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New photochromic bisthienylethene derivatives containing carbazole

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ABSTRACT: Some new photochromic bisthienylethene (BTE) derivatives containing carbazole were synthesized by Suzuki coupling method. All compounds were characterized by ^{1}H NMR spectra, ^{13}C NMR, mass spectra, etc. Their optical, photochromic, and electrochemical properties were described. Moreover, their two-photon absorption (TPA) properties were investigated both in open and closed forms, finding observable distinction in the TPA of the ring-open and ring-closed forms. These compounds could be used as two-photon switches and for the potential applications in three-dimensional optical storage. Copyright © 2007 John Wiley & Sons, Ltd.
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KEYWORDS: synthesis; photochromic; bisthienylethene; carbazole; two-photon

INTRODUCTION

Photochromic materials are potentially useful for advanced optoelectronic devices such as optical memory, optical switching, and displays.[1] Among various types of photochromic compounds, bisthienylethene (BTE) derivatives are the most promising compounds because of their excellent fatigue resistance and thermal stability in both isomeric forms, picosecond switching times, and a high photochemical quantum yields.[2–4] Currently, optical data can be stored in two-dimensional volume of photochromic materials; however, the techniques have fundamental limitations in the available memory density owing to their two-dimensional nature. The constraint can be overcome if memory can be stored within the three-dimensional volume of the material, increasing the data holding capacity by a factor proportional to the thickness of the medium. Within this theme, the two-photon absorption (TPA) of photochromic compounds can be utilized to achieve higher spatial resolution than one-photon absorption (OPA). Thus, it is desirable to synthesize and characterize active organic photochromic materials with large TPA cross-section values using high-density multilayer storage devices.[5,6]

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The open and closed-ring isomers of photochromic BTE derivatives differ from each other not only in their absorption but also in various physical and chemical properties, such as luminescence, refractive indices, oxidation/reduction potentials, chiral properties, magnetic interactions, and two-photon properties.[1–3,7,8] On the basis of our earlier work,[9–11] herein we incorporated the photochromic BTE unit and carbazole units via a Suzuki coupling reaction for the purpose of developing photochromic compounds with large two-photon cross-sections. Photochromic compounds C-BTE and C-BTE-C (Scheme 1) with good solubility in common organic solvents could be synthesized with structures between the colorless ring-opened and colored ring-closed forms by alternating irradiation with 254 nm and visible light (>500 nm) in THF. Moreover, two-photon as well as electrochemical properties of these new photochromic compounds were also discussed, which showed that C-BTE and C-BTE-C compounds could work as photoswitches and two-photon switches.

RESULTS AND DISCUSSIONS

Molecular design and synthesis

Here, the Suzuki coupling method is used to synthesize symmetric and unsymmetric BTE derivatives by controlling the ratio of starting materials and reagents.[12–14]

