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Graphical Abstract

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Photochromism of new unsymmetrical diarylethenes with an indazole moiety

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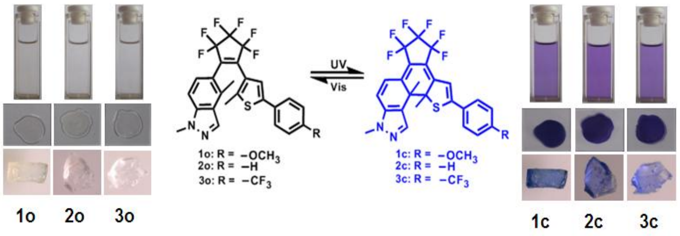

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Photochromism of new unsymmetrical diarylethenes with an indazole moiety

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A new class of photochromic diarylethenes with an indazole moiety has been synthesized firstly, and their photochromic and fluorescence properties have been investigated. The indazole moiety was connected directly to the central cyclopentene ring as one heteroaryl unit and availably participated in the photoinduced cyclization reaction in solution, amorphorous film, as well as in the crystalline phase. These diarylethenes exhibited excellent photochromism with good thermal stability and remarkable fatigue resistance. They also functioned as a notable fluorescence switch in both solution and amorphorous films. In addition, the different substituents at the *para*-position of the terminal benzene ring had a significant effect on their properties: the electron-donating methoxy group could enhance the quantum yields of cyclization and cycloreversion and fluorescence quantum yield, but the electron-withdrawing trifluoromethyl had opposite effect.[1]

Keyword: Photochromism; Diarylethenes; Indazole moiety; Substituent effect; Single crystal.

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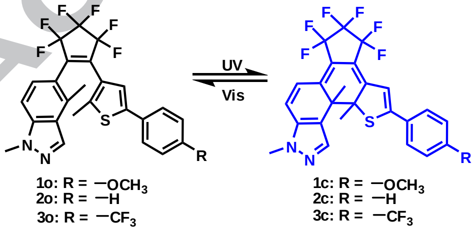
Research on photochromic compounds has received much attention because of their potential application to optoelectronics devices.[1] Among the wide variety of photochromic compounds (azobenzenes,[2] spiropyrans,[3] fulgides,[4] etc.), diarylethenes are one of most promising thermally irreversible photochromic molecules developed by Irie et al.[5] and Lehn et al.[6] These compounds are characterized by efficient photoisomerization with excellent thermal stability, remarkable fatigue resistance, high sensitivity, fast response, and non-destructive readout capability.[7]

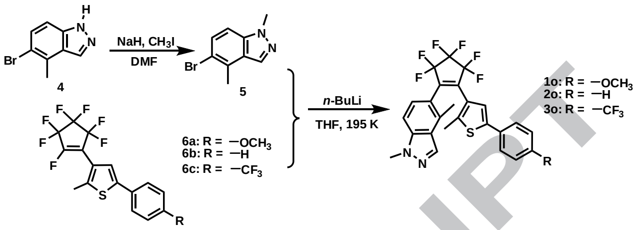
Design and synthesis of new diarylethenes with different heteroaryl moieties have become a hot topic in the research field because the heteroaryl moieties can effectively modulate their photochromic characteristics.[8] For example, diarylethenes with thiophene or benzothiophene moieties exhibit excellent thermal stability and the ones with indole units show effective fluorescent photoswitches.[9] However, diarylethenes with pyrrole rings are thermally unstable and can return to the open-ring isomers even in the dark.[10] In addition, functional substituents also have important effects on the photochromic properties of diarylethenes, such as the absorption maxima, absorption molar coefficients, and the quantum yields of cyclization and cycloreversion. [11] To date, the hexatriene backbones of the reported diarylethene systems are mainly confined to the five-membered heteroaryl rings,[12] with just a few reports concerning other six-membered heteroaryl moieties, such as benzene,[13] naphthalene,[14,11b] pyridine,[15] and pyrimidine.[16] Indazole is an attractive heteroaryl unit due to its low aromatic stabilization energy and a broad range of biological activities.[17] However, photochromic diarylethene derivatives with a six-membered heteroaryl indazole moiety have not hitherto been reported.

