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Cucurbit[8]uril-induced diarylethene thermally activated delay fluorescence supramolecular switch for sequential energy transfer

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ABSTRACT

Constructing molecular switches based on supramolecular assembly strategy is a research hotspot. In this work, we constructed an all visible-light-regulated supramolecular photo-switch based on pyridinium-modified diarylethene derivative (DTE-Me) and cucurbit[8]uril (CB[8]). CB[8] not only accelerated the photochromic process under 365 nm ultraviolet light but also shifted the absorption of open formed DTE-Me to the visible region, which led to efficient photocyclization under 450 nm visible light irradiation, while DTE-Me and DTE-Me/CB[7] remained unchanged under the same irradiating condition. Moreover, the complexation with CB[8] could induce the strong thermally activated delayed fluorescence (TADF) of guest molecular at 550 nm, which further shifted to 670 nm through two-step sequential energy transfer with sulforhodamine B (SRB) and Cy5. This energy transfer process could also be regulated with visible light, and the application for information encryption was also demonstrated. This assembly provides a convenient approach to construct all visible light-regulated TADF photo-switch. © 2022 Elsevier Ltd. All rights reserved.

1. Introduction

Constructing molecular switches based on supramolecular assembly strategy is a research hotspot [1]. As an excellent building block, diarylethene derivatives are attractive photochromic molecules on account of their excellent thermal stability and fatigue resistance [2-6]. In most cases, diarylethene derivatives could undergo a photocyclization reaction from open form (OF) to closed form (CF) through ultraviolet (UV) light irradiation. However, UV light has distinct disadvantages of phototoxicity and low transparency, which undoubtedly hinders its applications, in particular for biological applications. Upconversion [7-10] triplet sensitization [11–15], elongating the conjugated π system [16–18], and intramolecular proton transfer [19] are reported methods to obtain visible-light-regulated diarylethene photo-switches. Macrocyclic molecules could produce a significant impact on the absorption of guest molecules through non-covalent interaction [20], which could also be used to construct the visible-light-induced photocyclization diarylethene system. Among these macrocyclic molecules, cucurbit[8]uril (CB[8]) possessing a hydrophobic cavity, and

* Corresponding author. E-mail address: yuliu@nankai.edu.cn (Y. Liu). polar carbonyl hydrophilic portals could accommodate two guest molecules with charge-transfer property, which often leads to the absorption of guest molecules displaying apparent bathochromic shift (see Scheme 1).

Recently, we have reported supramolecular switch formed by molecular self-folding and dimerization [21-23]. However, supramolecular switch with thermally activated delayed fluorescence (TADF) is rare. TADF means that a chromophore is capable of absorbing thermal energy from its surroundings so that it can undergo reverse intersystem crossing (RISC) from the triplet state back to its singlet state, and then decay radiatively back to the ground state accompanied by fluorescent emission [24-26]. Molecules with TADF property are generally designed with donor and acceptor, in which the charge-transfer interaction between donor and acceptor could promote intersystem crossing (ISC) efficiently [27-31]. Herein, we synthesized a symmetrical diarylethene derivative (DTE-Me) modified with thiophene (donor) and pyridinium (acceptor), which could form n:n complex with CB[8] with enhanced charge-transfer interaction. CB[8] could accelerate the photocyclization process of DTE-Me upon 365 nm light irradiation. Moreover, CB[8] shifts the absorption bands of DTE-Me to the long wavelength region resulting in visible-light-induced photocyclization, while DTE-Me and DTE-Me/CB[7] remain OF state upon same light irradiation. Beneficial from the difference in photocyclization rate, multicolor







luminescence could be obtained under different irradiation times. The strong TADF of DTE-Me induced by CB[8] could serve as a donor to sequentially transfer its energy to suitable acceptor dye molecules SRB and Cy5, and the energy transfer process could be regulated by visible light. (Scheme 1)

