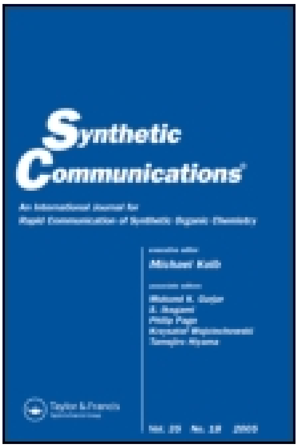

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Synthesis, Photochromism, and Effects of Metal Ions on Fluorescence of Dithienylethenes Containing Imidazo[2,1-a]isoquinoline

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SYNTHESIS, PHOTOCHROMISM, AND EFFECTS OF METAL IONS ON FLUORESCENCE OF DITHIENYLETHENES CONTAINING IMIDAZO[2,1-a] ISOQUINOLINE

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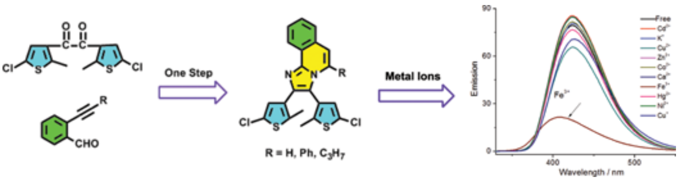
GRAPHICAL ABSTRACT



Abstract *Dithienylethene derivatives containing imidazo[2,1-a]isoquinolines were directly synthesized by treatment of the diketone with 2-ethynylbenzaldehyde in one pot. Their photochromism indicated that they can easily isomerize between the ring-open and ring-closed isomers upon irradiation with ultraviolet or visible light in* CH_2Cl_2*. At the same time, they display high selectivity toward* Fe^{3+} *by the fluorescence titration, such that the addition of* Fe^{3+} *can obviously suppress their fluorescence intensity.*

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Keywords Dithienylethene; effects of metal ions; imidazo[2,1-a]isoquinoline; photochromism

INTRODUCTION

Photo switches are some of the most attractive subjects in recent years because of their applications in molecular devices.[1,2] Photochromism is the reversible transformation between two isomers with different absorption spectra caused by alternating irradiation with ultraviolet (UV) and visible light. For example, azobenzene, spiropyran, and dithienylethene are common systems in organic photochromism.[3]

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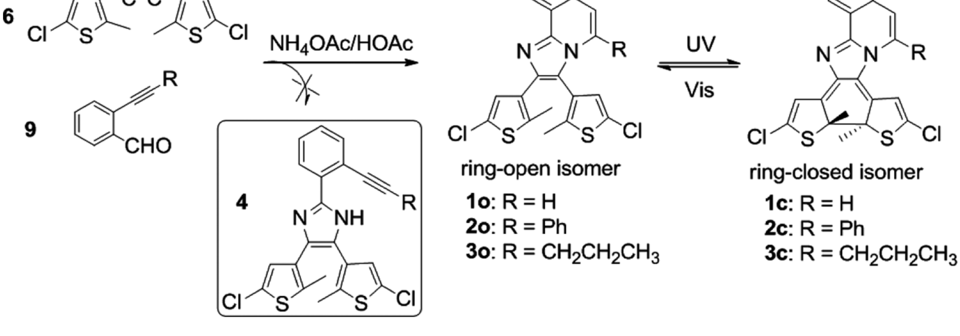
Among the various systems of photochromic compounds, dithienylethene derivatives with heterocyclic aryl rings, especially those bearing two thiophene or benzothiophene rings, are considered the most promising candidates for photoelectric applications because of the excellent thermal stability, remarkable fatigue resistance, and high sensitivity.[4] Moreover, they have been widely applied in optical memories.[5]

Imidazole is an important building block that was widely used in fields of medicine,[6] ionic liquids,[7] sensors,[8] and electronic and optical materials.[9] When imidazole was introduced into dithienylethene, it is expected to obtain dithienylethenes with excellent photochromism. Xiao et al.[10] and Yam et al.[11] have reported imidazole-substituted dithienylethenes. Recently we reported a series of imidazole-substituted dithienylethenes and found that the introduction of imidazole can affect their fluorescence.[12] Moreover, imidazole as a conjugated bridge has substituted cyclopentene in dithienylethene.[13] As we have mentioned, imidazole can be used as the building block of chemsensors. However, there are few reports describing the recognition of imidazole-based dithienylethenes.[14] Herein, we report dithienylethenes containing the imidazo[2,1-a]isoquinoline unit. In particular, we found that such a heterocycle can be formed in one pot by condensation of diketone with 2-ethynylbenzaldehyde. The photochromism showed that they can easily isomerize between the ring-open and ring-closed isomers. Furthermore, they display very high selectivity toward Fe^{3+}, such that the addition of Fe^{3+} obviously suppresses their fluorescence intensity.

