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Light-fueled dissipative self-assembly at molecular and macro-scale enabled by a visible-light-responsive transient hetero-complementary quadruple hydrogen bond

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ABSTRACT

The development of out-of-equilibrium self-assembly systems using light as input fuel is highly desirable and promising for the fabrication of smart supramolecular materials. Herein, we report the construction of new artificial light-fueled dissipative molecular and macroscopic self-assembly systems based on a visible-light-responsive transient quadruple H-bonding array, which consists of an azobenzene-modified ureidopyrimidinone (UPy) module (Azo-O-UPy) and a nonphotoactive diamidonaphthyridine (DAN) derived competitive binder (Napy-1). The visible light (410 nm) irradiation can induce the *E* to *Z* isomerization of the azobenzene unit of *E*-Azo-O-UPy to produce *Z*-Azo-O-UPy with an opened UPy binding site, which can complex with Napy-1 to form a quadruply H-bonded heterodimer. The heterodimer is metastable and can be quickly disassembled in dark, owing to the fast thermal relaxation of *Z*-Azo-O-UPy to *E*-Azo-O-UPy. While introducing such transient quadruple H-bonding interaction into a linear polymer system or a polymeric gel system, light-fueled out-of-equilibrium polymeric assembly both at molecular and macro-scale can be achieved.

Supramolecular self-assembly has been applied as a powerful bottom-up approach in the fabrication of numerous self-assembled systems and materials with great complexity and functions over the past few decades [1–13]. However, so far, most supramolecular self-assembly systems are residing in the thermodynamic equilibrium state. On the contrary, living systems in nature can ubiquitously generate self-assembled architectures (such as protein tubulin) far from thermodynamic equilibrium by continuously dissipating energy from external environment. Such fuel-driven dissipative self-assembly (DSA) processes play essential roles in living systems, contributing both to maintain their sophisticated dynamic structures and perform amazing biofunctions (such as homeostasis, motility and camouflage). Attracting by this peculiarity, in recent years, a strong interest has sparked in the development of artificial DSA systems [14–21], which can not only underpin a better understanding of living systems but also provide new access to novel life-like systems possessing structural adaption as well as biomimetic soft materials with programmable functions. However, despite the increasing efforts, the exploration of DSA systems is still in its infancy, and to achieve such systems remains an aspirational, but fundamentally challengeable task in chemistry and materials science.

In general, chemical fuels such as ATP [22,23], carbodiimide reagents [24], acid-base [25], redox reagents [26], as well as other fuel sources including biomolecules [27,28], chemical or electrode reaction [29,30], ultrasound [31], shearing force [32] and light [33–38], are used as inputting energies to supply a DSA process. Among these, although most DSA systems are driven by chemical fuels, light has such advantages as inputting energy source for the clean and spatiotemporal controllability features [39–46], which make the unique light-fueled DSA systems being especially attractive. However, up to now the light-driven DSA process is quite rare in reports, owing to the challenges in designing efficient light-triggered transient noncovalent interactions.

Recently, we have proposed a reversible photo-locking strategy for the construction of unique photoswitchable self- and hetero-

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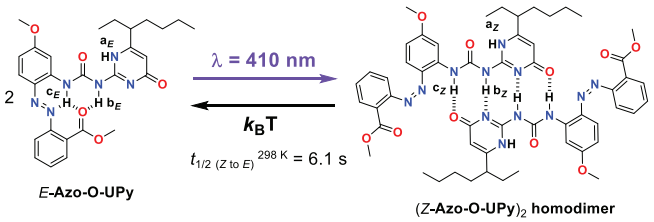
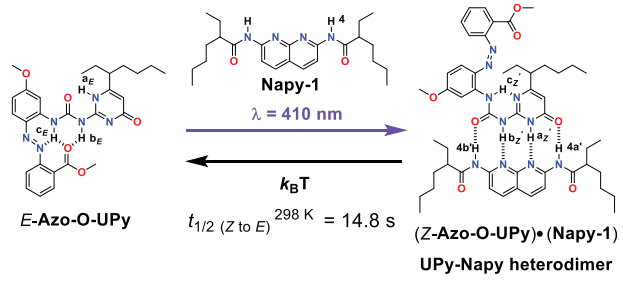