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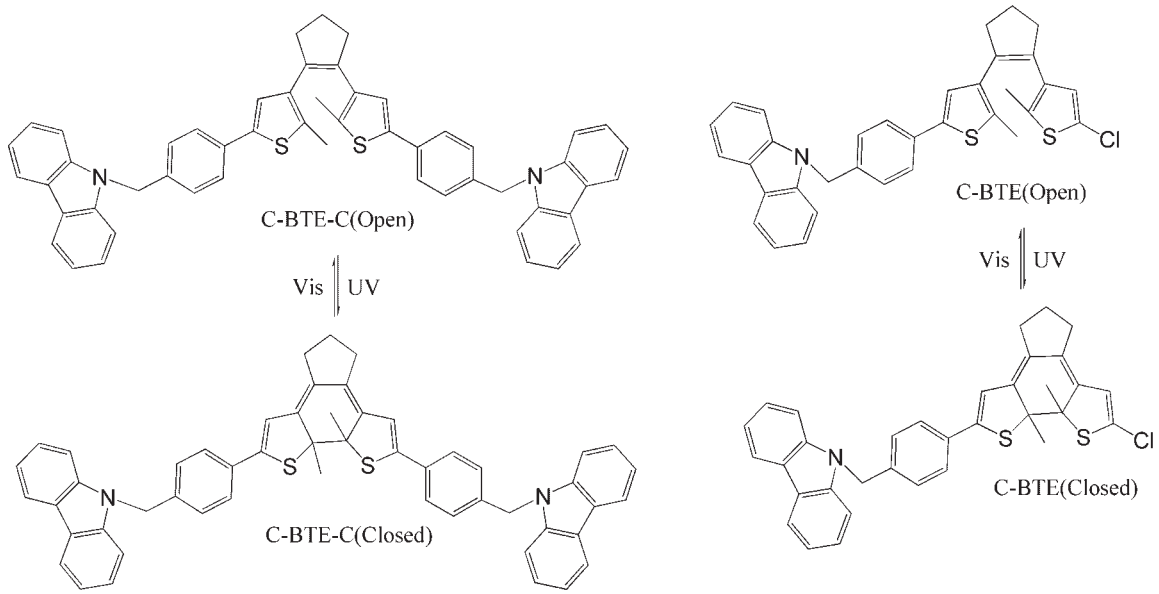
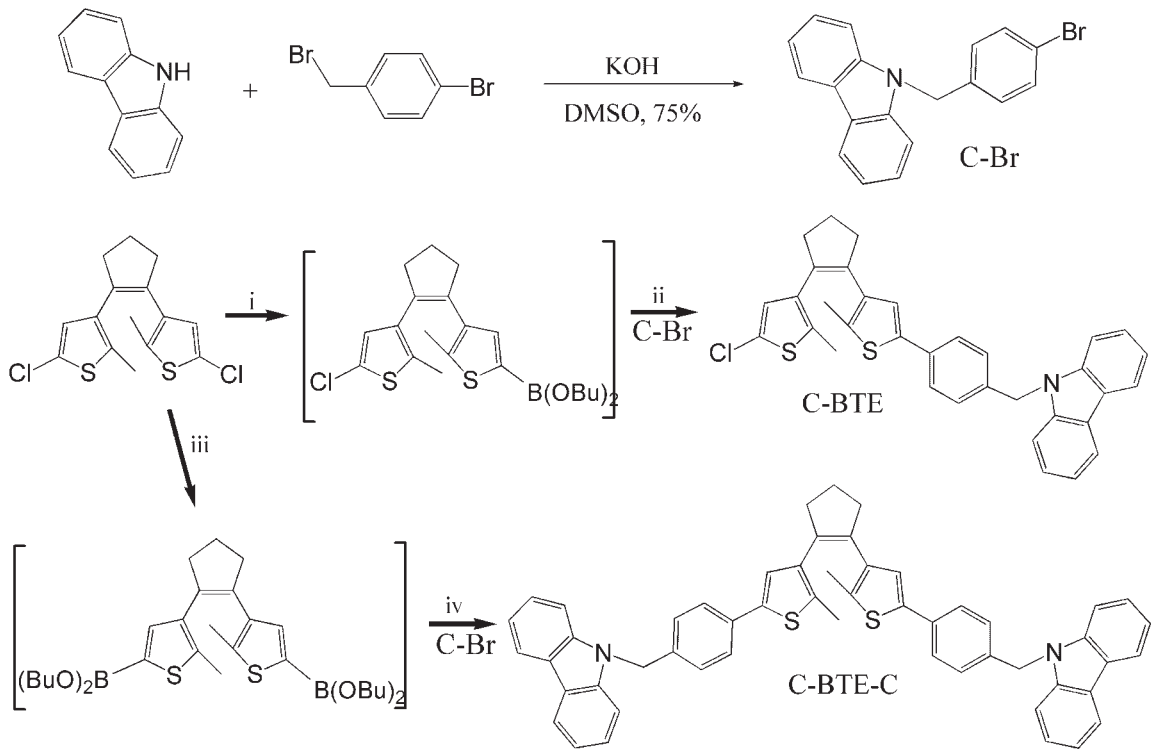


Scheme 1. The photochromic process of C-BTE and C-BTE-C (bisthienylethenes containing carbazole units)

1,2-Bis(5-chloro-2-methylthien-3-yl) cyclopentene was converted into the bis(boronic) esters of BTE via *n*-BuLi/$B(OBu)_3$, and then directly used in the next Suzuki reaction without any workup because during isolation the bis(boronic) esters are easily deboronized. Suzuki coupling with *N*-(4-bromobenzyl)-carbazole gave the corresponding unsymmetric C-BTE and symmetric C-BTE-C compounds by controlling the ratio of starting materials and reagents (Scheme 2).

