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Photochromism of a novel asymmetrical diarylethene with a (formyloxyethoxy)ethyl-linked naphthalimide moiety

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A novel asymmetrical diarylethene with a (formyloxyethoxy)ethyl-linked naphthalimide unit was synthesized, and its photochromic and fluorescence properties were systematically investigated in both solution and a poly(metyl methacrylate) film. The diarylethene showed significant photochromism and notable fluorescence switching properties upon irradiation with ultraviolet/visible light. Compared with the parent diarylethene, introduction of the naphthalimide moiety was effective to increase the molar absorption coefficient, the fluorescence quantum yield, and fluorescent modulation efficiency of the diarylethene. Copyright © 2014 John Wiley & Sons, Ltd.

Keywords: diarylethene; fluorescence switch; naphthalimide; photoirradiaton; photochromism

INTRODUCTION

Photochromic compounds have attracted tremendous attention because of their wide applications in optical memory media and photooptical switching devices.^[1,2] Among various types of photochromic compounds, diarylethenes with heterocyclic aryl rings are the most promising candidates for photoelectronic applications because of their excellent thermal stability, notable fatigue resistance, rapid response, and high reactivity in solid state.^[3–6]

To date, the molecular skeletons mainly focus on exploitation of the central ethene bridge and introduce heterocyclic aryl rings with different substituents. For instance, the central ethene bridge has been confined to a cyclopentene unit^[7] or its strong electron-withdrawing analogs, such as perfluorocyclopentene,^[8] maleic anhydride,^[9] and maleic imide.^[10,11] More recently, several groups have successfully synthesized novel diarylethenes with new rings as a central bridge to perform photochromism.^[12,13] Owing to the structural changes of diarylethenes upon photochromic reaction, numerous efforts have been undertaken for the

synthesis of diarylethene derivatives with a range of heteroaryl units, such as thiophene,^[14,15] benzothiophene,^[16] furan,^[17] benzofuran,^[18] thiazole,^[19] indole,^[20,21] pyrazole,^[22] and pyrrole rings.^[23]

In recent years, much effort has been focused on designing and synthesizing fluorescent diarylethene derivatives by introducing one or more fluorescent moieties.^[24–28] Among the fluorophores, a naphthalimide unit has attracted much research interest because of its unique photophysical properties.^[29,30] Its absorption and fluorescence emission spectra lie within the ultraviolet (UV) and visible (vis) regions, and the various photophysical properties can be easily tuned through skillful structural design. The versatility of the structure resulted from either the aromatic "naphthalene" moiety or the "N-imide site". Consequently, the 1,8-naphthalimide derivatives have been extensively used as strongly absorbing and colorful dyes in the field of anion recognition and sensing.^[31,32] Recently, 1,8-naphthalimide has been introduced to the diarylethene systems.^[33–35] For instance, Irie *et al.*^[33] developed the naphthalimide

as a donor in photochromic fluorescence resonance energy transfer (pcFRET); the fluorescent emission of the donor was modulated by cyclical transformations of the photochromic acceptor. Tian et al.^[34] reported novel diarylethenes containing naphthalimide as the center ethene bridge that exhibited considerably high cyclization quantum yield and good fatigue resistance. Yi et al.^[35] studied the switchable supramolecular self-assemblies on the basis of the interaction between a melamine group containing a photochromic diarylethene unit and naphthalimide derivate. Both the absorption and fluorescence spectra of the assembly can be reversibly switched by alternating UV/Vis light irradiation. The valuable achievements were helpful for us to explore the character of diarylethene derivatives labeled with a naphthalimide moiety. Moreover, benzofuran is a fascinating aryl because of its relative lower aromatic stabilization energy that can be potential to improve the thermal stability of the closed-ring isomers of diarylethenes.^[36] In this study, a novel diarylethene, 1-(2-methyl-3-benzofuryl)-2-{2-methyl-5-{4-[N-(2-(2-formyloxyethoxy)ethyl)naphthalimide] phenyl}-3-thienyl}perfluorocyclopentene (10), was synthesized. In the molecule, 2-methylbenzofuran is selected as one of the two aryl groups, and naphthalimide occupies a fluorescent group. In order to evaluate the effect of the naphthalimide on the photochromic behavior, 1-(2-methyl-3-benzofuryl)-2-[2methyl-5-(4-formylphenyl)-3-thienyl]perfluorocyclopentene (20) is synthesized as a model compound. Their photochromism and fluorescence in both solution and poly(metyl methacrylate) (PMMA) amorphous films were studied. The photochromic processes of diarylethenes 1 and 2 are shown in Scheme 1.