Scheme 1. Schematic representation of light-driven transient self-complementary quadruple H-bonding of Azo-O-UPy.

complementary quadruple H-bonding arrays which can exhibit significant ON/OFF switching ratio of dimerization binding strength upon different light irradiations [47,48]. Herein, we demonstrate that a photoresponsive quadruple H-bonding motif with a short-lived ($t_{1/2}$ (Z) = 6.1 s at 298 K) Z-isomer can be constructed by introducing a methoxyl-modified azobenzene unit into the UPy moiety as the "photo-locker" (Azo-O-UPy in Scheme 1). After visible light (410 nm) irradiation, the obtained Z-Azo-O-UPy not only can form a homodimer by the self-complementary quadruple H-bonding between the opened UPy units, it also can form hetero-complementary quadruple H-bonds with a nonphotoactive competitive binder (Napy-1) to give a heterodimer (Scheme 2). Notably, both the homodimer and heterodimer are found to quickly dissociate in the solution upon thermal relaxation in dark at ambient condition. Moreover, the unique ability of light-driven transient hetero-dimerization of Azo-O-UPy and Napy-1 can further enable the fabrication of unprecedented light-fueled polymeric DSA systems at molecular and macro-scale.

The complexation behavior of Azo-O-UPy before light irradiation was firstly investigated by recording its concentration-varied ^{1}H NMR spectra (Fig. S1 in Supporting information). The N–H protons in the UPy unit of E-Azo-O-UPy were found to appear at 11.9 (H-a_E), 9.9 (H-b_E) and 12.6 ppm (H-c_E), respectively, the chemical shift values of which were almost the same to those observed in the previously reported E-Azo-UPy monomer [47]. This indicates that the pristine E-Azo-O-UPy is existing as a closed conformation with its partial UPy recognition site locked by the intramolecular H-bonds (Scheme 1, left), which can restrict the E-Azo-O-UPy molecule from dimerization through intermolecular quadruple H-bonds. To further reveal the dimerization inhibition efficiency of E-Azo-O-UPy, diluted ^{1}H NMR experiments were performed. Only one set of proton signals with very small chemical shift changes (-0.09 ppm $< \Delta\delta < 0$) were observed as the concentration of E-Azo-O-UPy increased (Fig. S1). This suggests that E-Azo-O-UPy is unable to form a quadruply H-bonded homodimer in the chloroform solution. Such dimerization inhibition is further investigated by increasing the solution polarity, during which the ^{1}H NMR spectra of E-Azo-O-UPy only reveal one set signals with small chemical shift changes (Fig. S2 in Supporting information), rather than displaying two sets of signals corresponding to the homodimer of



Scheme 2. Schematic representation of light-driven transient hetero-complementary quadruple H-bonding of Azo-O-UPy and Napy-1.

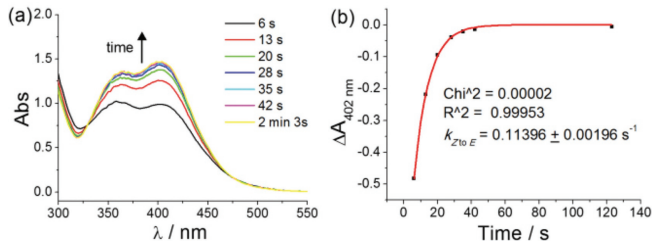


Fig. 1. (a) Time lapse absorption spectra of purple light-irradiated Azo-O-UPy (0.10 mmol/L) in $CHCl_3$ at 298 K under dark, (b) plot of the change in absorption: $\Delta A_{402\ nm} = A_{402nm} - A_{402nm\ (before\ purple\ light\ irradiation)}$ *versus* the recording time.

(E-Azo-O-UPy)$_2$ and free E-Azo-O-UPy, respectively. These results indicate that the locked conformation of E-Azo-O-UPy can effectively impede it from self-dimerization by intermolecular quadruple H-bonding interaction.