In this paper, we designed and synthesized a new class of diarylethenes with both indazole and thiophene moieties (**1o–3o**). The indazole moiety is connected directly to the central ethene unit as a six-membered heteroaryl moiety and participates in the photochromic hexatriene–cyclohexadiene reaction (Scheme 1). All of these diarylethenes exhibited thermally irreversible photochormism in solution, PMMA films, and even in the single crystalline phase. The photochromism of diarylethenes **1o–3o** is shown in Scheme 1.



Scheme 1. Photochromism of diarylethenes **1o–3o**.

The synthetic route for diarylethenes **1o–3o** is shown in Scheme 2. *N*-methylation of 5-bromo-4-methyl-1*H*-indazole and coupling with iodomethane gave compound **5**.[18] Compound **5** was lithiated and then separately coupled with **6a–c**[16] to give diarylethenes **1o–3o**. The structures of **1o–3o** were confirmed by elemental analysis, NMR, IR, and MS. The detailed experimental procedures and data are summarized in the Supplementary Information (SI).



Scheme 2. Synthetic route to diarylethenes **1o–3o**.

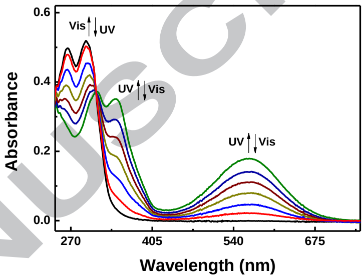


Fig. 1. Absorption spectral changes of **1** upon alternating irradiation with UV and visible light in hexane (2.0 × 10^{-5} mol L^{-1}).

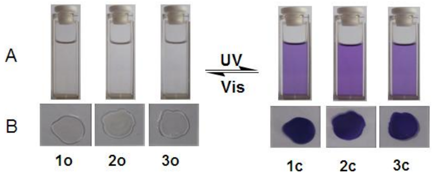


Fig. 2. The color changes of **1–3** by photoirradiation in different media at room temperature: (A) in hexane, (B) in PMMA films.

The photochromic properties of **1–3** induced by photoirradiation were investigated in both hexane (2.0 × 10^{-5} mol L^{-1}) and PMMA films (10%, w/w). The absorption spectral changes of diarylethene **1** in hexane are shown in Fig. 1, and the color changes of **1–3** by photoirradiation are shown in Fig. 2. In hexane, **1o** showed a sharp absorption peak at 295 nm (ε, 4.89 × 10^4 L mol^{-1} cm^{-1}) due to $\pi \rightarrow \pi^*$ transition.[19] Upon irradiation with 297 nm light, a new visible absorption band centered at 566 nm (ε, 8.95 × 10^3 L mol^{-1} cm^{-1}) was observed with a notable color change from colorless to violet due to the formation of the closed-ring isomer **1c**. Reversely, the colored solution turned colorless upon irradiation with visible light of appropriate wavelength ($\lambda > 500$ nm). Diarylethenes **2** and **3** exhibited similar photochromism as observed for **1** in hexane, and their absorption maxima were observed at 560 nm for **2c** and 558 nm for **3c** (Fig. S1). In the photostationary state, the isosbestic points were observed at 312 nm for **1**, 301 nm for **2**, and 320 nm for **3**. Moreover, the photoconversion ratios of **1–3** were measured by HPLC analysis in the photostationary state, with the value of 53% for **1**, 58% for **2**, and 39% for **3** (Fig. S2). In PMMA films, **1–3** also showed similar photochromism as observed in hexane, and their spectral changes are shown in Fig. S3. Upon irradiation with UV light, the colors of the diarylethene/PMMA films **1–3** changed from colorless to violet, with the appearance of a new broad absorption band centered at 579 nm for **1c**, 568 nm for **2c**, and 576 nm for **3c**. Upon irradiation with appropriate visible light, these colored films reverted to colorless due to the reproduction of **1o–3o**. Compared to those in hexane, the absorption maxima of the closed-ring isomers **1c–3c** in PMMA films exhibited a notable bathochromic shift, with a redshift value of 13 nm for **1c**, 8 nm for **2c**, and 18 nm for **3c**. The polar effect of the polymer matrix and the stabilization of molecular in solid state may be resulted in this redshift phenomenon.[20]

Table 1. Absorption spectral properties of diarylethenes **1–3** in hexane (2.0 × 10^{-5} mol L^{-1}) and PMMA films (10%, w/w) at room temperature.

