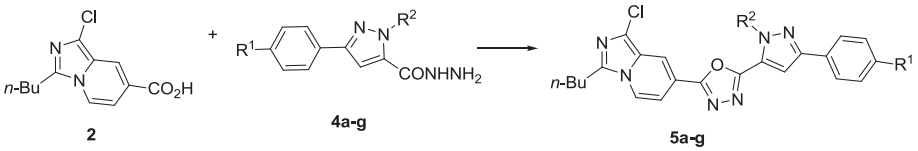
HIGHLIGHTS

- Novel 1,3,4-oxadiazole derivatives were prepared and fully characterized.
- UV–vis absorption and fluorescence spectroscopy of all compounds were measured.
- The minimized structures, the molecular orbital (HOMO and LUMO) energies and the orbital maps of compound **5a–g** were calculated.

GRAPHICAL ABSTRACT



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ABSTRACT

A series of novel 5-(3-aryl-1*H*-pyrazol-5-yl)-2-(3-butyl-1-chloroimidazo[1,5-a]- pyridin-7-yl)-1,3,4-oxadiazole derivatives has been synthesized from 3-butyl-1-chloroimidazo[1,5-a]pyridine-7-carboxylic acid and ethyl 3-aryl-1*H*-pyrazole-5-carboxylate. The compounds were characterized using IR, ^{1}H NMR, HRMS and UV–vis absorption. The fluorescence spectral characteristics of the compounds in dichloromethane were investigated. The results showed that absorption λ_{max} and emission λ_{max} was less correlated with substituent groups on N-1 position of pyrazole moiety and para position of benzene moiety. The calculated molecular orbital correlates well with their absorption.

Introduction

1,3,4-Oxadiazoles have attracted intense attention for their potential use in organic electronics because either polymers or small molecules of this kind have been widely used as electron-transporting materials or electroluminescence (EL) materials in organic light emitting diodes (OLEDs) [1–10]. In addition, electron transporting 1,3,4-oxadiazole moiety has been connected to many chelating ligands to obtain luminescent complexes with more new functions [11–14].

Pyrazole and Imidazo[1,5-a]pyridine unit are important core structures in a number of natural products. Many pyrazole derivatives are known to exhibit a wide range of biological properties such as anti-hyperglycemic, analgesic, anti-inflammatory, anti-pyretic, anti-bacterial, antifungal, anti-hypoglycemic, sedative-hypnotic activity, antitumor and anticoagulant activity [15,16]. Imidazo[1,5-a]pyridines derivatives have also been reported to show broad spectrum of biological activities including cardiotonic agents, aromatase inhibitors in estrogen-dependent diseases, thromboxane A2 synthetase inhibitors, and angiotensin II receptor antagonists [17,18]. They also have potential applications in organic light-emitting diodes (OLEDs), in organic thin-layer field effect transistors (FETs), and as precursors of N-heterocyclic carbenes [19,20].

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The design and synthesis of fluorescent small molecules with desirable bioactivities is of considerable current interest in biology research. Thus, in continuation of our efforts in synthesizing various bioactive molecules [21–25] and fluorescent small molecules [26–31] and based on a fragmented approach, we proposed that 1,3,4-oxadiazole linking imidazo[1,5-a]pyridine and pyrazole might have interesting bioactivities such as anticancer activity. On the other hand, three aromatic rings construct π-conjugation system that has cause for expecting improvement in UV–vis absorption and fluorescence spectral characteristics. Herein, we would like to report the synthesis, UV–vis absorption and fluorescence spectral characteristics of novel 5-(3-aryl-1*H*-pyrazol-5-yl)-2-(3-butyl-1-chloroimidazo- [1,5-a]pyridin-7-yl)-1,3,4-oxadiazole derivatives.

Experimental

General

All reagents were commercially available and used without further purification. Melting points were determined on an XD-4 digital micro melting point apparatus. ^{1}H NMR spectra were recorded on a Bruker Avance 300 (300 MHz) spectrometer, using $CDCl_3$ as solvent and tetramethylsilane (TMS) as internal standard. IR spectra were recorded with an IR spectrophotometer VERTEX 70 FT-IR (Bruker Optics). HRMS spectra were recorded on a Q-TOF6510 spectrograph (Agilent). UV–vis spectra were recorded on a U-4100 (Hitachi). Fluorescent measurements were recorded on a Perkin-Elmer LS-55 luminescence spectrophotometer.