Upon purple light (λ = 410 nm) irradiation, the $E \rightarrow Z$ photoisomerization of Azo-O-UPy can be triggered to give the Z-Azo-O-UPy (Fig. 1). However, the obtained Z-Azo-O-UPy could be quickly converted back to its original E-isomer in the chloroform solution under thermal relaxation in dark as revealed by UV–vis spectroscopic experiment, from which the lifetimes of Z-Azo-O-UPy were calculated as $t_{1/2}$ ($Z \rightarrow E$) = 6.1 s at 25 °C (Fig. 1b). In addition, the recorded UV–vis spectra of alternative visible-light (410 nm) irradiation and thermal relaxation in dark show good reversibility and light fatigue resistance for the E/Z isomerization process of Azo-O-UPy (Fig. S4 in Supporting information). Nevertheless, the short lifetime of Z-Azo-O-UPy discourages us from investigating its quadruply H-bonded dimerization behavior at ambient condition (Scheme 1).

As a result, the ^{1}H NMR spectroscopic experiments at low temperature were then carried out. As revealed in Fig. 2 and Fig. S6 (Supporting information), when the solution of E-Azo-O-UPy was irradiated by the purple light (410 nm) at 233 K, 51% of E-Azo-O-UPy was found to be converted to Z-Azo-O-UPy which could form quadruply H-bonded homodimer of (Z-Azo-O-UPy)$_2$ (Scheme 1). This can be supported by that the N–H protons signals (H-a_Z, H-b_Z and H-c_Z) of Z-Azo-O-UPy at 233 K were appeared at 13.0, 12.8 and 12.7 ppm (Fig. 2b), respectively. These signals are shifted downfield significantly while comparing to pristine E-Azo-O-UPy, but very close to those observed in previous reported homodimer of (Z-Azo-UPy)$_2$ [47]. When the purple-light-irradiated mixture solution of Azo-O-UPy was further treated at room temperature in dark, the formed homodimer of (Z-Azo-O-UPy)$_2$ was disassembled quickly as revealed by the recovery of proton signals of pristine

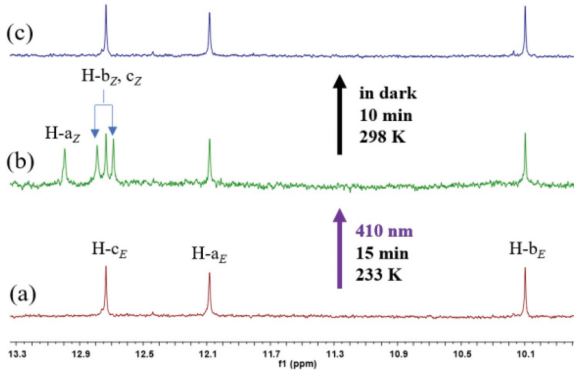


Fig. 2. Partial ^{1}H NMR spectra (600 MHz, 0.25 mmol/L, $CDCl_3$, 233 K) of the solution of (a) E-Azo-O-UPy, (b) the PSS$_Z$ (410 nm) mixtures of Azo-O-UPy at 233 K, and (c) the PSS$_Z$ (410 nm) mixtures of Azo-O-UPy at 233 K after keeping in the dark for 10 min at 298 K.

E-Azo-O-UPy (Fig. 2c). These results indicate that Azo-O-UPy can form transient quadruple H-bonds upon purple light (410 nm) irradiation and thermal relaxation in dark.