Compd	$\lambda_{o,max}$/nm[a] (ε/L mol^{-1} cm^{-1})		$\lambda_{c,max}$/nm[b] (ε/L mol^{-1} cm^{-1})		Φ[c]		PR[d] (%)
	hexane	PMMA film	hexane	PMMA film	$\Phi_{o\text{-}c}$	$\Phi_{c\text{-}o}$	
1	295 (4.89 × 10^4)	298	566 (8.95 × 10^3)	579	0.34	0.16	53
2	295 (3.76 × 10^4)	299	560 (7.33 × 10^3)	568	0.27	0.10	58
3	301 (6.59 × 10^4)	301	558 (9.04 × 10^3)	576	0.24	0.07	39

[a] The absorption maxima of the open-ring isomers.
[b] The absorption maxima of the closed-ring isomers.
[c] Quantum yields of cyclization ($\Phi_{o\text{-}c}$) and cycloreversion ($\Phi_{c\text{-}o}$).
[d] Photoconversion ratios in the photostationary state.

The absorption spectral parameters of **1–3** in both hexane and PMMA films are listed in Table 1. These data showed that different substituents at *para*-position of the terminal benzene ring had significant effects on the photochromic features of **1–3**, including the absorption maxima, molar absorption coefficients, and quantum yields of cyclization and cycloreversion. Among the three diarylethenes, the molar absorption coefficients of the two isomers of the unsubstituted parent compound **2** are the smallest. Replacing the hydrogen atom at the *para*-position of the terminal benzene ring with either an electron-donating methoxy group (compound **1**) or with an electron-withdrawing trifluoromethyl group (compound **3**) resulted in bigger molar absorption coefficients. The result was consistent with that of diarylethenes with a pyridine,[15] pyrimidine,[16] thiazole,[12c] or isoxazole moiety.[21] As shown in Table 1, the quantum yields of unsubstituted parent diarylethene **2** were 0.27 for the cyclization reaction and 0.10 for the cycloreversion reaction. When the electron-donating methoxy group was substituted at the *para*-position of the terminal benzene ring, the quantum yields of cyclization and cycloreversion (compound **1**) increased significantly. However, a reverse trend was observed when the electron-withdrawing trifluoromethyl group was introduced at the same position (compound **3**). The result was in good agreement with that of the reported thiazole-containing diarylethenes.[12c] In addition, the absorption maxima of the closed-ring isomers **1c–3c** were observed at the range of 558–579 nm, which were much longer than those of analogous diarylethenes with a benzene[13] or pyrimidine moiety.[16] The results indicated that the indazole moiety could be effective to shift the absorption maximum of diarylethene to a longer wavelength.

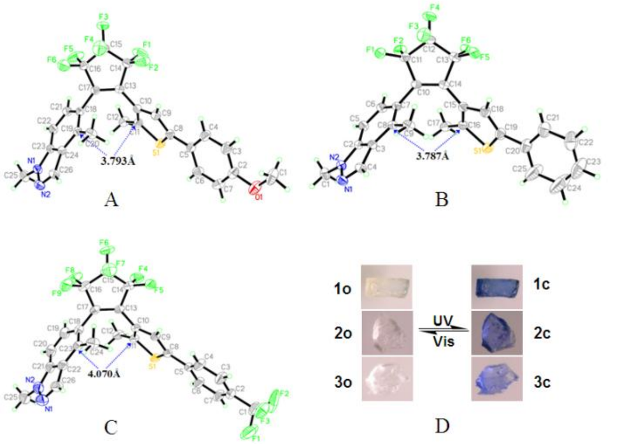
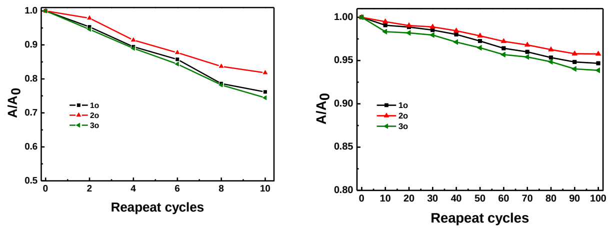


Fig. 3. ORTEP drawings of crystals and color changes of **1o–3o**: (A) **1o**, (B) **2o**, (C) **3o**, (D) photographs demonstrating their photochromic processes in the crystalline phase.