Synthesis

The synthetic approach for the preparation of substituted 1,3,4-oxadiazole derivatives is shown in Fig. 1 (Supplementary data). Compounds **2** and **4a–g** were synthesized according to the literature method [32,33]. A solution of compound **4** (1 mmol) and 3-butyl-1-chloroimidazo[1,5-a]pyridine-7-carboxylic acid (1 mmol) in freshly distilled $POCl_3$ (caution: Reacts violently with water; incompatible with many metals, alcohols, amines, phenol, DMSO, strong bases) (15 ml) was refluxed for 9–12 h. Excess $POCl_3$ was distilled off and the residue was poured into iced water. The precipitate was filtered, washed with water, dried and recrystallized from ethanol to give compound **5** in 33–46%.

*2-(1-Benzyl-3-phenyl-1H-pyrazol-5-yl)-5-(3-butyl-1-chloroimidazo[1,5-a]pyridin-7-yl)-1,3,4-oxadiazole (**5a**)*

Yellow solid (46% yield): mp 174.8–177.5 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.05 (s, 1H), 7.91 (m, 2H), 7.77 (d, *J* = 7.2 Hz, 1H), 7.48–7.26 (m, 10H), 6.98 (m, 2H), 6.00 (s, 2H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.85 (m, 2H), 1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); IR (KBr) *ν* = 3073, 2954, 2920, 2851, 1633, 1491, 1457, 1366, 1296 cm^{-1}; HRMS: *m*/*z* calcd for $C_{29}H_{26}ClN_6O$ $[M+H]^+$ 509.1857, found 509.1863.

*2-(3-Butyl-1-chloroimidazo[1,5-a]pyridin-7-yl)-5-(1-(4-nitrobenzyl)-3-phenyl-1H-pyrazol-5-yl)-1,3,4-oxadiazole (**5b**)*

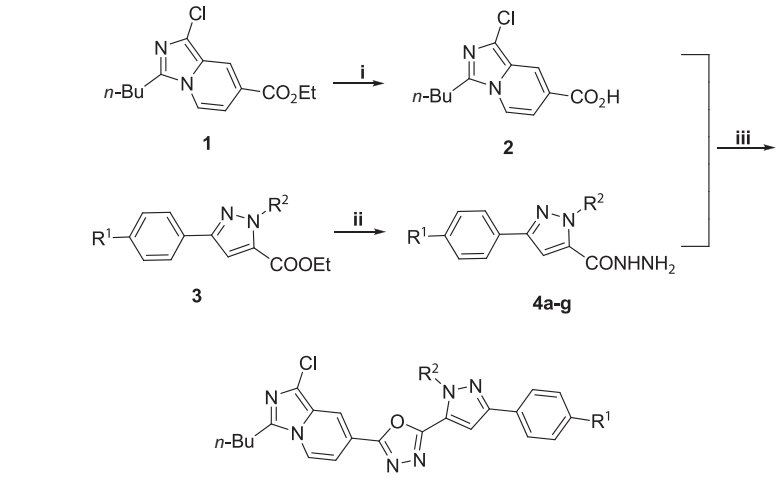
Yellow solid (35% yield): mp 220.8–223.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.16 (d, *J* = 8.7 Hz, 2H), 8.09 (s, 1H), 7.91 (d, *J* = 7.2 Hz, 2H), 7.79 (d, *J* = 7.5 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 2H), 7.50–7.40 (m, 3H), 7.36 (s, 1H), 7.28 (m, 1H), 6.11 (s, 2H), 2.98 (t, *J* = 7.2 Hz, 2H), 1.85 (m, 2H), 1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); IR (KBr) *ν* = 3091, 2956, 2868, 1632, 1493, 1473, 1360, 1246 cm^{-1}; HRMS: *m*/*z* calcd for $C_{29}H_{25}ClN_7O_3$ $[M+H]^+$ 554.1707, found 554.1703.