RESULTS AND DISCUSSION

Scheme 1 shows the synthesis of dithienylethenes **1**–**3**. 2-Methylthiophene was used as the starting material, and after chlorination and Friedel–Crafts reaction, diketone was obtained. Diketone was treated with aldehyde in the presence of ammonium acetate and acetic acid to give dithienylethenes **1**–**3**. According to previous literature, we found imidazole-substituted phenylacetylene was cyclized in the catalysis of palladium to get imidazo[2,1-a]isoquinoline cycle in usual.[15] For example, Larock had reported 2,3-disubstituted indoles by the reaction of iodoanilines with internal



Scheme 1. Synthesis and photoisomerization of dithienylethenes **1**–**3**.

Table 1. Absorption maxima and quantum yields of dithienylethenes **1o–3o** in CH_2Cl_2 at 2.0×10^{-5} mol/L

Compound	$\lambda_{o,max}{}^{Abs}$/nm[a] ($\varepsilon \times 10^{-3}$/L mol^{-1} cm^{-1})	$\lambda_{c,max}{}^{Abs}$/nm[b] ($\varepsilon \times 10^{-4}$/L mol^{-1} cm^{-1})	$\varphi_{o\text{-}c}$[c]	$\varphi_{c\text{-}o}$[c]	φ_f[d]
1o	264 (2.20)	622 (3.12)	0.540	0.067	0.012
2o	276 (0.95)	612 (4.03)	0.192	0.020	0.018
3o	284 (4.00)	582 (4.30)	0.243	0.028	0.013

[a] Absorption maxima of ring-open isomers.
[b] Absorption maxima of ring-closed isomer.
[c] Quantum yields of ring-open ($\varphi_{c\text{-}o}$) and ring-closed isomers ($\varphi_{o\text{-}c}$).
[d] Fluorescence quantum yields of **1**–**3** before UV irradiation.

acetylenes. Pd-catalyzed conversion of alkyne has also been used in the synthesis of pyrroles, tryptophans, benzofurans, benzopyrans, furopyridines, benzimidazo[2,1-a] isoquinolines.[16] Imidazo[5,1-a]isoquinoline via a sequential alkyl–aryl and aryl–heteroaryl bond formation on the imidazole ring has been reported by Jafarpour and Ashtiani.[17] In addition, gold(I) can catalyze such cyclization.[18] Fujii et al. reported a novel copper-catalyzed synthesis of 3-(aminomethyl)isoquinoline polycyclic compounds.[19] However, it is surprising that the dithienylethene imidazo[2,1-a] isoquinolines were synthesized by diketone and aldehyde in one step, in which we did not use the expensive catalysis of palladium to get imidazo[2,1-a]isoquinoline cycle as usual. At the same time, we did not observe the existence of intermediate **4**. Therefore, it is expected that such a one-pot method will be widely applied in the synthesis of functional imidazo[2,1-a]isoquinolines in the future.

The UV/vis absorption spectra of **1o**–**3o** were measured at room temperature in CH_2Cl_2. As shown in Table 1, the absorption maxima were observed at 270–280 nm because of similar conjugation, as a result of a $\pi \rightarrow \pi^*$ transition.[20] Subsequently, photochromism of **1o**–**3o** were measured at room temperature in CH_2Cl_2. For example, Fig. 1 shows the changes in the absorption spectra of **3o** in CH_2Cl_2, induced by irradiation with UV and visible light ($\lambda > 450$ nm). The absorption maximum **3o**