Absorption spectra

All synthesized compounds could readily dissolve in common organic solvents, such as chloroform, dichloromethane, and THF. These BTE derivatives show typical photochromism in solutions. The THF solution of C-BTE (ring-open form) was colorless. Upon irradiation with light of 254 nm, the solution gradually turned pink and new absorption bands at 339 and 490 nm appeared from



Scheme 2. Routes for synthesizing C-BTE and C-BTE-C. Reagents: (i) *n*-BuLi (1 eq), $B(OBu)_3$, THF, −78°C; (ii) 2 M Na_2CO_3, $Pd(PPh_3)_4$, THF, 60°C, 43%; (iii) *n*-BuLi (2 eq), $B(OBu)_3$, THF, −78°C; (iv) 2 M Na_2CO_3, $Pd(PPh_3)_4$, THF, 60°C, 34.2%

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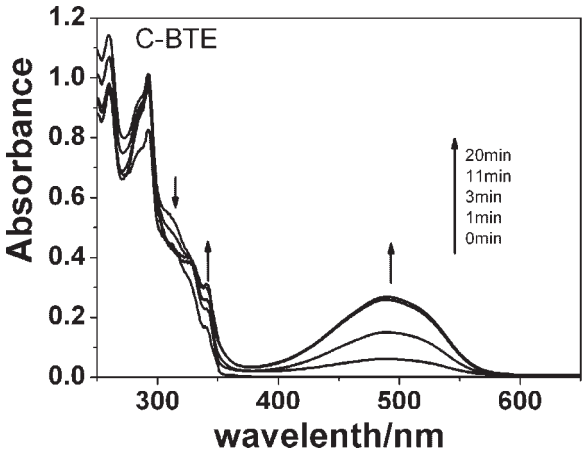

Figure 1. Absorption changes of C-BTE in THF (1.0×10^{-5} mol L^{-1}) under different irradiation times by 254 nm light

the ring-closed form of BTE (Fig. 1). A similar result was also obtained from Fig. 2. When symmetric C-BTE-C in THF was irradiated at 254 nm, the solution gradually turned purple and the new absorption bands appeared at 366 and 540 nm, which were ascribed to the closed form isomer. The maximal absorption band of the closed-ring form of C-BTE-C was red-shifted with respect to that of C-BTE in THF, due to the formation of an extended π-conjugation system.[15,16]

Both of the two compounds in THF attained a photo-stationary state (PSS) at about 15 min. Especially, the colored isomers (closed forms) of these compounds are very stable in dark at the room temperature. Irradiation of their PSS with >500 nm light could lead to a complete recovery of the initial absorption signal, and the pink or purple solution turned colorless.

HPLC is a suitable way to measure the ratios since the ring-open and closed forms of BTE derivatives are difficult to be separated by silica column chromatography. The ratio between the ring-open and closed forms can be obtained as 1.0:0.6 from the ratio of the integrated area of HPLC peaks. Similarly, the ratio of C-BTE ring-open and closed forms in the PSS was 1.0:0.56.

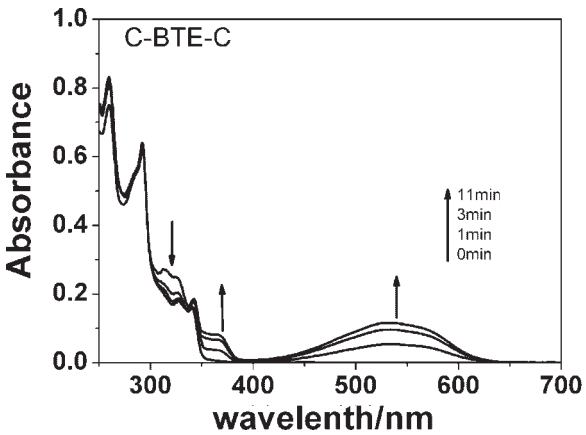

Figure 2. Absorption changes of C-BTE-C in THF (1.0×10^{-5} mol L^{-1}) under different irradiation times by 254 nm light

Fluorescence spectra

Typical fluorescence spectral changes of these new BTE derivatives were obtained for the photochromic reaction. The emission spectra excited at 300 nm for C-BTE and 315 nm for C-BTE-C are shown in Fig. 3. In the open form of C-BTE (Fig. 3(a)), the fluorescence intensity peaks are located at 349 and 363 nm. After irradiation at 254 nm to reach the PSS, the fluorescence intensity of C-BTE in THF increased. A similar result was obtained from Fig. 3(b), when C-BTE-C was excited at 315 nm, and the fluorescence intensity appeared at 349, 364, and 385 nm, and after irradiation at 254 nm, the fluorescence intensity of C-BTE-C was also significantly increased. In Fig. 3(a), the fluorescence peaks at 349 and 363 nm were ascribed to carbazole and the fluorescence intensity of BTE was quenched due to the Cl group, which was a fluorescence quenching group. After irradiation at 254 nm, the absorption of the open form of BTE declined. Therefore, when excited at 300 nm, the amount of