*2-(3-Butyl-1-chloroimidazo[1,5-a]pyridin-7-yl)-5-(1-(4-fluorobenzyl)-3-phenyl-1H-pyrazol-5-yl)-1,3,4-oxadiazole (**5c**)*

Yellow solid (33% yield): mp 178.3–179.0 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.11 (s, 1H), 7.91 (m, 3H), 7.46–7.36 (m, 7H), 6.99 (m, 2H), 5.97 (s, 2H), 3.01 (t, *J* = 7.2 Hz, 2H), 1.89 (m, 2H), 1.48 (m, 2H), 0.99 (t, *J* = 7.2 Hz, 3H); IR (KBr) *ν* = 3068, 2957, 2868, 1633, 1494, 1473, 1228 cm^{-1}; HRMS: *m*/*z* calcd for $C_{29}H_{25}ClFN_6O$ $[M+H]^+$ 527.1762, found 527.1764.

*2-(3-Butyl-1-chloroimidazo[1,5-a]pyridin-7-yl)-5-(1-(4-chlorobenzyl)-3-phenyl-1H-pyrazol-5-yl)-1,3,4-oxadiazole (**5d**)*

Yellow solid (41% yield): mp 176.2–177.2 °C; ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.06 (s, 1H), 7.91 (m, 3H), 7.80 (s, 1H), 7.48–7.28 (m, 8H), 5.96 (s, 2H), 3.00 (t, *J* = 7.2 Hz, 2H), 1.86 (m, 2H), 1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); IR (KBr) *ν* = 3065, 2957,



Reagents and Conditions:
(i) KOH,C_2H_5OH, H_2O, reflux;
(ii) $N_2H_4{\cdot}H_2O$, C_2H_5OH, reflux;
(iii) $POCl_3$, reflux.

5a R^1 = H, R^2 = Bn
5b R^1 = H, R^2 = *p*-NO_2Bn
5c R^1 = H, R^2 = *p*-FBn
5d R^1 = H, R^2 = *p*-ClBn
5e R^1 = Me, R^2 = Bn
5f R^1 = Me, R^2 = *p*-NO_2Bn
5g R^1 = Me, R^2 = *p*-FBn

Fig. 1. The synthetic route of unsymmetrical 1,3,4-oxadiazole derivatives.

2869, 1635, 1521, 1473, 1345, 1247 cm^{-1}; HRMS: *m/z* calcd for $C_{29}H_{25}Cl_2N_6O$ $[M+H]^+$ 543.1467, found 543.1471.

*2-(1-Benzyl-3-p-tolyl-1H-pyrazol-5-yl)-5-(3-butyl-1-chloroimidazo[1,5-a]pyridin-7-yl)-1,3,4-oxadiazole (**5e**)*

Yellow solid (42% yield): mp 182.8–183.3 °C; ^1H NMR (300 MHz, $CDCl_3$): δ 8.06 (s, 1H), 7.80 (m, 3H), 7.41–7.28 (m, 9H), 7.36 (s, 1H), 7.28 (m, 1H), 5.99 (s, 2H), 3.00 (t, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.85 (m, 2H), 1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); IR (KBr) *v* = 3066, 2954, 2867, 1633, 1482, 1455, 1345, 1300, 1245 cm^{-1}; HRMS: *m/z* calcd for $C_{30}H_{28}ClN_6O$ $[M+H]^+$ 523.2013, found 523.2009.

*2-(3-Butyl-1-chloroimidazo[1,5-a]pyridin-7-yl)-5-(1-(4-nitrobenzyl)-3-p-tolyl-1H-pyrazol-5-yl)-1,3,4-oxadiazole (**5f**)*

Yellow solid (40% yield): mp 222.7–223.8 °C; ^1H NMR (300 MHz, $CDCl_3$): δ 8.16 (d, *J* = 8.7 Hz, 2H), 8.08 (s, 1H), 7.80 (d, 3H), 7.54 (d, *J* = 8.7 Hz, 2H), 7.31–7.28 (m, 4H), 6.09 (s, 2H), 2.99 (t, *J* = 7.2 Hz, 2H), 2.41 (s, 3H), 1.85 (m, 2H), 1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); IR (KBr) *v* = 3072, 2953, 2867, 1632, 1481, 1319, 1230 cm^{-1}; HRMS: *m/z* calcd for $C_{30}H_{27}ClN_7O_3$ $[M+H]^+$ 568.1864, found 568.1872.