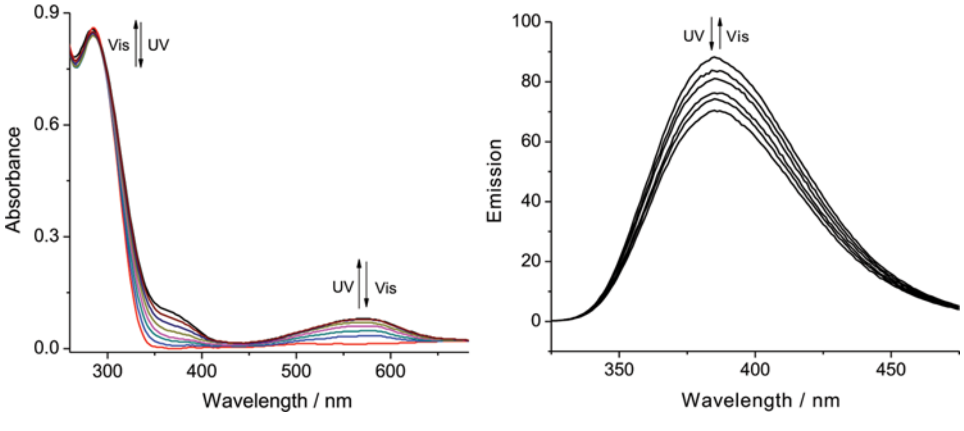


Figure 1. (left) Absorption spectral change of **3o**; (right) emission intensity changes of compound **3**. (Figure is provided in color online.)

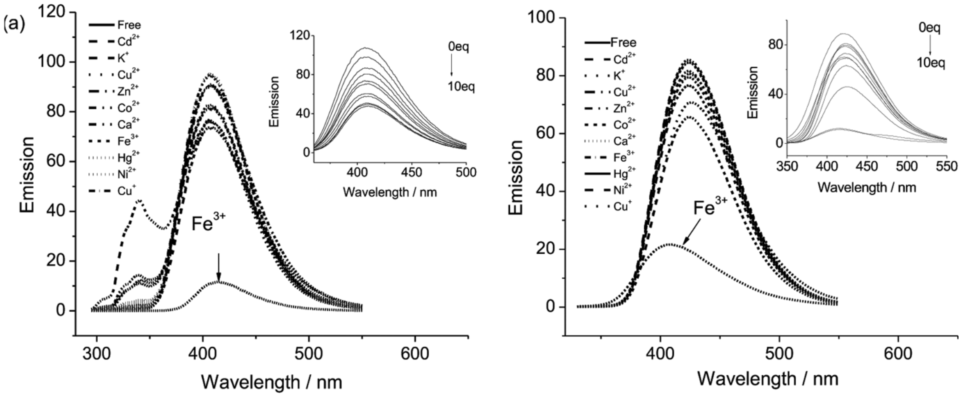


Figure 2. Fluorescence responses of **1o** (left) and **3o** (right) undergo different metal ions (10 eq.) in acetonitrile (2.0×10^{-5} mol/L) solution. (Inset) The fluorescence response changes by the addition of Fe^{3+} from 0 to 10 equiv.

was observed at 284 nm. After irradiation with 365-nm UV light, the colorless CH_2Cl_2 solution reverted to purple immediately. At the same time, a new absorption band was observed at 582 nm because of the formation of ring-closed isomer **3c** and the delocalization of π-electrons over both thiophene rings.[21] Subsequently, upon irradiation with visible light, the decrease of an absorption band was observed at 582 nm along with the increase of the absorption band at 284 nm. Similar results were observed when the solutions of **1** and **2** in CH_2Cl_2 were irradiation with UV light or visible light. These results indicated that dithienylethenes **1**–**3** can easily isomerize between the ring-open and ring-closed isomers upon irradiation with UVlight or visible light in solution. Furthermore, such heterocycle unit can substitute cyclopentene and imidazole bridge as conjugated bridge in dithienylethene.[10–12] Therefore, decoration on the bridge as a strategy will be used to synthesize dithienylethenes with novel structures and excellent properties in the future. The relative quantum yields of cyclization reaction ($\varphi_{o\text{-}c}$) and cycloreversion reaction ($\varphi_{c\text{-}o}$) were determined in CH_2Cl_2 by comparing reaction yield with 1,2-bis(2-methyl-5-phenyl-3-thienyl) perfluorocyclopentene.[16] The data are summarized in Table 1, in which we found that the cyclization reaction ($\varphi_{o\text{-}c}$) and cycloreversion reaction ($\varphi_{c\text{-}o}$) of **1** were much greater than **2** and **3**, respectively. The results indicated that the introduction of substituted groups such as phenyl and propyl groups in imidazo[2,1-a]isoquinoline heterocycle can decrease the cyclization reaction ($\varphi_{o\text{-}c}$) and cycloreversion reaction ($\varphi_{c\text{-}o}$) in such a system.