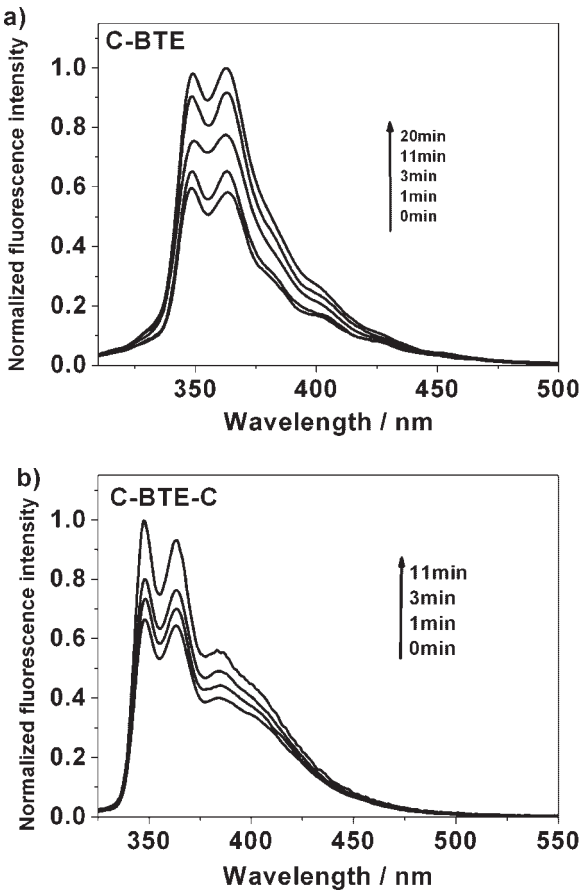

Figure 3. Fluorescence changes of C-BTE ($\lambda_{ex} = 300$ nm) and C-BTE-C ($\lambda_{ex} = 315$ nm) in THF (1.0×10^{-5} mol L^{-1}) under different irradiation times by 254 nm light

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photons absorbed by the carbazole molecule was increased and fluorescence intensity of carbazole increased.[17] For C-BTE-C, the fluorescence peaks at 349 and 363 nm were ascribed to carbazole and the peak at 385 nm was ascribed to BTE. When irradiated at 254 nm, fluorescence intensity of carbazole units increased. The enhanced luminescence of the closed ring of C-BTE and C-BTE-C upon irradiation with 254 nm light might be attributed to the formation of an extended π-conjugation system that results from the photocyclization[18] and the very small spectral overlap between the emission and the absorption bands of the closed-ring form.[19]

The fluorescence lifetimes of C-BTE and C-BTE-C (open forms) were obtained by an interactive nonlinear deconvolution fitting procedure and the fluorescent lifetimes were well fitted with a single exponential function. The fluorescence of open form of C-BTE in THF decayed with a lifetime of 1.86 ns ($\chi^2 = 1.112$, excited at 360 nm) and the fluorescence lifetime of the open form of C-BTE-C was 2.11 ns ($\chi^2 = 1.185$, excited at 360 nm).

Notably, irradiation of C-BTE and C-BTE-C with >500 nm light could lead to a complete recovery of the initial fluorescence signal. Figure 4 shows that the fluorescence intensity of C-BTE-C at 349 nm changed reversibly upon excitation at 315 nm by alternating irradiation with 254 nm and >500 nm light. This cycle could be repeated more than 10 times, indicative of their good fatigue resistance.