*2-(3-Butyl-1-chloroimidazo[1,5-a]pyridin-7-yl)-5-(1-(4-fluorobenzyl)-3-p-tolyl-1H-pyrazol-5-yl)-1,3,4-oxadiazole (**5g**)*

Yellow solid (35% yield): mp 164.9–167.7 °C; ^1H NMR (300 MHz, $CDCl_3$): δ 8.07 (s, 1H), 7.79 (m, 3H), 7.43 (m, 2H), 7.28 (m, 4H), 6.98 (m, 2H), 5.95 (s, 2H), 2.98 (t, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.85 (m, 2H), 1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H); IR (KBr) *v* = 3079, 2959, 2860, 1635, 1526, 1481, 1345, 1246 cm^{-1}; HRMS: *m/z* calcd for $C_{30}H_{27}ClFN_6O$ $[M+H]^+$ 541.1919, found 541.1913.

Results and discussion

Synthesis and structure characterization

Starting ethyl 3-butyl-1-chloroimidazo[1,5-a]pyridine-7-carboxylate **1** can be easily synthesized by the reactions of commercially available 2-butyl-4-chloro-1*H*-imidazole-5-carbaldehyde and ethyl 4-bromobut-2-enoate in the presence of K_2CO_3. 3-Aryl-1*H*-pyrazole-5-carbohydrazide derivatives **4** were synthesized by the reaction of ethyl 3-aryl-1H-pyrazole-5-carboxylate derivatives **3** and hydrate hydrazine according to the literature[33]. Subsequently, diacylhydrazides **5** were synthesized by the reaction of acid **2** and 3-aryl-1*H*-pyrazole-5-carbohydrazide derivatives 5 in 33–46% yields. The structures of products **5a–g** were characterized by spectroscopic methods (^1H NMR, ^{13}C NMR, IR and HRMS). For example, compound **5g**, obtained as yellow crystals, gave a $[M+H]^-$ion peak at *m/z* 541.1913 in the HRMS, in accord with the molecular formula $C_{30}H_{27}ClFN_6O$. The IR spectra showed the characteristic absorption bands at 3079, 2959, 2860 (C—H), 1635 (C=C), 1526 (C=N), 1481 (N—N), and 1246 (C—O—C) cm^{-1}.

The ^1H NMR spectra ($CDCl_3$) revealed three distinct singlets at δ 2.40 (3H, Ar-CH_3), 5.95 (2H, Ph-CH_2-) and 8.07 (1H, imidazo[1,5-a]pyridine moiety). Moreover, it showed peaks at δ 2.98 (t, *J* = 7.2 Hz, 2H), 1.85 (m, 2H), 1.46 (m, 2H), 0.98 (t, *J* = 7.2 Hz, 3H) assigned to the protons of butyl group. All other signals are consistent with the structure of **5g**.

Absorption spectral

UV–is absorption spectra of compounds **5a–g** were observed in dichloromethane solution with the concentration of 1×10^{-5} M, as shown in Fig. 2 and Table 1. Several absorption peaks could be observed in the wavelength range from 220 to 450 nm, while almost

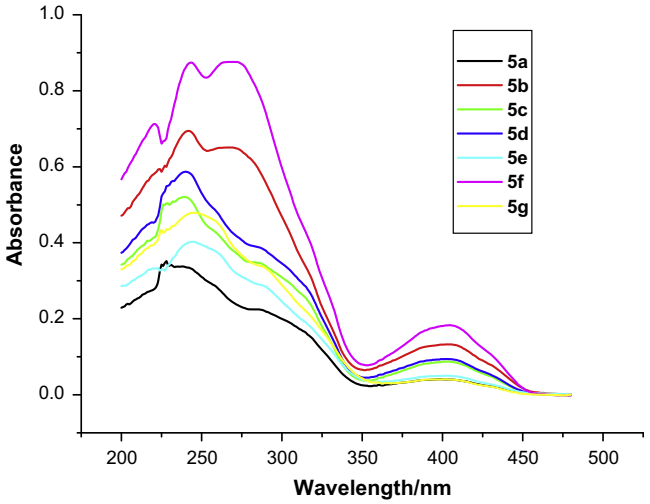


Fig. 2. UV–vis absorption spectra of compounds **5a–g** taken in dichloromethane ($c = 1 \times 10^{-5}$ M).

Table 1
The optical characteristics of compounds **5a–g**.