The hetero-complementary H-bonding between Azo-O-UPy and nonphotoactive Napy-1 is further evaluated (Scheme 2). Before light irradiation, a small complexation constant ($K_{dim}^{298K} = 67$ L/mol) of *E*-Azo-O-UPy and Napy-1 is obtained based on the ^{1}H NMR titration experiments at room temperature (Fig. S7 in Supporting information). Comparing to the previously reported significant dimerization constant ($K_{dim}^{298K} > 1 \times 10^5$ L/mol) of UPy-DAN pair after light irradiation [47], we can conclude that the dimerization of *E*-Azo-O-UPy and Napy-1 is greatly suppressed at pristine state (Scheme S2 in Supporting information). When the mixture solution of *E*-Azo-O-UPy and Napy-1 was irradiated by purple light (410 nm), the *E*→*Z* photoisomerization of *E*-Azo-O-UPy component in the mixture could be triggered to generate *Z*-Azo-O-UPy, which could spontaneously dimerize with Napy-1 by forming intermolecular hetero-complementary quadruple H-bonds (Scheme 2). However, the obtained heterodimer of (*Z*-Azo-O-UPy)·(Napy-1) has a lifetime of $t_{1/2}$ (*Z*→*E*) = 14.8 s at 25 °C (Fig. S8 in Supporting information), which is too short for us to get the insight of hetero-dimerization of Azo-O-UPy and Napy-1 from ^{1}H NMR spectroscopic investigation under room temperature. Therefore, the ^{1}H NMR spectra for the solution of Azo-O-UPy and Napy-1 at low temperature of 233 K were then recorded.

As revealed in Fig. 3 and Fig. S9 (Supporting information), when the mixture solution of pristine Azo-O-UPy and Napy-1 was irradiated by purple light (410 nm) at 233 K, significant newly-appeared proton signals (H-az′, H-bz′ and H-cz′ in Fig. 3b) corresponding to the heterodimer of (*Z*-Azo-O-UPy)·(Napy-1) were observed from the ^{1}H NMR spectrum of the resulting PSS$_Z$ (410 nm) mixtures. This indicates the photo-triggered formation of quadruply H-bonded heterodimer of (*Z*-Azo-O-UPy)·(Napy-1) is efficient, though Azo-O-UPy reveals a moderate photoisomeric ratio (*Z*/*E* = 58/42) in the mixture during the purple light irradiation process. Furthermore, the obtained heterodimer is dissociated upon thermal relaxation at room temperature in dark (Fig. 3c). These results clearly show that the light-fueled transient hetero-complementary quadruple H-bond can be constructed from Azo-O-UPy and Napy-1.

To further reveal the potential application of using such light-fueled transient hetero-complementary quadruple H-bonding array of Azo-O-UPy and Napy-1 for the construction of light-fueled dissipative supramolecular self-assembly systems, linear polymers with side chains incorporating the Azo-O-UPy unit (Azo-O-UPy-P) and Napy-1 unit (Napy-P) were then synthesized (Fig. 4). Time-

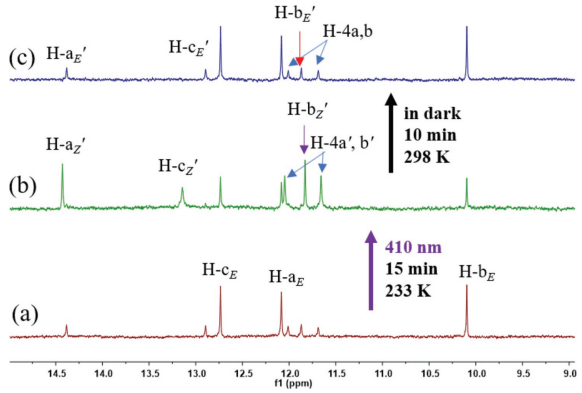


Fig. 3. Partial ^{1}H NMR spectra (600 MHz, $CDCl_3$, 233 K) of the solution of (a) *E*-Azo-O-UPy (0.25 mmol/L) and Napy-1 (0.25 mmol/L), (b) the PSS$_Z$ (410 nm) mixtures of Azo-O-UPy (0.25 mmol/L) and Napy-1 (0.25 mmol/L) at 233 K, and (c) the PSS$_Z$ (410 nm) mixtures of Azo-O-UPy (0.25 mmol/L) and Napy-1 (0.25 mmol/L) at 233 K after keeping in the dark for 10 min at 298 K.