Electrochemical properties

Electrochemical studies were performed on C-BTE and C-BTE-C by a VersaStar II electrochemical analyzer. Figure 5 shows cyclic voltammograms of C-BTE-C in dichloromethane containing tetrabutylammonium tetrafluoroborate ($0.1\ \mathrm{mol\,L^{-1}}$). The oxidation levels of C-BTE-C were 1.42 and 1.22 V before the irradiation, and after subsequent irradiation with 254 nm to reach the PSS the oxidation states appeared at 1.42 and 1.11 V. Similarly, in C-BTE, the open-ring isomer of C-BTE showed three oxidation states at 1.60, 1.47, and 1.34 V. After 20 min irradiation with 254 nm to reach the PSS, the new oxidation states appeared at 1.15, 0.79, and 0.67 V. The oxidation states of the ring-closed form appeared at lower potentials with respect to that of the ring-open form, which clearly indicates that the ring-closed isomers of C-BTE and C-BTE-C are easily oxidized than the ring-open isomers.[18]

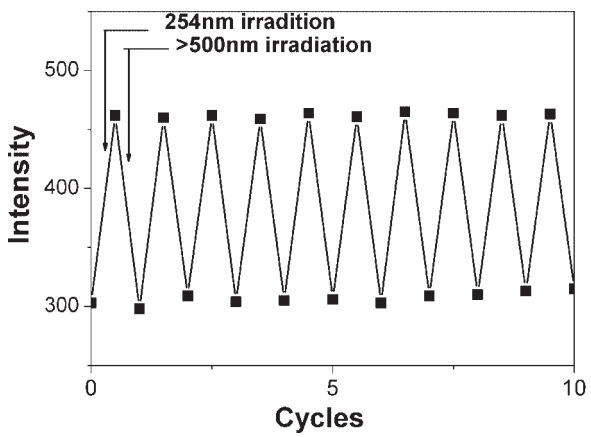

Figure 4. Modulated emission intensity at peak of 349 nm (excited at 315 nm) of C-BTE-C in THF during alternating irradiation at the wavelength of 254 nm and >500 nm light, respectively

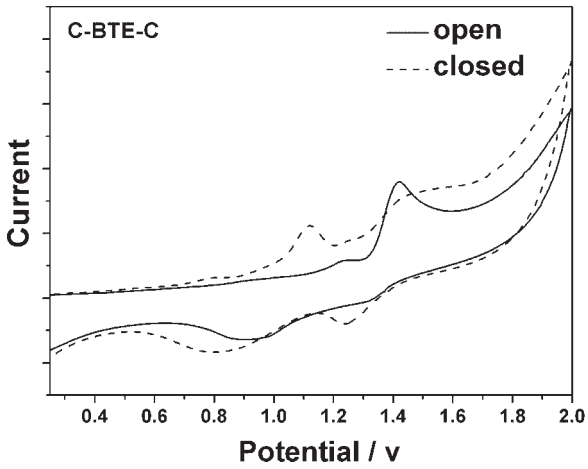

Figure 5. Cyclic voltammograms of C-BTE-C in dichloromethane containing tetrabutylammonium tetrafluoroborate ($0.1\ \mathrm{mol\,L^{-1}}$) before (solid line) and after (dashed line) irradiation with 254 nm light at the scan of $100\ \mathrm{mV\,s^{-1}}$

Two-photon properties

Recently, extensive efforts have been concentrated on the synthesis of organic materials having large TPA cross-section, and the investigation of their chemical structure and TPA property relationships. It is known that TPA can be enhanced either by increasing the conjugation length or by an appropriate combination of electron donors and acceptors.

A typical TPA optical power limiting curve (e.g., taking C-BTE-C (open form) at 600 nm) is shown in Fig. 6. Value of TPA cross-section (given in $\mathrm{GM} = 10^{-50}\ \mathrm{cm^4\,s\,photon^{-1}\,molecule^{-1}}$) for C-BTE-C (open form) was measured to be 5506.64 GM, and the coefficient (β) was 1.0 cm/GW. TPA cross-section values for C-BTE and C-BTE-C (open and closed form) were measured at different wavelength of the laser pulses, as shown in Fig. 7. For C-BTE, TPA cross-section values were measured at every 25 nm interval between 575 and 750 nm. If the wavelengths were shorter than 575 nm, the transmission of the closed form was too low, and if the wavelengths were longer than 750 nm, the robustness of

