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A highly efficient light-harvesting system with sequential energy transfer based on a multicharged supramolecular assembly[†]

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A supramolecular assembly was constructed by the nonconvalent interaction of pillar[5]arene (WP5) with a pyridinium modified tetraphenylethene (Py-TPE) derivative, in which Py-TPE/WP5 acted as a donor, and sulforhodamine 101 and sulfonated aluminum phthalocyanine acted as acceptors to realize a highly efficient light-harvesting system with two-step sequential energy transfer.

Light-harvesting with the conversion of light energy to chemical energy has received considerable attention on account of its various applications in solar cells,¹ photocatalysis,² luminescent materials,³ bioimaging and photodynamic therapy.⁴ In a natural lightharvesting system, over 200 chromophores act as antennae to absorb sunlight and transfer the light energy to an acceptor of the reaction center.⁵ In order to design an effective artificial light-harvesting system in aqueous solution, some indispensable criteria need to be considered. The donor antenna chromophores not only require tight stacking and orderly spatial arrangement without aggregationcaused quenching (ACQ), but also possess distinct luminescence properties. Then spectral matching and suitable distance between the donor and acceptor could minimize the energy loss. Moreover, each acceptor must be surrounded by multiple antenna chromophores.⁶ Recently, several scaffolds have been reported to develop artificial light-harvesting systems, such as metallacages,^{2b,7} multiporphyrin arrays,8 organic nanocrystals,9 organic polymers,10 supramolecular host-guest systems,11 etc. Recently, Stang and coworkers reported fluorescent hexagonal Pt(II) metallacycles as a platform to fabricate artificial light-harvesting systems.7 Cao et al. reported aggregation-induced emission and light-harvesting function of tetraphenylethene-based tetracationic dicyclophane.12 Tang and Cao et al. fabricated a conjugated polymeric supramolecular network and realized efficient light-harvesting with an ultrahigh antenna effect.13 We also developed efficient artificial

light-harvesting supramolecular nanoparticles in aqueous solution and exhibited excellent energy transfer efficiency.¹⁴

However, almost all of the artificial light-harvesting systems focused on conventional one-step energy transfer containing two chromophores from the donor to acceptor. Compared with the one-step energy transfer, the two-step energy transfer could realize broad-spectrum tunable photoluminescence and a larger Stokes shift between the donor and final acceptor even in the absence of spectral overlap. Therefore, it is still quite challenging to design an efficient artificial light-harvesting system with sequential energy transfer by supramolecular assembly. Herein, we design a novel efficient artificial lightharvesting supramolecular assembly with sequential energy transfer based on the non-covalent interaction of a water-soluble pillar[5]arene (WP5), pyridinium modified tetraphenylethene (Py-TPE) derivative, sulforhodamine 101 (SR101) and sulfonated aluminum phthalocyanine (AlPcS₄) in aqueous solution (Scheme 1). Benefitting from the aggregation-induced emission (AIE) properties of Py-TPE, WP5 could induce the aggregation of Py-TPE and result in aggregation induced emission enhancement (AIEE) due to the tight and orderly spatial organization,



Scheme 1 Construction of a light-harvesting system with sequential energy transfer in aqueous solution.

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which makes the Py-TPE/WP5 assembly as an excellent donor chromophore. Then sulforhodamine 101, Eosin Y or Nile Red acting as a first energy acceptor respectively could be loaded into the hydrophobic layer of the Py-TPE/WP5 supramolecular assembly, and achieve highly efficient energy transfer in aqueous solution at a donor/acceptor ratio of 150:1. Subsequently, with SR101 as a bridge acceptor and AlPcS₄ as the second acceptor, two-step sequential energy transfer took place from Py-TPE/WP5 to SR101 and then to AlPcS₄ accompanied by fluorescence change from blue to NIR emission, and the Stokes shift reached 340 nm. Compared with the previously reported light-harvesting system,^{2a} the supramolecular assembly exhibits sequential energy transfer with a high donor/acceptor ratio, antenna effect and energy transfer efficiency.