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Scheme 1. Photochromism of diarylethenes 1o and 2o

RESULTS AND DISCUSSION

Synthesis of diarylethene

The synthetic route to the target compound is shown in Scheme 2. Diarylethene **20** was prepared by the reported method.^[37] Compounds **3** and **5** were then synthesized by Jones oxidation^[38] and acylation reaction,^[39] respectively. Finally, diarylethene **10** was synthesized by the esterification reaction of **3** with **5**. The structure of diarylethene **10** was confirmed by mass spectrum, nuclear magnetic resonance (NMR) spectros-copy, elementary analysis, and infrared spectroscopy.

Photochromism

Diarylethenes **1** and **2** showed reversible color and absorption spectrum changes upon alternating irradiation with UV/Vis light

in both methanol $(2.0 \times 10^{-5} \text{ mol L}^{-1})$ and PMMA films. As shown in Fig. 1(A), the absorption maximum of compound **10** was observed at 317 nm (ε , 3.69 × 10⁴ L mol⁻¹ cm⁻¹) in methanol. Upon irradiation with 313 nm light, the colorless solution of 10 turned violet with the emergence of a new vis absorption band centered at 535 nm (ε , 1.84 × 10⁴ L mol⁻¹ cm⁻¹), which could be attributed to the closed-ring form 1c. The violet solution became colorless upon irradiation with vis light ($\lambda > 450$ nm), indicating that 1c returned to its initial form 10. The absorption spectral change was accompanied by a clear isosbestic point that was observed at 342 nm. In order to clarify the difference between the diarylethenes with or without the fluorescent dye, the photochro-

mism of **20** was studied under the same conditions. The absorption spectral changes of **20** induced by photoirradiation in methanol are shown in Fig. 1(B). In methanol, the open-ring isomer of diarylethene **20** was 326 nm (ε , 2.91 × 10⁴ L mol⁻¹ cm⁻¹). Irradiation **20** with 313 nm light, a new absorption band centered at 537 nm (ε , 1.64 × 10⁴ L mol⁻¹ cm⁻¹) because of the formation of **2c**, accompanied with a color change from colorless to violet. The photoconversion ratios of **1** and **2** in the photostationary state were determined using analytical HPLC, and the results are shown in Fig. 2. It was calculated that the photoconversion ratios were 93% for **1** and 88% for **2**, respectively.

The cyclization/cycloreversion quantum yields of **1** and **2** were measured and summarized in Table 1. 1,2-Bis(2-methyl-5-phenyl-3-thienyl)perfluorocyclopentene^[40] was used as a reference for the cyclization and the cycloreversion reactions. The standard deviation of the experimental error is $\pm 4\%$. The cyclization



Scheme 2. Synthetic route for diarylethenes 10



Figure 1. Absorption spectra and color changes of diarylethenes **1o** and **2o** by irradiation in methanol $(2.0 \times 10^{-5} \text{ mol L}^{-1})$ at room temperature: (A) **1o** and (B) **2o**



Figure 2. Photoconversion ratios of 10 and 20 in the photostationary state by HPLC method: (A) 1o and (B) 2o

quantum yield of 1 is higher than that of 2, whereas its cycloreversion quantum yield is lower than that of 2. As can also be seen from the data in Table 1, the absorption maxima (λ_{max}) , molar absorption coefficient, and photoconversion ratio were obviously different. The naphthalimide moiety was effective to increase the molar absorption coefficient and the photoconversion ratio.

For practical applications in optical devices, it is important that the photochromic materials can maintain good photochromism in solid supports such as PMMA films.^[41] **1** and **2** showed similar photochromism in PMMA films as observed in methanol (Fig. 3). Irradiation of 1o/PMMA film with 313 nm UV light resulted in the appearance of a new absorption band at 542 nm, accompanied with a color change from colorless to violet. The maximum absorption peak of 2c/PMMA was at 544 nm. Compared with those in methanol, the absorption maxima of the closed-ring isomers in PMMA films were bathochromic shifted by 7 nm for both 1c and 2c. The result was in good agreement with that of the reported diarylethenes.^[42-44] The reason may be attributed to the polar effect of the polymer matrix and stabilization of molecular arrangement in solid media.[45]

Fatigue resistance of diarylethenes is also critical to the practical applications in optical devices.^[1,46] The fatigue resistance of **1** was examined in both methanol and a PMMA film (Fig. 4(A) and 4(B)) with alternating UV/Vis irradiation in the air at room temperature. After repeating 10 coloration/decoloration cycles, no obvious degradation was detected by UV/Vis absorption spectroscopy. The result indicated that 1c showed remarkable fatigue resistance in solution. Moreover, the thermal stability of the open-ring and closed-ring isomers of 1 was examined in methanol at room temperature and 65 °C. At room temperature, storing the solution in the dark for more than 10 days, no change was observed in the UV/Vis spectra. When the methanol solution containing 1 was heated under reflux (65 °C) for more than 4 days in darkness, no decomposition was detected by UV/vis spectroscopy.