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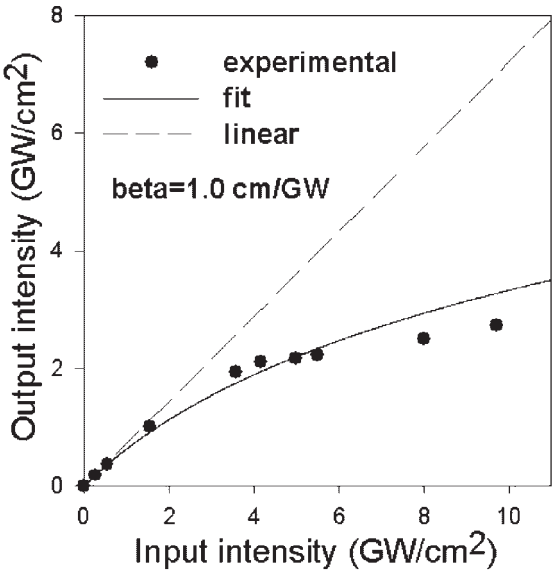

Figure 6. Measured output intensity *versus* input intensity of the 600 nm laser pulses of compound C-BTE-C (open form)

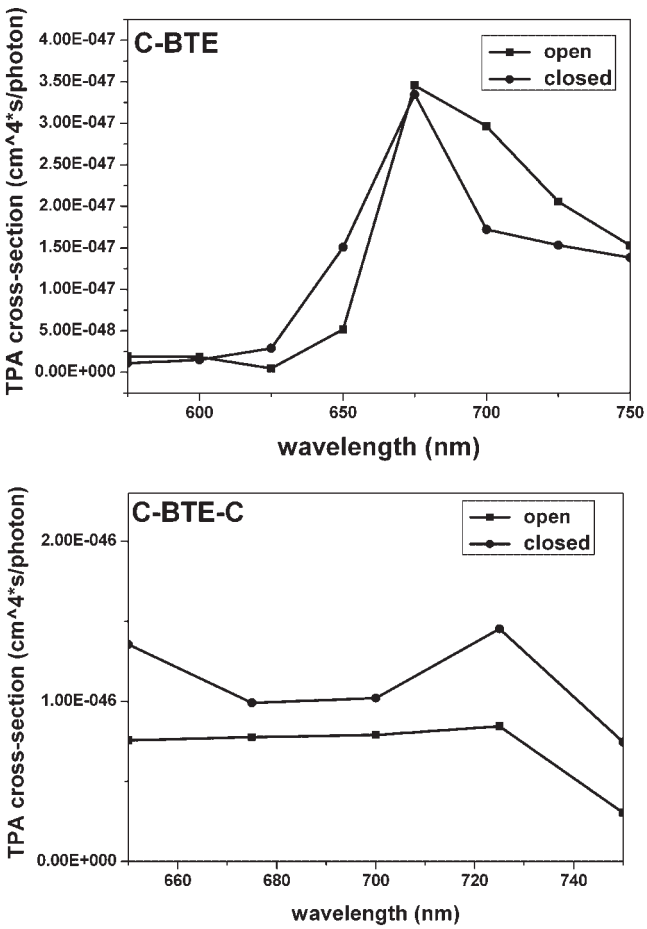

Figure 7. TPA cross-section values for C-BTE and C-BTE-C were measured at different wavelengths of the laser pulses

the laser energy would make big errors in the measurements. For C-BTE-C, TPA cross-section values were measured at every 25 nm interval between 650 and 750 nm. TPA cross-section values of C-BTE-C are larger than those of C-BTE, since carbazole is a strong electron-donating group and the C-BTE-C has a D-Π-D structure. Also TPA was enhanced by the increased conjugation length.[20] There are observable distinctions in the TPA of the ring-open and ring-closed forms of the two compounds at the same wavelength, which can be used as a two-photon switch. Compared to TPA values of the reported BTE derivatives,[6] the two compounds have significant TPA values, especially for C-BTE-C, indicating the potential utility of these compounds as three-dimensional information storage media.

CONCLUSION

In summary, new photochromic C-BTE and C-BTE-C were successfully synthesized via the Suzuki coupling method by controlling the ratios of the starting materials and reagents. They show good photochromic properties, excellent fatigue resistance, solubility, electrochemical properties, and large TPA cross-sections. There are observable distinctions in the TPA of the ring-open and ring-closed forms of the two compounds. These photochromic compounds could be used as two-photon switches and have potential application for three-dimensional optical data storage. Further investigation of the applications is currently in progress.