To know better the relation between the conformation and the photochromic behaviors of these diarylethenes in the crystalline phase, the structural conformations of the crystals of **1o–3o** were provided by X-ray crystallographic analysis. Their ORTEP drawings and color changes induced by photoirradiation are shown in Fig. 3. Their packing views along the x direction are shown in Fig. 4S, and the X-ray crystallographic analysis data are listed in Tables S1 and S2. The three indazole-containing diarylethenes crystallized with photoactive *anti*-parallel conformations in the crystalline phase, and the distances between the two reactive carbon atoms are 3.793 Å for **1o** (C11…C19), 3.787 Å for **2o** (C8…C16), and 4.070 Å for **3o** (C11…C23). Therefore, they could be expected to undergo photochromism in the single crystalline phase.[22] As expected, the crystals of **1o–3o** display favourable photochromism by photoirradiation in the single crystalline phase. Upon irradiation with 297 nm light, the colorless crystal of **1o**–**3o** turned violet, and the colored crystals reverted to colorless upon irradiation with visible light (λ > 500 nm). The result is consistent with that of the pyridine-containing diarylethenes,[15] but it is contrary to that of naphthalene-containing diarylethenes.[14]

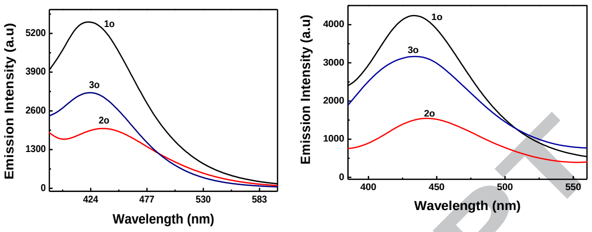
The thermal stability and fatigue resistance of photochromic compounds are crucial factors for practical applications in optical devices.[23] The thermal stability of the open-ring and closed-ring isomers of **1**–**3** was evaluated in hexane by UV–vis spectroscopy both at room temperature and at 343 K. Storing these solutions in hexane at room temperature in the dark and then exposing them to air for more than 10 days, we found no changes in the UV–vis spectra of diarylethenes **1**–**3**. At 343 K, diarylethenes **1**–**3** still showed excellent thermal stability for more than 2 hr. The results suggested that the indazole-containing diarylethenes had good thermal stability at both room temperature and higher temperature. The fatigue resistance of **1–3** was tested in both hexane and PMMA films by alternative irradiation with UV and visible light in air at room temperature, as shown in Fig. 4. In hexane, the coloration and decoloration cycles of **1–3** were repeated 10 times with the degradation of 24% for **1c**, 18% for **2c**, and 25% for **3c**, respectively. The relatively large degradation may result from the formation of epoxides.[12b] In PMMA films, the fatigue resistance of **1–3** enhanced notably, as compared to that in solution. After 100 cycles, the degradation percentages of **1–3** in PMMA films were determined to be 5% for **1c**, 4% for **2c**, and 6% for **3c**. This remarkable improvement may result from suppression of O_2 diffusion in the solid medium.[24] Compared to diarylethenes with a pyrimidine moiety,[16] the fatigue resistance of **1–3** was much better in both solution and PMMA films. But their fatigue resistance was much weaker that that of the diarylethenes with a naphthalene moiety.[14]



(A) (B)

Fig. 4. Fatigue resistance of **1–3** in hexane and in PMMA films in air atmosphere at room temperature: (A) in hexane, (B) in PMMA films. Initial absorptance of the sample was fixed to 1.0.