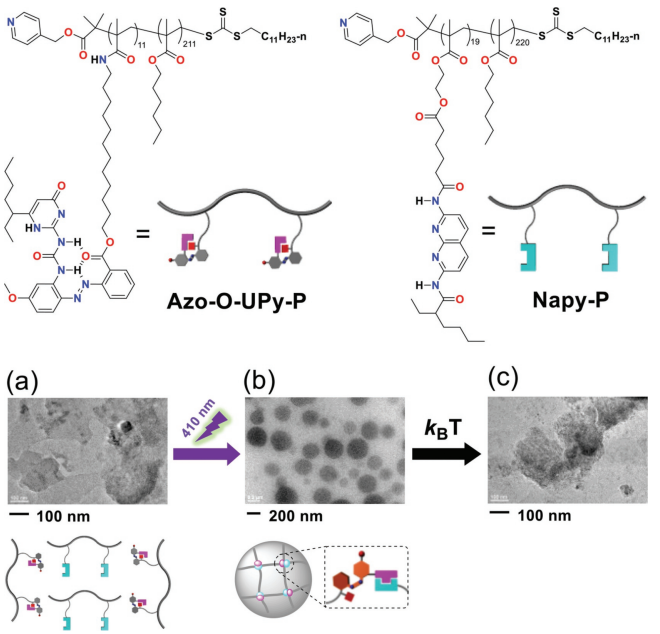


Fig. 4. The chemical structures of Azo-O-UPy-P and Napy-P, and the TEM images of the sample obtained by evaporating the solution of Azo-O-UPy-P (0.13 mg/mL) and Napy-P (0.083 mg/mL) in $CHCl_3$ under conditions of (a) before light irradiation, (b) after irradiation by purple light (410 nm) for 2 min, (c) the purple light irradiated solution followed by thermal equilibration over 1 h for 20 min at 25 °C.

dependent UV–vis spectra of the chloroform solution of Azo-O-UPy-P and Napy-P after purple light irradiation were recorded, and the half lifetime for the thermal relaxation of *Z*-Azo-O-UPy-P was calculated as $t_{1/2}$ (*Z*→*E*) = 619 s at 25 °C (Fig. S10 in Supporting information).

This value is about 101 times to that of *Z*-Azo-O-Upy ($t_{1/2}$ (*Z*→*E*) = 6.1 s at 25 °C), suggesting the hetero-complementary quadruple H-bonds formed in the polymeric self-assemblies has an increased thermal stability. In addition, the *E*/*Z* isomerization process of Azo-O-UPy-P component in the polymeric assembly system is found to exhibit good reversibility and light fatigue resistance (Fig. S11 in Supporting information). These features facilitate the investigation of dissipative self-assembly behavior of Azo-O-UPy-P and Napy-P at ambient condition.

Before light irradiation, there is no significant Tyndall phenomenon for the mixture solution of *E*-Azo-O-UPy-P and Napy-P (Fig. S12a in Supporting information). Besides, only small sized entity with diameter of 4.2 nm is observed from the dynamic light scattering (DLS) profile of this non-irradiated solution (black curve in Fig. S13 in Supporting information), and the TEM image also reveals ill-defined morphology of Azo-O-UPy-P and Napy-P in the chloroform (Fig. 4a). These results suggest that the pristine *E*-Azo-O-UPy-P with locked UPy unit does not form macromolecular assemblies with Napy-P. However, upon purple light (410 nm) irradiation, the solution of Azo-O-UPy-P and Napy-P exhibits obvious Tyndall phenomenon (Fig. S12b in Supporting information), indicating the formation of polymeric aggregates in the solution. This is further supported by the observation of polymeric nanoparticles with an average size of 220 nm from the dynamic light scattering (DLS) profile (red curve in Fig. S13) and TEM image (Fig. 4b) of the obtained PSS$_Z$ (410 nm) mixture solution. The generation of polymeric nanoparticles can be attributed to the formation of transient hetero-complementary quadruple H-bonded UPy-DAN crosslinks between the polymer chains of *Z*-Azo-O-UPy-P and Napy-P (Fig. 4b) [49]. Moreover, when treating the purple light (410 nm) irradiated solution of Azo-O-UPy-P and Napy-P in the dark at room temperature, the Tyndall phenomenon is vanished