EXPERIMENTAL

General procedure

^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AM 500 spectrometer with tetramethyl silane (TMS) as the internal reference. MS were recorded by EI mass spectroscopy. Absorption spectra were measured by a Varian Cary500 UV–Vis spectrophotometer. Fluorescence spectra were measured with a Varian Cary Eclipse Fluorescence spectrophotometer. Fluorescence lifetimes were measured by an Edingburg Lifespec-Ps spectrofluorometer. The optical switch experiments were carried out using a photochemical reaction apparatus with a 200 W Hg lamp. Cyclic voltammetry measurements were carried out with a platinum electrode using millimolar solutions in CH_2Cl_2 containing 0.1 M of the support electrolyte, tetrabutylammonium tetrafluoroborate, in a three-electrode cell and potentiostat assembly by VersaStarII electrochemical analyzer. HPLC analyses were determined by Agilen 1100 and column Extend-C18 (5 μm, Column 4.6 × 150 mm) eluted by methanol/acetonitrile (1.5:1) at a flow rate of 0.6 ml min^{-1}.

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Linear transmission spectra were recorded on a Shimadzu UV-3600 PC spectrophotometer and TPA cross-sections were measured by the nonlinear transmittance method. The excitation source was an OPO system (Continuum, Pather OPO) pumped by the triple frequency output of a Q-switched Nd:YAG laser system (Continuum, Precision II 8010) producing ~7 ns-duration, ~1 cm^{-1} spectral-width laser pulses with a repetition rate of 10 Hz. The wavelengths can be tuned from 450 to 1025 nm. The concentration of samples was fixed at 0.01 M dichloromethane, and quartz cells having a path length of 1 cm were used for all measurements.

Materials

Reagents and starting materials were used as received. Solvents were distilled and dried before use. 1,2-Bis(5-chloro-2-methylthien-3-yl) cyclopentene was synthesized and purified according to the established procedures.[21,22]

Synthesis of *N*-(4-bromobenzyl)-carbazole

A mixture of carbazole (1 g, 6.0 mmol), KOH (0.6 g, 10.8 mmol), and DMSO (30 ml) was stirred for 30 min at room temperature under nitrogen. Then the solution of 4-bromobenzyl-bromide (1.8 g, 7.2 mmol) in DMSO (10 ml) was added dropwise for about 2 h. Subsequently, the reactive mixture was heated at a temperature of 55°C for another 2 h and cooled to the room temperature. The reactive mixture was poured into H_2O (100 ml) to a solid sample. The resulting solid was recrystallized from ethanol to get *N*-(4-bromobenzyl)-carbazole as a white powder (yield 75%), m.p. 184–186°C.

^{1}H NMR (500 MHz, $CDCl_3$, ppm): $\delta = 5.63$ (s, 2H, —CH_2—), 7.08 (d, $J = 8.4$ Hz, 2H, Ph-H), 7.21 (t, $J1 = 7.1$ Hz, $J2 = 7.2$ Hz, 2H, Ph-H), 7.40–7.46 (m, 4H, Ph-H), 7.58 (d, $J = 8.2$ Hz, 2H, Ph-H), 8.17 (d, $J = 7.7$ Hz, 2H, Ph-H). ^{13}C NMR ($CDCl_3$, ppm): 140.46, 136.18, 131.90, 128.11, 125.94, 123.08, 121.31, 120.48, 119.41, 108.72, 46.01. MS (*m/z*) [M^+] Calcd. for $C_{19}H_{14}BrN$: 335.0, Found: 335.0.

Synthesis of C-BTE

To the solution of 1,2-bis(5-chloro-2-methylthien-3-yl) cyclopentene (0.50 g, 1.52 mmol) in anhydrous THF (10 ml) *n*-BuLi (1 ml of 1.6 M solution in hexane, 1.6 mmol) was added using a syringe in two portions under nitrogen at −78°C. This solution was stirred for 30 min at room temperature, then $B(OBu)_3$ (0.5 ml, 1.7 mmol) was added to one portion. This reddish solution was stirred for 1 h at room temperature and was then used in the Suzuki cross coupling reaction without any workup because the product was deboronized during isolation.

A mixture of *N*-(4-bromobenzyl)-carbazole (0.51 g, 1.52 mmol), $Pd(PPh_3)_4$ (0.14 g), and THF (10 ml) was stirred for 15 min at room temperature. Then aqueous Na_2CO_3 (10 ml, 2 M) was added. The reactive mixture was heated at a temperature of 60°C and the solution of bis(boronic) esters of BTE was added dropwise via a syringe. Subsequently, the mixture was refluxed for 20 h and cooled to room temperature. The reactive mixture was poured into H_2O and extracted with ether and dried with anhydrous Na_2SO_4. After concentrating, the compound was purified by column chromatography on silica (petroleum ether/ethyl acetate = 10:1 v/v) to yield C-BTE (yield 43%).