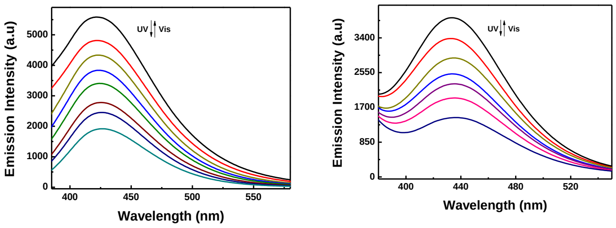
The fluorescence switching properties based on the photochromism of diarylethenes have attracted much attention because of their potential applications in molecularscale optoelectronics, ion-sensors, and digital photoswitches.[25] The fluorescence spectra of diarylethenes **1o–3o** both in hexane (2.0×10^{-5} mol L^{-1}) and in PMMA films (10%, w/w) were measured at room temperature, as shown in Fig. 5. In hexane, the emission peaks were observed at 463 nm for **1o**, 429 nm for **2o**, and 372 nm for **3o** when excited at 300 nm, and were observed at 469 nm (λ_{ex}, 319 nm) for **1o**, 442 nm (λ_{ex}, 300 nm) for **2o**, and 418 nm (λ_{ex}, 291 nm) for **3o** in PMMA films. Compared to those in hexane, the emission peaks of **1o–3o** showed a notable bathochromic shift in PMMA films, with a redshift value of 15 nm for **1**, 17 nm for **2**, and 12 nm for **3**. Compared to the unsubstituted parent compound **2o**, either the electron-donating methoxy group of **1o** or the trifluoromethyl group of **3o** shifted the emission peak to a shorter wavelength and greatly increased the emission intensity. The fluorescence quantum yields of diarylethenes **1o**–**3o** were measured by Absolute PL quantum yield spectrometer C11347, with the values of 0.014 for **1o**, 0.010 for **2o**, and 0.011 for **3o**, which were larger than those of the isoxazole-containing diarylethenes.[21] In addition, the electron-donating methoxy group could enhance the fluorescence quantum yield of the diarylethenes with an indazole moiety to a certain extent. The result is agreed with those of diarylethenes with a pyrimidine moiety,[16] but different from those of diarylethenes with a naphthalene or pyridine moiety, whose fluorescence quantum yields enhanced with the increase of electron-withdrawing ability of the substituents.[14,15]



(A) (B)

Fig. 5. Emission spectra of diarylethenes **1**–**3** in hexane and PMMA films at room temperature: (A) in hexane (2.0×10^{-5} mol L^{-1}), (B) in PMMA films (10%, w/w).

As has been observed for most of the reported diarylethenes,[26] **1–3** show notable fluorescence switches upon photoirradiation in both hexane and PMMA films. Fig. 6 shows the fluorescence spectral changes of **1** by alternating irradiation with UV and visible light in hexane and a PMMA film. Upon irradiation with 297 nm UV light, its emission intensity was quenched to ca. 34% in hexane and 37% in a PMMA film when reached the photostationary state. Back irradiation with visible light restored the original emission spectrum due to the formation of the open-ring isomer **1o**. Similarly, the emission intensity of **2** and **3** was quenched ca. 26% and 21% in hexane, and 41% for **2** and 44% for **3** in PMMA films, respectively (Fig. S5 and S6). The result exhibited that the fluorescent modulation efficiency of **1–3** in hexane was much larger than that in PMMA films. The result is in agreement with that of diarylethenes with an pyrimidine[16] or pyrrole moiety,[27] but contrary to that of diarylethenes with a benzene moiety.[13] Therefore, the diarylethenes could be potentially used as photoswitchable devices, such as optical memory and photo-controllable switches.[25a,26a,28]



(A) (B)

Fig. 6. Fluorescence spectral changes of **1** by photoirradiation at room temperature: (A) in hexane (2.0×10^{-5} mol L^{-1}), excited at 300 nm, (B) in PMMA films (10%, w/w), excited at 319 nm.

In summary, three new unsymmetrical diarylethenes with an indazole moietiy were synthesized, and their structures were determined by single-crystal X-ray diffraction analysis. The diarylethenes exhibited favorable photochromism with good thermal stability and functioned as a notable fluorescence photoswitch in solution and PMMA films. The behaviors of the three indazole-containing diarylethenes were different from each other due to the substitution effects in the terminal benzene ring. The indazole moiety induced new photochromic properties differing from other diarylethenes with a six-membered aryl moiety reported so far. Our experimental results may be useful for design and synthesis of new photochromic diarylethenes based on different six-membered aryl moieties.

Acknowledgments

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