Subsequently, we investigated the fluorescence spectral changes in CH_2Cl_2 upon irradiation with UV light at room temperature. As shown in Fig. 1, when dithienylethene **3o** was excited at 284-nm light, the fluorescence maxima of the ring-open isomer was observed at 380 nm. However, the fluorescence intensity decreased significantly after irradiation with 365-nm light because of the formation of the ring-closed isomer. Similar results were also observed for **1o** and **2o** upon irradiation with UV light. The fluorescence quantum yields were measured as shown in Table 1. Subsequently, we investigated the changes of fluorescence properties of **1**–**3** in acetonitrile at room temperature when different metal cations (K^+, Ca^{2+}, Cu^{2+}, Zn^{2+}, Co^{2+},

Ni^{2+}, Cd^{2+}, Fe^{3+}, and Hg^{2+}) were added. As shown in Fig. 2, the fluorescence intensity of **1** decreased significantly in the presence of the Fe^{3+} ion, whereas the other metal cations did not exhibit such a quenching effect under the same conditions, which indicated that the compound **1** displayed the "turn-off" fluorescence and high selectively toward Fe^{3+}. Moreover, the fluorescence quenching of the compounds upon the addition of Fe^{3+} may be attributed to a complex formed between **1** and Fe^{3+}, and there was a ligand-to-metal charge transfer (LMCT),[22] in which the electronic charge is transferred from the ligand to the coordinating metal. The Job plot for the binding between **1** and Fe^{3+} showed a 1:1 stoichiometry, and the binding constant ($K_b = 4.4 \times 10^4\,M^{-1}$) was estimated from the change in the spectral intensities (Fig. 2).

Subsequently, to understand the effect of Fe^{3+} ion on the fluorescence properties of **1o** more clearly, we performed the titration experiment as shown in Fig. 2 (Inset). According to the titration experiment, we found that with the increase of Fe^{3+} ion equivalent, the fluorescence intensity decreased significantly. However, the quenching intensity slowly tended to be saturated when the amount of Fe^{3+} ion is more than 7 equivalents. Similar results were obtained when the Fe^{3+} ion was added to the solution of **2** and **3** in acetonitrile. Formation of 1:1 complexes formed among **2, 3** and Fe^{3+} were determined from the emission spectra changes, and their binding constants were $K_b = 5.3 \times 10^4$ and $K_b = 3.8 \times 10^4\,M^{-1}$. According to the experiments, we found that dithienylethenes **1–3** displayed high selectivity to Fe^{3+}, such that the addition of Fe^{3+} can obviously suppress their fluorescence intensity.

In conclusion, a class of dithienylethenes containing imidazo[2,1-a]isoquinolines was directly synthesized in one pot by condensation and cyclization, when the thiophene-substituted diketone and 2-ethynylbenzaldehyde were used as starting materials. Their photochromism were investigated in CH_2Cl_2 solution. The results indicated that they can easily isomerize between the ring-open and ring-closed isomers upon irradiation with UV light or visible light. Furthermore, we investigated their recognition properties toward metal cations, which indicated that they display high selectivity to Fe^{3+}, such that the addition of Fe^{3+} can obviously suppress their fluorescence intensity. In our studies, the decoration of the bridge will be probably used as a strategy to synthesize the multifunctional photochromic dithienylethenes.

EXPERIMENTAL

All manipulations were carried out under an argon atmosphere using standard Schlenk techniques, unless otherwise stated. Tetrahydrofuran (THF) was distilled under nitrogen from sodium-benzophenone. 1,2-Bis(5-chloro-2-methylthiophen-3-yl)ethane-1,2-dione **6** was prepared by literature methods.[15] Different acetylene-substituted benzaldehydes **9** were prepared by modified procedures of reported methods.[16] All other starting materials were obtained commercially as analytical-grade and used without further purification. The relative quantum yields were determined by comparing reaction yield with the known yield of the compound 1,2-bis(2-methyl-5-phenyl-3-thienyl)perfluorocyclopentene.[16] Elemental analyses (C, H, N) were performed by the Microanalytical Services, College of Chemistry, CCNU. ^1H and ^{13}C NMR spectra were collected on American Varian Mercury Plus 400 spectrometer (400 MHz). ^1H and ^{13}C NMR chemical shifts are relative to TMS. UV-vis spectra were obtained on U-3310 UV spectrophotometer. Fluorescence spectra were taken on a Fluoromax-P luminescence spectrometer (Horiba Jobin Yvon Inc.).