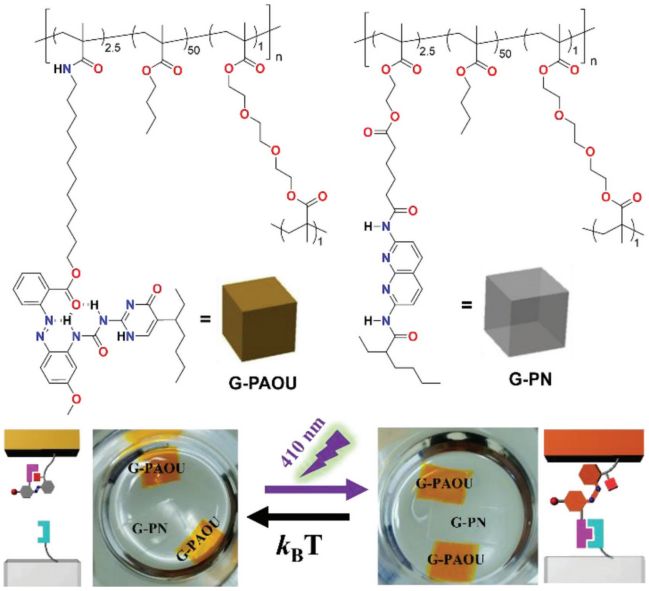


Fig. 5. The chemical structures of the crosslinked polymers with side chains containing Azo-O-UPy unit (G-PAOU) or DAN unit (G-PN), and the photographs and schematic representation of the light-fueled dissipative macroscopic assembly behavior of polymeric gels fabricated from G-PAOU and G-PN in $CHCl_3$.

significantly (Fig. S12c in Supporting information), and the polymeric nanoparticles in the solution are dissociated (Fig. 4c). This can be attributed to the reversed *Z* to *E* isomerization of the Azo-O-UPy unit in the polymeric system during the thermal relaxation process.

Given that the hetero-complementary multiple H-bonding motifs are distinguished with the unique ability of associating two different partners with high fidelity and tunable affinity, we anticipate such light-driven transient hetero-complementary quadruple H-bonding array of Azo-O-UPy and Napy-1 is highly desirable to achieve attracting light-fueled quadruply H-bonded macroscopic self-assembly process, which is hardly constructed yet. To test this idea, polymeric gels of G-PAOU and G-PN were then prepared from the crosslinked polymers incorporated with Azo-O-UPy unit and DAN unit (Fig. 5), respectively. While the gel pieces of G-PAOU and G-PN were mixing and shaking together in $CHCl_3$ under purple light (410 nm) irradiation, they adhered with each other to form self-assembled aggregate (Movie S1 in Supporting information), which could be attributed to the formation of interfacial quadruple H-bonds between the *Z*-Azo-O-UPy unit of G-PAOU and DAN unit of G-PN (Fig. 5). Moreover, when such self-assembled gel pieces were kept in the dark, they were separated gradually to give the pristine detached ones (Movie S2 in Supporting information). This can be owed to the thermal-induced dissociation of hetero-complementary quadruple H-bonds at the interfaces between the gel pieces. The above results show that the established light-driven transient hetero-complementary quadruple H-bond of Azo-O-UPy and Napy-1 is indeed applicable for the construction of light-fueled out-of-equilibrium macroscopic self-assembly system.

In summary, we describe a unique type of hetero-complementary quadruple H-bonding array consisting of photoresponsive Azo-O-UPy and nonphotoactive Napy-1, between which transient intermolecular quadruple H-bonds can be formed upon purple light (410 nm) irradiation, and then quickly dissociated *via* thermal relaxation. The light-driven bonding and fast thermally debonding in dark of such quadruple H-bonding motif are carefully evaluated. Subsequently, this ability is applied in the fabrication of light-fueled nonequilibrium multiple H-bonded self-assembly systems including molecular-scaled polymers and macroscopic polymeric gel subjects. Noting the widespread use of quadruple H-bonding interaction in supramolecular chemistry and materials science, we envisage that such light-fueled quadruple H-bonded DSA systems will offer new opportunities in the quest for smart functional supramolecular materials.

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.

Acknowledgments

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.06.062.

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