2. Results and discussion

The diarylethene derivative (DTE-Me) and reference compound (Me) were comprehensively characterized (Schemes S1–S2 and Figs. S1–S9), and the photochromic property was first investigated. Upon 365 nm light irradiation, the protons of DTE-Me displayed obvious shifts, for example, the protons in thiophene (H_A) shifted from 7.80 to 7.29 ppm while the protons in methyl (H_B) showed opposite shifts from 2.04 to 2.17 ppm, indicating the conversion from OF isomer (DTE_{OF}-Me) to CF isomer (DTE_{CF}-Me). The photoconversion efficiency was calculated to be 97% through ¹H NMR spectra (Fig. S10). Subsequently, the irradiation of >600 nm light resulted in the complete recovery of ¹H NMR spectrum suggesting the excellent photochromic characteristic.

The UV-vis spectra were also used to monitor the photochromic process. When irradiated by 365 nm UV light, the absorbance of DTE_{OF}-Me at 268 and 365 nm decreased and a new absorption at 620 nm appeared due to the formation of DTE_{CF}-Me. The photostationary state was achieved after 340 s UV light irradiation (Fig. S11). Upon adding CB[7], the peak at 365 nm shifted to 380 nm, but the solution was still colorless (Fig. 1a). The binding constant $(K_{\rm s})$ was calculated to be 4.22 \times 10¹¹ M⁻² by UV-vis titration spectra, and the binding stoichiometry of DTE-Me/CB[7] was determined to be 1:2 by Job's plot (Figs. S12 and S13). The peak for DTE_{CF}-Me/CB[7] also displayed bathochromic shift and DTE_{CF}-Me/ CB[7] came to photostationary state with a shorter time of 65 s (Fig. 1b and c and S11). The binding stoichiometry and K_s of DTE-Me/CB[8] were determined to be 1:1 and 0.83 \times 10⁷ M⁻¹, respectively (Figs. S12 and S13). Impressively, the time for photocvclization of DTE-Me/CB[8] further decreased to 24 s. The cyclization reaction rate constant of DTE-Me, DTE-Me/CB[7], and DTE-Me/CB[8] were determined to be 0.0007, 0.0037, and 0.0114 s^{-1} , respectively, by monitoring only the initial conversion (Fig. S14). These results indicated that CB[n] could accelerate the cyclization reaction. Besides, these CF isomers could return to OF isomers through >600 nm visible light irradiation (Fig. S15). However, the rate of back cyclization of DTE-Me/CB8 was lower than DTE-Me and DTE-Me/CB7 (Fig. S16).

It should be noted that the absorption of DTE_{OF}-Me shifted to visible light region upon adding CB[8] and the color of the solution turned into yellow, which inspired us to trigger the photocyclization of DTE_{OF}-Me/CB[8] with visible light (Fig. 1a). As expected, the absorption at 665 nm increased after visible light irradiation of 450 nm, suggesting the efficient photocyclization (Fig. 1d and S17). But DTE_{OF}-Me and DTE_{OF}-Me/CB[7] only produced slight change in absorption after the same irradiating condition. DTE_{OF}-Me and DTE_{OF}-Me/CB[7] did not possess sufficient absorption around 450 nm, while the intermolecular charge-transfer interaction induced by CB[8] could red-shifted the absorption into visible region, which resulted in the low-energy visible light of 450 nm could motivate the photocyclization reaction of DTE-Me.