Fluorescence switching property

The fluorescence properties of 1o and 2o in methanol $(2.0 \times 10^{-5} \text{ mol L}^{-1})$ and PMMA films (10% w/w) were measured at room temperature. In methanol, the emission peaks of 10 and 20 appeared at 392 and 445 nm when excited at 348 and 295 nm light, respectively. Choice of anthracene in acetonitrile $(\Phi_f = 0.27)$ as a reference, the fluorescence quantum yields of 10 and 20 were 0.028 and 0.005 with 5% experimental error, respectively. Like the reported diarylethenes, [47,48] 10 and 20 exhibited remarkable fluorescence switching properties in methanol during the photoisomerization. Upon irradiation with

Table 1. Photoreaction parameters of 1 and 2 in methanol $(2.0 \times 10^{-5} \text{ mol L}^{-1})$							
Compds	$\lambda_{o,max}/\text{nm}^{a}$ ($\varepsilon L^{-1} \text{ mol}^{-1} \text{ cm}^{-1}$)		$\lambda_{c,max}/\text{nm}^{b}$ ($\varepsilon L^{-1} \text{ mol}^{-1} \text{ cm}^{-1}$)		${\varPhi}^{\sf c}$		PR ^d
	Methanol	PMMA film	Methanol	PMMA film	Φ_{o-c}	Φ_{c-o}	
1	317 (3.69×10 ⁴)	319	535 (1.84×10 ⁴)	542	0.39	0.01	93%
2	326 (2.91×10 ⁴)	331	537 (1.64×10 ⁴)	544	0.36	0.04	88%

*Standard deviation of the experimental error: \pm 4%.

^aAbsorption maxima of open-ring forms.

^bAbsorption maxima of closed-ring forms.

^cQuantum yields of cyclization reaction (Φ_{c-c}) and cycloreversion reaction (Φ_{c-o}), respectively.

^dPhotoconversion ratios of **1** and **2** (PR%) in the photostationary state.





Figure 4. Fatigue resistance of 1 in both methanol and a PMMA film



Figure 5. Emission intensity changes of diarylethenes 10 and 20 upon irradiation with 313 nm light in methanol $(2.0 \times 10^{-5} \text{ mol L}^{-1})$ at room temperature: (A) **10** and (B) **20**



Figure 6. Emission intensity changes of diarylethenes **10** and **20** upon irradiation with 313 nm light in PMMA films (10% *w/w*) at room temperature: (A) **10** and (B) **20**



Figure 7. Fluorescent switch cycles of diarylethene 1o: (A) in methanol and (B) in a PMMA film

313 nm UV light, the emission intensity of 1o and 2o decreased obviously. The back irradiation with appropriate vis light regenerated the open-ring isomers and recovered the original emission intensity. When it arrived at the photostationary state, the emission intensity of 1o and 20 was guenched to ca 20 and 40% (shown in Fig. 5), respectively. The reversible fluorescence quenching mechanism of 1o and 2o is different. The fluorescence quenching of 20 was ascribed to the enlargement of π delocalization^[1,2], electron while that of 10 was ascribed to the intramolecular energy transfer.^[49,50] The N-alkylsubstituted 1,8-naphthalimide derivative emits fluorescence at a short wavelength.^[51] When diarylethene 1 is in the open-ring form $(\lambda_{max} = 317)$ nm, in methanol), the 1,8naphthalimide unit emits fluorescence because the energy state of the diarylethene unit is higher than the fluorescent state. However, when it is in the closed-ring isomer ($\lambda_{max} =$ 542 nm, in methanol) upon irradiation with UV light, its energy state is lower than the fluorescent state. As a result, the fluorescence of the 1,8naphthalimide unit is efficiently quenched. In PMMA films, the emission peaks of 10 and 20 were observed at 416 and 425 nm when excited at 281 and 290-nm light, respectively. The emission intensity changes induced by photoirradiation of 1 and 2 in PMMA films are shown in Fig. 6. Compared with those in methanol, the emission peaks in PMMA films had an obvious red shift of 24 nm for 1 and a hypochromatic shift of 20 nm for 2. The fluorescent intensity was guenched to 17% for 1 and 21% for 2. As described previously, the fluorescent modulation efficiency of 1 was greater than that of the parent diarylethene 2 in both methanol and PMMA films

indicating that introduction of naphthalimide to the diarylethene was effective to enhance the emission intensity, fluorescent modulation efficiency, and fluorescence quantum yield.