C-BTE: ^{1}H NMR (500 MHz, DMSO, ppm): $\delta = 1.77$ (s, 3H, —CH_3), 1.84 (s, 3H, —CH_3), 1.91–1.95 (m, 2H, —CH_2—), 2.66–2.72 (m, 4H, —$CH_2C{=}CCH_2$—), 5.61(s, 2H, —CH_2—), 6.77 (s, 1H, thiophene-H), 7.10 (s, 1H, thiophene-H), 7.13 (d, $J = 8.1$ Hz, 2H, Ph-H), 7.20 (t, $J1 = 7.3$ Hz, $J2 = 7.6$ Hz, 2H, Ph-H),7.37 (d, $J = 7.9$ Hz, 2H, Ph-H), 7.40 (t, $J1 = 8.1$ Hz, $J2 = 7.2$ Hz, 2H, Ph-H), 7.59 (d, $J = 8.2$ Hz, 2H, Ph-H), 8.16 (d, $J = 7.7$ Hz, 2H, Ph-H). ^{13}C NMR (d_6-DMSO, ppm): 140.53, 139.08, 137.29, 136.76, 135.61, 135.33, 134.06, 133.78, 133.41, 133.10, 127.90, 127.72, 126.32, 125.47, 124.52, 123.95, 122.68, 120.80, 119.53, 109.94, 45.69, 38.37, 38.20, 22.65, 14.34, 14.17. MS (*m/z*) [M^+] Calcd. for $C_{34}H_{28}ClNS_2$: 549.1, Found: 549.1.

Synthesis of C-BTE-C

Compounds C-BTE-C were prepared in the same manner as described above for the preparation of C-BTE. To the solution of 1,2-bis(5-chloro-2-methylthien-3-yl) cyclopentene (0.50 g, 1.52 mmol) in anhydrous THF (10 ml) was added *n*-BuLi (2.5 ml of 1.6 M solution in hexane, 4.0 mmol) using a syringe in two portions under nitrogen at −78°C. Then $B(OBu)_3$ (1 ml, 3.4 mmol) was added to one portion. At the same reaction condition as for BTE, the solution of the symmetric bis(boronic) esters of BTE was obtained. Then the mixture of *N*-(4-bromobenzyl)-carbazole (1.2 g, 3.1 mmol), $Pd(PPh_3)_4$ (0.30 g), and THF (10 ml) was stirred for 15 min at room temperature. Then aqueous Na_2CO_3 (10 ml, 2 M) was added. The reactive mixture was heated at 60°C and the solution of bis(boronic) esters of BTE was added dropwise via a syringe. C-BTE-C was similarly obtained (petroleum ether/ethyl acetate = 6:1 v/v) with a yield of 34.2%.

C-BTE-C: ^{1}H NMR (500 MHz, DMSO, ppm): $\delta = 1.83$ (s, 6H, —CH_3), 1.95–1.98 (m, 2H, —CH_2—), 2.73–2.76 (t, 4H, —$CH_2C{=}CCH_2$—), 5.62(s, 4H, —CH_2—),7.10 (s, 2H, thiophene-H), 7.12 (d, $J = 8.1$ Hz, 4H, Ph-H), 7.24 (t, $J1 = 7.3$ Hz, $J2 = 7.5$ Hz, 4H, Ph-H), 7.37 (d, $J = 8.2$ Hz, 4H, Ph-H), 7.42 (t, $J1 = 7.3$ Hz, $J2 = 8.1$ Hz, 4H, Ph-H), 7.61 (d, $J = 8.2$ Hz, 4H, Ph-H), 8.17 (d, $J = 7.7$ Hz, 4H, Ph-H). ^{13}C NMR (d_6-DMSO, ppm): 140.52, 138.88, 137.23, 137.00, 134.62, 133.99, 133.16, 127.88, 126.32,

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125.46, 124.66, 122.67, 120.80, 119.53, 109.94, 45.67, 41.77, 41.56, 14.36. MS (*m/z*) [M^+] Calcd. for $C_{53}H_{42}N_2S_2$: 770.3, Found: 770.1.

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Supporting information available

Supporting Information is available online from Wiley InterScience or from the author.

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