Compounds	λ_{max}		E (mol^{-1} cm^{-1} L)	
5a	400	238	4100	33,800
5b	403	241	13,300	69,400
5c	403	239	8700	52,100
5d	402	240	9400	58,700
5e	400	244	5000	40,300
5f	404	244	18,100	87,400
5g	400	246	4100	47,900

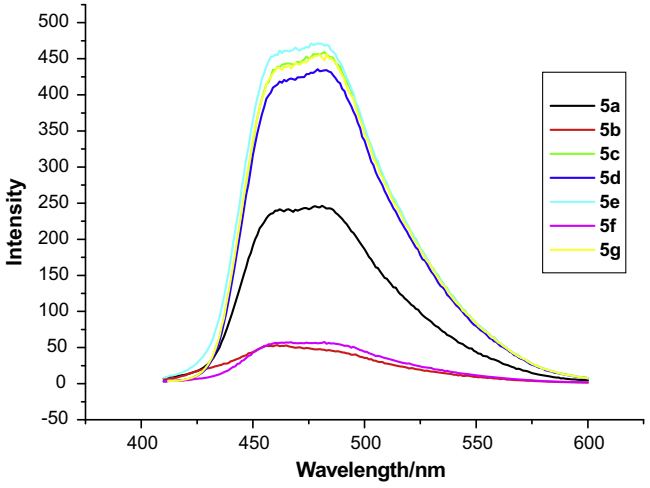


Fig. 3. Fluorescence spectra of the compounds **5a–g** in dichloromethane (10^{-6} M).

no absorption was observed beyond 450 nm. It was observed that compounds **5a–g** have similar absorption characteristics, with absorption peaks ranging from 400 to 404 nm, which are attributed to the π–π*transition of conjugate backbone. Comparing with imidazo[1,5-a]pyridinyl 1,3,4-oxadiazole reported in previous paper[31], compounds **5a–g** have red shifted about 20 nm, the reason being that the pyrazole which are linked to 1,3,4-oxadiazole can extend the π-conjugation system. Furthermore, the substituents in N-1 position of pyrazole moiety and para position of benzene moiety hardly influenced the absorption.

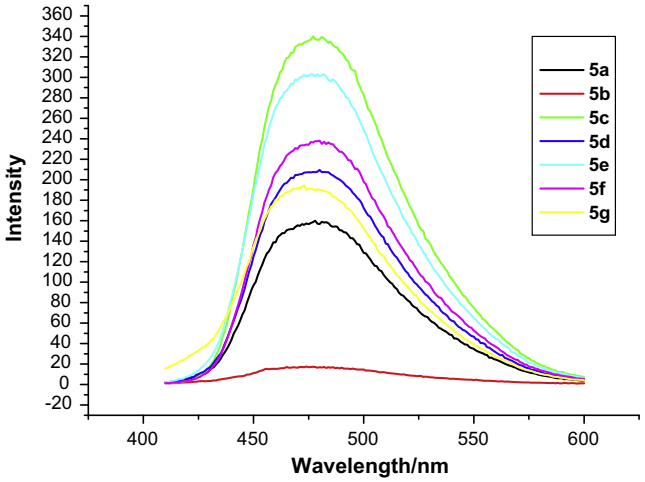


Fig. 4. Fluorescence spectra of the compounds **5a–g** in acetonitrile (10^{-6} M).

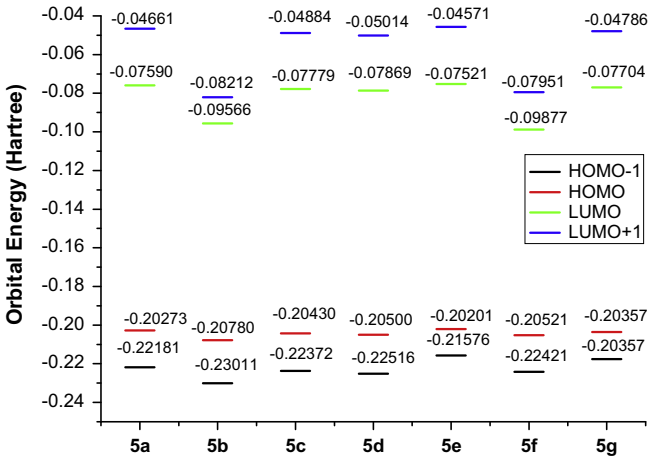


Fig. 6. Molecular orbital energy levels of compounds **5a–g**.