In order to verify the binding mode of DTE-Me with CB[7] and CB[8], reference compound Me with better water-solubility was synthesized. The protons H_{c-h} shifted to upfield, while H_{a,b} shifted to downfield indicating that the phenylpyridinium moieties were induced into the cavity of CB[8] with "head-to-tail" pattern, while the methyl in thiophene was located outside the cavity of CB[8] (Fig. S18). This packing pattern was in favor of intermolecular charge transfer as confirmed by the redshift absorption of DTE-Me after complexing with CB[8]. When CB[7] was added to the solution of Me, the NMR shifts were slightly different, in which protons H_{d-g} shifted to upfield while H_{a-c.h} shifted to downfield implying that methyl in both thiophene and pyridinium were located outside the cavity of CB[7] (Fig. S19). Though CB[7] could accommodate a phenylpyridinium in its cavity, but the intermolecular chargetransfer interaction was weaker than DTE-Me/CB[8], which led to the main absorption band was still in UV region. When CB[6] was added to the solution of Me, no apparent ¹H NMR shifts were observed and the UV-vis, and photoluminescent spectra of DTE-Me/CB[6] also did not show obvious changes, suggesting that the affinity between DTE-Me and CB[6] was weak (Figs. S20 and S21).

The emission of DTE_{OF} -Me was very faint but dramatically enhanced after adding CB[8] (Fig. 2a). The enhanced emission at 550 nm could be probably attributed to the restriction of intramolecular rotation and suppression of non-radiative transition after complexing with CB[8]. CB[7] could also induce the DTE_{OF} -Me into its cavity to enhance the emission, but the emission was blue shifted to 500 nm due to the charge-transfer interaction was weaken (Fig. S22a). Interestingly, when N₂ was bubbled into the solution, the emission intensity of DTE_{OF} -Me/CB[8] at 550 nm increased, while DTE-Me/CB[7] did not show such enhancement (Fig. 2b and S22b). The excited triplet state is very sensitive to the



Scheme 1. Schematic illustration and chemical structure of the photo-controlled thermally activated delayed fluorescence energy transfer of DTE-Me/CB[8]@SRB@Cy5.



Fig. 1. UV-vis spectral changes of (a) DTE_{OF}-Me and (b) DTE_{CF}-Me upon adding CB[7] and CB[8]. (c) Absorbance variation curves of DTE_{OF}-Me, DTE_{OF}-Me/CB[7], and DTE_{OF}-Me/CB[8] over the irradiation time of 365 nm UV light. (d) Absorbance variation curves of DTE_{OF}-Me, DTE_{OF}-Me/CB[7], and DTE_{OF}-Me/CB[8] over the irradiation time of 450 nm visible light. ([DTE-Me] = 10 μ M, [CB[7]] = 20 μ M, [CB[8]] = 10 μ M).

oxygen which could quench the excited triplet state through nonirradiative pathways [29]. Therefore, the decrease in the emission intensity indicated the existence of an excited triplet state in DTE-Me/CB[8]. As shown in temperature variation experiments, the emission intensity at 550 nm displayed great decrease when the temperature decreased from 277K to 77K, indicating the existence of the RISC of excitons because the ambient thermal energy can facilitate RISC from T_1 to S_1 [31]. (Fig. 2c) Time-resolved emission



Fig. 2. a) Fluorescent changes of DTE_{OF}-Me (10 μ M) after adding 0.0–2.0 equivalent CB[8]. $\lambda_{ex} = 400$ nm. (b) Fluorescent changes of DTE_{OF}-Me/CB[8] upon bubbling N₂. (c) Fluorescent changes of DTE_{OF}-Me/CB[8] under different temperatures. (d) Time-resolved emission spectroscopy of DTE_{OF}-Me/CB[8].

spectroscopy display one main emission at 550 nm (Fig. 2d), and the gated emission overlapped well with the photoluminescence (Fig. S23). All these results clearly confirmed that the emission of DTE_{OF} -Me/CB[8] at 550 nm was TADF. The lifetime of DTE_{OF} -Me was 1.65 ns, which was prolonged to 8.31 ns and 0.12 µs after adding CB [8] (Fig. S24). The shorter lifetime came from the singlet excited state of prompt fluorescence and the longer one resulted from TADF. The quantum yield also increased from 0.31% to 16.87% upon complexing with CB[8] for the hydrophobic cavity of CB[8] could reduce nonradiative energy consumption (Table S1).