As shown in Fig. 7, the fluorescent switch had good reversibility with alternating UV (313 nm) and visible light (>450 nm) irradiation for more than 10 cycles. The reversible changes can be used in a repeated "write-erase" process.^[52]

CONCLUSIONS

In conclusion, a novel fluorescent diarylethene with a (formyloxyethoxy)ethyl-linked naphthalimide unit was synthesized, and the effect of the naphthalimide moiety on the photochromism and fluorescence was studied. Although the naphthalimide moiety is effective to enhance the fluorescence quantum yield, there is no substantial overlap between the naphthalimide emission spectrum and the absorption spectrum of the open-ring and closed-ring isomers of the diheteroarylethene. As a result, diarylethene as an acceptor in pcFRET was not realized. Further studies on the FRET principle are now in progress.

EXPERIMENTAL SECTION

General

Nuclear magnetic resonance spectra were recorded on a Bruker AV400 (400 MHz) spectrometer with CDCl₃ as the solvent and TMS as an internal standard. Melting points were measured on a WRS-1B melting point apparatus. UV/Vis spectra were recorded on a PerkinElmer Lambda 900 spectrometer. Photoirradiation was carried out with an SHG-200 UV lamp, a CX-21 ultraviolet fluorescence analysis cabinet, and a BMH-250 vis lamp. Radiation of appropriate wavelength was isolated by different light filters. Luminescence spectra were measured on a Hitachi 4600 fluorescence spectrophotometer (the widths of the emission slit were selected 5.0 and 5.0 nm). The fluorescence quantum yield measurement was measured by excitation of the respective diarylethene (absorbance at 0.05) and compared with the emission of anthracene in acetonitrile (standard, $\Phi_f = 0.27$). All solvents used were of spectrograde and were purified by distillation prior to use. All reagents were obtained from J&K Scientific Ltd without further purification. The PMMA films were prepared by first dissolving 10-mg diarylethene sample and 100 mg PMMA in chloroform (1 mL) with the aid of ultrasound; then, the homogeneous solution was spin coated on a quartz substrate $(10 \times 10 \times 1 \text{ mm})$.

1-(2-Methyl-3-benzofuryl)-2-[2-methyl-5-(4-carboxyphenyl)-3-thienyl]perfluorocyclopentene (3)

To a stirred acetone (50 mL) solution of 20 (0.34 g, 0.66 mmol), dropwise Jones reagent that was prepared from CrO₃ (0.13 g, 1.29 mmol), H₂SO₄ (0.09 mL), and H_2O (3.00 mL) was added. The mixture was further stirred overnight. The reaction mixture was extracted with ether, and the organic layer was washed with brine, dried over MgSO₄, filtered, and evaporated. The crude product was purified by silica gel column chromatography (dichloromethane) to yield 30 (0.25 g, 74%) as a white powder. M.p. 208–209 °C. ¹H NMR (CDCl₃, 400 MHz), δ (ppm): 1.91 (s, 3H, -CH₃), 2.14 (s, 3H, -CH₃), 6.78 (s, 1H, thienyl-H), 7.20-7.24 (m, 1H, benzofuryl-H), 7.26-7.31 (m, 1H, 1H, benzofuryl-H), 7.43 (d, 1H, J=8.0 Hz, benzofuryl-H), 7.49 (d, 1H, J=8.0 Hz, benzofuryl-H), 7.64 (d, 2H, J = 8.0 Hz, phenyl-H), and 8.12 (d, 2H, J = 8.0 Hz, phenyl-H); ¹³C NMR (CDCl₃, 100 MHz, tetramethylsilane (TMS)): 13.36, 14.84, 107.74, 111.56, 117.23, 121.01, 121.86, 122.63, 123.04, 123.36, 124.24, 124.69, 127.44, 130.29, 130.84, 135.92, 136.05, 136.67, 138.43, 139.04, 154.73, and 169.04; high-resolution mass spectrometry (HRMS) (electrospray ionization (ESI⁻): *m/z*: calcd for C₂₆H₁₆F₆O₃S: 522.0724; found 521.0717; anal.

calcd for $C_{26}H_{16}F_6O_3S$ (%): C, 59.77, H, 3.09, and O, 9.19; found C, 59.71, H, 3.14, and O, 9.16.