Table 2
Data of fluorescence spectra of compounds **5a–g**.

Compounds	λ_{em} (nm)		Stoke's shift (nm)
	CH_2Cl_2	CH_3CN	CH_2Cl_2
5a	478	478	78
5b	461	477	58
5c	482	477	79
5d	483	480	81
5e	480	479	80
5f	467	480	53
5g	483	479	83

Fluorescence

Fluorescence spectral characteristics of the compounds **5a–g** in dichloromethane and acetonitrile solution with the concentration of 10^{-6} M were investigated. From Figs. 3 and 4 and Table 2 it can be found that the maximum emission spectra of compounds in two different solvents are also hardly dependent on the groups in N-1 position of pyrazole moiety and para position of benzene moiety. The intensity of fluorescence is correlated with substituents on para position of N-1 benzene moiety. Generally, fluorescence intensity of **5b** and **5f** with nitro group on para position of N-1 benzene moiety is weaker than others in two different solvents.

Theoretical calculation

To enhance our understanding of the relationship between molecular structures and the electronic spectra of these new 1,3,4-oxadiazoles derivatives, we carried out structure optimization and molecular orbital (MO) calculations on compound **5a–g** based on a simplified model with density functional theory (DFT) on the level of B3LYP. The minimized structures are shown in Fig. 5 (Supplementary data) and the calculated molecular orbital (HOMO and LUMO) energies are shown in Fig. 6.

The minimized structures of compound **5** revealed that imidazo[1,5-a]pyridine ring present a coplanar conformation with the 1,3,4-oxadiazole ring and the pyrazole ring while the benzene ring attached to N-1 does not. This indicates that imidazo[1,5-a]pyridine group conjugated with the 1,3,4-oxadiazole ring and the pyrazole ring efficiently through the single bond yet the benzene does not. This result is in accordance with the similar absorptions spectra of compound **5** with different substituents on the benzene ring.

The molecular energy levels of the frontier molecular orbital shown in Fig. 6 revealed that the introduction of electron-withdrawing group to benzene ring reduced the energy levels of HOMO and also reduced the energy levels of LUMO for the compound; therefore, the energy gap between HOMO and LUMO changed little. This corresponds well to the experimentally recorded similar absorption spectra of compound **5**.

The frontier molecular orbital maps of compound **5** in Fig. 7 (supplementary data) revealed that the contribution of the ben-

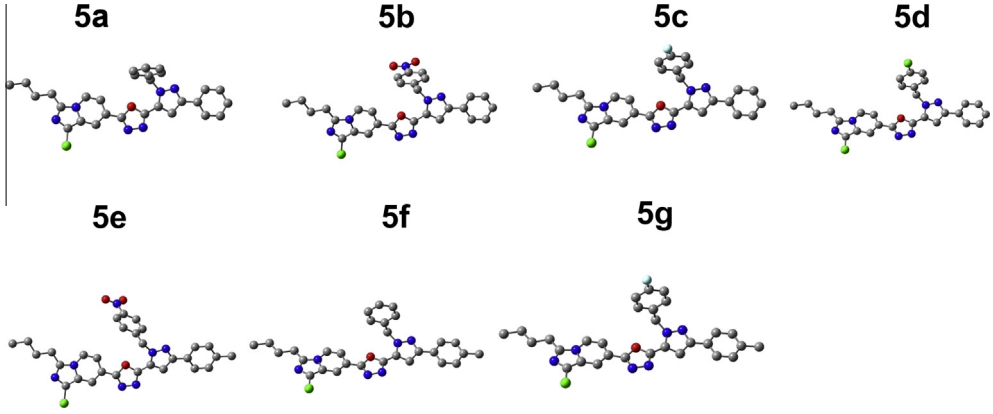


Fig. 5. The minimized structures of compounds **5a–g**.