The photochromism of diarylethene also had a significant influence on the photoluminescent properties. The green fluorescence of DTE_{OF}-Me/CB[7] disappeared when irradiated by 365 nm UV light but only showed negligible decrease after irradiating by 450 nm visible light for 5 s (Figs. S25a-b). Upon alternating irradiation with 365 and >600 nm light, the emission intensity at 500 nm could decrease or increase for several times (Fig. S25c). However, both the irradiation of 365 nm UV light and 450 nm visible light could lead to the yellow fluorescence of DTE_{OF}-Me/CB[8] sharply decreased, and the quenching efficiency was calculated to be 96% and 97%, respectively, by the changes of emission intensity at 550 nm (Fig. 3a and b). Subsequently, the emission intensity could completely recover with the irradiation of >600 nm light (Fig. S26). This process could also be repeated several times without apparent fading indicating the excellent fatigue resistance (Fig. 3c). The gated emission also decreased upon 450 nm light irradiation suggesting the efficiently photo-controlled TADF (Fig. S27).

Owing to the difference of photochromic rate between DTE-Me/CB[7] and DTE-Me/CB[8], the multicolor luminescence could be obtained by irradiating the mixed solution of DTE-Me/CB[7] and DTE-Me/CB[8] with a ratio of 3:2 (Fig. 3d). The mixture showed yellow fluorescence before 365 nm light irradiation because of CB [8] could greatly enhance the emission at 550 nm. Upon 365 nm UV light irradiation, DTE-Me/CB[8] converted into CF quickly, while most DTE-Me/CB[7] remain OF resulting in the emission color changing from yellow to cyan blue. With the prolonged irradiation time, DTE-Me/CB[7] also converted into CF and the emission color turned into blue finally. The emission color changes were shown in

corresponding CIE diagrams (Fig. 3e). Besides, the application for information encryption was also demonstrated (Fig. 3f). The solution of DTE-Me/CB[8] could be used as fluorescent ink to draw pattern in paper. When irradiated by 450 nm visible light, the pattern disappeared, and the disappeared pattern could recover after > 600 nm light irradiation to realize information encryption.

Considering the excellent photoluminescence and photochromic properties of DTE-Me/CB[8], SRB and Cy5 as acceptors were loaded into supramolecular assembly to achieve photoswitchable two-step sequential energy transfer process. The TEM images of DTE-Me/CB[8] showed the topological morphology of nanorod (Fig. 4a and S28a). After co-assembly with SRB, the topological morphology turned into nanoparticles with diameter of 0.46 µm (Fig. 4b and S28b-c). After adding the second dye Cy5, the diameter of the nanoparticles slightly reduced (Fig. 4c and S28d). These changes of topological morphology suggested the coassembly process, which was in favor of energy transfer. As outlined in Fig. 4d, the fluorescence emission of DTE_{OF}-Me/CB[8] perfectly matched with the absorption band of SRB, which is an essential requirement for the fluorescence resonance energy transfer (FRET). With the increasing concentration of SRB, the fluorescent emission of DTE_{OF}-Me/CB[8] at 550 nm decreased; meanwhile, the emission of SRB at 596 nm increased (Fig. 4e). However, the emission of DTE-Me/CB[8] was too broad, leading to insignificant emission enhancement of SRB at 600 nm. Hence, we gave the normalized photoluminescence of DTE_{OF}-Me/CB[8] with different concentrations of SRB to make these changes apparent (Fig. S29). The fluorescence overlapped with the gated emission indicated that the TADF could also undergo energy transfer process (Fig. 4f). The fluorescence of SRB under the same concentration and excitation wavelength was extremely weak implying the efficient energy transfer process (Fig. S29b). Upon 450 nm visible light irradiation, the emission intensity of DTE_{OF}-Me/CB[8]@SRB decreased and the quenching efficiency was calculated to be 81% through the emission intensity at 596 nm (Fig. S30a). The quenched fluorescence could be ascribed to the generation of CF diarylethene isomer. Besides, the fluorescent intensity could also recover to initial state upon >600 nm light irradiation (Fig. S30b).