N-[2-(2-Hydroxyethoxy)ethyl]naphthalimide (5)

To a stirred ethanol solution of **4** (0.40 g, 2.00 mmol), dropwise 2-(2-aminoethoxy)ethanol (0.32 mL, 3.20 mmol) was added in ethanol (15 mL) in a single-necked flask; the mixture was heated to reflux for 4 h. After being cooled down to room temperature, the solvent was removed under reduced pressure. The solid was purified through flash column chromatography to obtain a white solid **5** (0.49 g, 85% yield). M.p. 114–115 °C. ¹H NMR (400 MHz, DMSO-*d*₆), δ (ppm): 3.89–3.94 (m, 4H, -CH₂CH₂-), 4.44–4.50 (m, 4H, -CH₂CH₂-), 4.55 (s, 1H, -OH), 7.98 (t, 2H, *J* = 8.0 Hz, naphthyl-H), 8.56 (d, 2H, *J* = 8.0 Hz, naphthyl-H), and 8.59 (d, 2H, *J* = 8.0 Hz, naphthyl-H); ¹³C NMR (DMSO-*d*₆, 100 MHz, TMS): 39.63, 61.48, 67.84, 72.46, 123.73, 125.61, 128.46, 137.73, 137.94, 130.76, and 159.54; HRMS (ESI⁻): *m/z*: calcd for C₁₆H₁₅NO₄, 285.1001; found 285.0992; anal. calcd for C₁₆H₁₅NO₄ (%): C, 67.36, H, 5.30, N, 4.91, and O, 22.43; found C, 67.41, H, 5.34, N, 5.02, and O, 22.51.

1-(2-Methyl-3-benzofuryl)-2-{2-methyl-5-{4-[N-(2-(2formyloxyethoxy)ethyl)naphthalimide]phenyl}-3-thienyl} perfluorocyclopentene (10)

To a stirred anhydrous N,N-dimethylformamide (3 mL) solution of compounds 5 (0.13 g, 0.45 mmol) and 3 (0.18 g, 0.35 mmol), 1-ethyl-3-(3dimethylaminopropyl)carbodiimide (0.87 g, 0.45 mmol). hydroxybenzotriazole (0.61 mg, 0.45 mmol), and triethylamine (60 µL) were added. The mixture was stirred at 20 °C for 3 h under nitrogen, and then, it was concentrated, diluted with 5% of K₂CO₃ (aqueous), and extracted with ethyl acetate. The combined organic layer was dried with MgSO4, filtered, and evaporated. The crude product was purified by silica gel column chromatography using petroleum ether/ethyl acetate (v/v = 2/1) as the eluent to yield **10** (0.10 g, 36%) as a white solid. M.p. 145-146 °C. ¹H NMR (CDCl₃, 400 MHz), δ (ppm): 1.92 (s, 3H, -CH₃), 2.15 (s, 3H, --CH₃), 3.89–3.94 (m, 4H, -CH2CH2-), 4.44-4.50 (m, 4H, -CH2CH2-), 7.23 (s, 1H, thienyl-H), 7.25-7.29 (m, 2H, benzofuryl-H), 7.39 (d, 1H, J = 8.0 Hz, benzofuryl-H), 7.43 (d, 1H, J = 8.0 Hz, benzofuryl-H), 7.47 (d, 2H, J = 8.0 Hz, phenyl-H), 7.68–7.72 (t, 2H, J=8.0, naphthyl-H), 7.86 (d, 2H, J=8.0, phenyl-H), 8.16 (d, 2H, J = 8.0 Hz, naphthyl-H), and 8.54 (d, 2H, J = 8.0 Hz, naphthyl-H); ¹³C NMR (CDCl₃, 100 MHz), δ (ppm): 13.36, 14.84, 39.10, 64.14, 68.08, 68.58, 105.44, 111.10, 119.95, 122.56, 123.67, 124.65, 125.01, 126.01, 126.19, 126.88, 128.21, 129.15, 130.30, 131.29, 131.57, 133.89, 137.13, 141.09, 142.70, 154.19, 156.15, 164.25, and 165.87; IR (KBr, v, cm⁻¹): 344, 747, 781, 896, 837, 989, 1067, 1093, 1123, 1274, 1273, 1333, 1344, 1662, 1714, and 2864; HRMS (ESI⁻): *m/z*: calcd for C₄₂H₂₉F₆NO₆S 789.1620; found 788.1622; anal. calcd for $C_{42}H_{29}F_6NO_6S$ (%): C, 63.88, H, 3.70, N, 1.77, and O, 12.16; found C, 63.82, H, 3.73, N, 1.79, and O, 12.15.

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