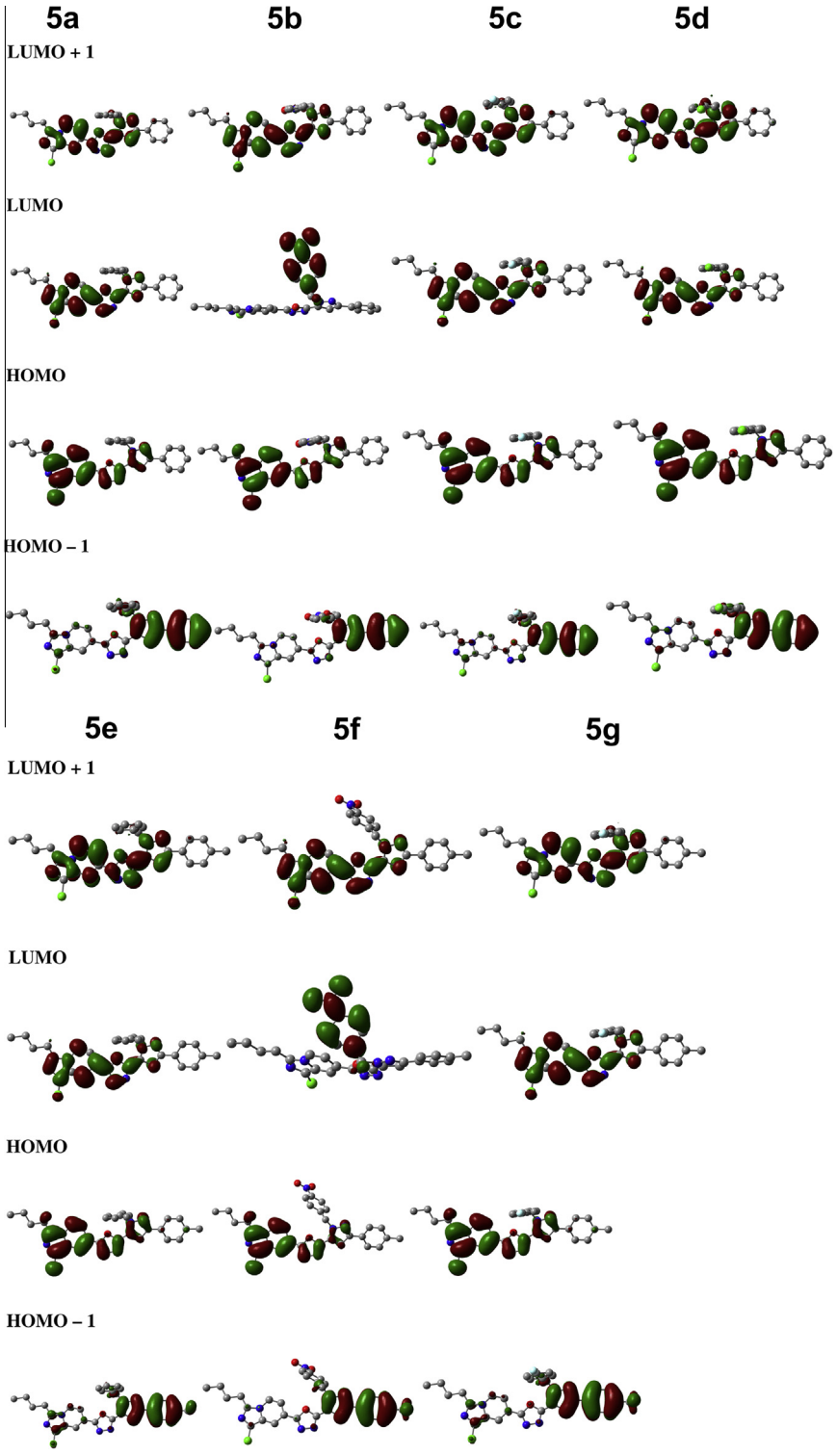


Fig. 7. Molecular orbital maps of compounds **5a–g**.

zene group to HOMO and LUMO is small. Therefore, the calculated results explain well the similar absorption maximum of compound **5** because of the little contribution of the benzene group to HOMO and LUMO.

Conclusion

In summary, a series of novel imidazo[1,5-a]pyridine and pyrazole linked 1,3,4-oxadiazole derivatives have been synthesized. The structures of the compounds obtained were determined by IR, ^{1}H NMR and HRMS spectra. Studies on the optical properties indicate that the substituents on benzene ring have no significant impact on the UV–vis absorption and fluorescence emission. Quantum calculation correlates well with their absorption and fluorescence emission.

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Appendix A. Supplementary material

Supplementary material associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2013.12.016.

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