Fig. 3. Fluorescent changes of DTE_{OF} -Me/CB[8] upon (a) 365 nm UV light and (b) 450 nm visible light irradiation. (c) Fatigue resistance of DTE-Me/CB[8] (10 μ M) upon alternating 450 and > 600 nm visible light irradiation. (d) Fluorescent changes of the mixture of DTE_{OF} -Me/CB[7] and DTE_{OF} -Me/CB[8] upon 365 nm UV light irradiation ([DTE_{OF} -Me]:[CB[7]]: [CB[8]] = 5:3:2) and (e) the corresponding CIE 1931 chromaticity diagrams. (f) The application of information encryption for DTE-Me/CB[8].

C. Wang and Y. Liu



Fig. 4. TEM images of (a) DTE-Me/CB[8], (b) DTE-Me/CB[8]@SRB, and (c) DTE-Me/CB[8]@SRB@Cy5. (d) The emission of DTE_{OF}-Me/CB[8]@SRB, Cy5, and the absorption of SRB and Cy5. (e) Photoluminescence of DTE_{OF}-Me/CB[8](20 μM) with different concentrations of SRB. (f) Photoluminescence and gated emission of DTE-Me/CB[8]@SRB. (g) Fluorescent emission spectral change of DTE_{OF}-Me/CB[8]@SRB@Cy5. (i) Fluorescent emission spectral change of DTE_{OF}-Me/CB[8]@SRB@Cy5. (j) Fluorescent emission spectral change of DTE_{OF}-Me/CB[8]@SRB@Cy5. (i) Fluorescent emission spectral change of DTE_{OF}-Me/CB[8]@SRB@Cy5. (j) Fluorescent emission spectral change

Subsequently, we further loaded another NIR dyes (Cy5) as the second acceptor into the DTE-Me/CB[8]@SRB. With the gradual addition of Cy5, the emission intensity at 596 nm decreased because the absorption of Cy5 overlapped well with the emission of SRB (Fig. 4g). The photoluminescence of DTE-Me/CB[8]@SRB also overlapped with the gated emission (Fig. 4h). In a control experiment, free Cy5 barely fluoresced when excited at 400 nm (Fig. S31). The NIR emission at 670 nm of Cy5 could also be quenched and recovered by 450 and >600 nm visible light, respectively (Fig. 4i and S32). The lifetime of DTE-Me/CB[8] ($\tau = 0.12 \ \mu$ s) was decreased upon addition of SRB to form DTE-Me/CB[8]@SRB ($\tau = 0.07 \ \mu$ s), and then further decreased upon addition of Cy5 to form DTE-Me/ CB[8]@SRB@Cy5 ($\tau = 0.06 \ \mu$ s) due to the two-step sequential energy transfer process (Fig. S33).

3. Conclusion

In conclusion, a supramolecular photo-switch with a higher photochromic rate and enhanced TADF was constructed, in which CB[8] could tightly bind and activated DTE-Me, resulting in the efficient visible-light-induced photocyclization. The presence of CB [8] could enhance the intermolecular charge-transfer interaction and shift the absorption of DTE-Me to visible region, leading to the photocyclization reaction that can be triggered by 450 nm visible light. Interestingly, the multicolor luminescence was achieved due to the different photochromic rates. Besides, the strong TADF induced by CB[8] is not only used as fluorescent ink for information encryption but also served as donor to sequentially transfer the energy to acceptor dye SRB and Cy5. This energy transfer process could also be regulated by visible light. This supramolecular TADF photo-switch may pave a simple way to design more intelligent luminescent materials.

Author contribution statement

Conghui Wang: investigation, data curation, writing the original draft.

Yu Liu: conceptualization, supervision.

Data availability

The raw data required to reproduce these findings are available to download from [INSERT PERMANENT WEB LINK(s)]. The processed data required to reproduce these findings are available to download from [INSERT PERMANENT WEB LINK(s)].

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.mtchem.2022.100954.

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