General Synthetic Method

Compound **9** (1.0 mmol) and ammonium acetate (0.62 g, 8.0 mmol) were added to a solution of compound **6** (0.32 g, 1.0 mmol) in acetic acid (15 mL), and the mixture was refluxed until the staring material disappeared [thin-layer chromatographic (TLC) detection]. The mixture solution was cooled to room temperature and then slowly poured into $NaHCO_3$ solution (10%, 100 mL), and the product was extracted with CH_2Cl_2 (3×50 mL). The combined organic phase was washed with water and dried with Na_2SO_4. After evaporation of the solvent, the crude product was purified by flash column chromatography (petroleum ether/dichloromethane = 1:1) to afford target compounds **1o–3o** in 19–51% yields.

Compound 1o

Yield 49%. ^1H NMR (400 MHz, $CDCl_3$): $\delta = 2.00$ (s, 3H, CH_3), 2.31 (s, 3H, CH_3), 6.82 (s, 1H, thiophene-H), 6.84 (s, 1H, thiophene-H), 6.99 (d, $J = 7.6$ Hz, 1H, ethylene-H), 7.51–7.67 (m, 4H, Ar-H), 8.69 (d, $J = 7.6$ Hz, 1H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 13.7$ (s, CH_3), 14.2 (s, CH_3), 113.0, 117.2, 120.8, 123.2, 123.5, 124.9, 125.2, 126.7, 126.8, 127.2, 127.3, 128.1, 128.2, 129.3, 130.5, 135.5, 137.7, 138.6, 142.5 (s, Ar, thiophene). MS (m/z): 428.05 $[M]^+$. Anal. calcd for $C_{21}H_{14}Cl_2N_2S_2$: C, 58.74; H, 3.29; N, 6.52. Found: C, 58.62; H, 3.21; N, 6.64.

Compound 2o

Yield 51%. ^1H NMR (400 MHz, $CDCl_3$): $\delta = 1.88$ (s, 3H, CH_3), 2.36 (s, 3H, CH_3), 5.99 (s, 1H, thiophene-H), 6.57 (s, 1H, thiophene-H), 6.97 (s, 1H, ethylene-H), 7.01–7.04 (m, 2H, Ar-H), 7.25–7.37 (m, 3H, Ar-H), 7.60–7.72 (m, 3H, Ar-H), 8.77 (d, $J = 7.6$ Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 13.8$ (s, CH_3), 14.4 (s, CH_3), 115.6, 118.2, 123.0, 123.5, 125.1, 125.4, 126.5, 127.0, 127.4, 127.8, 128.0, 128.6, 128.9, 129.4, 130.6, 133.7, 136.0, 136.7, 136.9, 139.0, 144.1 (s, Ar, thiophene). MS (m/z): 504.11 $[M]^+$. Anal. calcd. for $C_{27}H_{18}Cl_2N_2S_2$: C, 63.90; H, 3.97; N, 5.52. Found: C, 63.99; H, 3.91; N, 5.38.

Compound 3o

Yield 19%. ^1H NMR (400 MHz, $CDCl_3$): $\delta = 0.75$ (t, $J_1 = 7.2$ Hz, $J_2 = 7.2$ Hz 3H, CH_3), 1.41–1.49 (m, 2H, CH_2), 2.16 (s, 3H, CH_3), 2.46 (s, 3H, CH_3), 2.50–2.68 (m, 2H, CH_2), 6.64 (s, 1H, thiophene-H), 6.83 (s, 1H, ethylene-H), 6.87 (s, 1H, thiophene-H), 7.53–7.65 (m, 3H, Ar-H), 8.70 (d, $J = 7.2$ Hz, 1H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 13.6$ (s, CH_3), 13.8 (s, CH_3), 14.6 (s, CH_3), 21.9 (s, CH_2), 34.4 (s, CH_2), 112.4, 117.4, 122.6, 123.5, 124.9, 125.8, 126.2, 127.0, 127.3, 127.7, 128.4, 129.2, 129.6, 130.2, 135.3, 136.4, 137.7, 139.2, 144.5 (s, Ar, thiophene). MS (m/z): 473.89 $[M]^+$. Anal. calcd. for $C_{24}H_{22}Cl_2N_2S_2$: C, 60.88; H, 4.68; N, 5.92. Found: C, 60.68; H, 4.79; N, 5.71.

Additional details can be found online in the Supporting Information.

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