It is well known that tetraphenylethene (TPE) possesses aggregation-induced emission (AIE) properties and is nonfluorescent in an isolated solubilized state but exhibits enhanced emission upon aggregation due to the restriction of intramolecular rotations.¹⁵ Combining the advantages of AIE, we constructed a unique water-soluble fluorescence supramolecular assembly between WP5 and Py-TPE via electrostatic interactions and π - π stacking. In order to research the behavior of aggregation-induced emission enhancement (AIEE), a fluorescence titration experiment was performed. Upon increasing the concentration of WP5, the fluorescence intensity of Py-TPE significantly enhanced and increased 25 times compared with free Py-TPE (Fig. 1a). Because of the obvious AIEE, the fluorescence quantum yield significantly increased. The fluorescence quantum yield of free Py-TPE was measured to be 1.68%, however that of the Py-TPE/WP5 system was increased to 6.21%. Then we studied the effect of different macrocycles on the luminescence behavior of Py-TPE. The addition of anionic cyclodextrin (COOH-\beta-CD) also induced emission enhancement (14 times), and the fluorescence intensity was nearly unchanged with the addition of cucurbit[7]uril (CB[7]) (Fig. S5, ESI[†]). Compared with other macrocycles, the negative charge density of WP5 was higher and the Py-TPE/WP5 assembly exhibited a stronger aggregation ability and fluorescence enhancement. It follows that the fluorescence enhancement stems from the formation of π -stacked aggregates of Py-TPE by the negatively charged macrocyclic host; the supramolecular aggregates were compact stacking to restrict the intramolecular rotation of the phenyl rings and thus show AIEE.

Similar to many other induced aggregation assemblies by charged macrocyclic molecules reported before,¹⁶ Py-TPE could



Fig. 1 (a) Fluorescence spectra of Py-TPE (20 μ M) with the addition of WP5 in aqueous solution (λ_{ex} = 350 nm), inset: photographs of Py-TPE and Py-TPE/WP5 under UV light. (b) TEM images of the Py-TPE/WP5 assembly.

be induced by WP5 to form a supramolecular assembly via noncovalent interactions cooperatively, including electrostatic, π -stacking and hydrophobic interactions. To gain more insights into the process of assembly of WP5 and Py-TPE, optical transmittance, the Tyndall effect, transmission electron microscopy (TEM), zeta potential and dynamic light scattering (DLS) were investigated. In the presence of WP5, the optical transmittance at 450 nm versus the concentration of Py-TPE gradually decreased and an inflection point at 25 µM was observed (Fig. S6a and b, ESI[†]). Therefore, an aggregation induced CAC value of Py-TPE was 25 µM in the presence of WP5. However, the optical transmittance at 450 nm versus the concentration of Pv-TPE remained nearly unchanged without WP5 (Fig. S7, ESI[†]), revealing that WP5 plays a significant role in inducing the aggregation of Py-TPE. Moreover, the preferable mixing ratio between Py-TPE and WP5 was also investigated. By gradually increasing the concentration of WP5 to Py-TPE solution, the optical transmittance at 450 nm sharply decreased and then gradually recovered (Fig. S6c and d, ESI†). The sharp decrease of the optical transmittance revealed that a large supramolecular aggregate formed between Py-TPE and WP5. Then, the addition of an excess amount of WP5 resulted in the formation of a simple inclusion complex accompanied by the disassembly of the aggregate, resulting in an increase in the optical transmittance. As a result, the preferable molar ratio for the supramolecular assembly was WP5:Py-TPE = 1:10. A simple mixture of WP5 and Py-TPE in aqueous solution showed the obvious Tyndall effect (Fig. S8, ESI⁺), indicating the formation of a supramolecular assembly in solution. The DLS results illustrated the formation of a supramolecular assembly with an average hydrodynamic diameter of ca. 217 nm (Fig. S8e, ESI⁺), accompanied by a narrow size distribution. Moreover, the TEM image of the Py-TPE/WP5 supramolecular assembly showed a number of spherical nanoparticles with a diameter of ca. 50-200 nm (Fig. 1b). The zeta potential of Py-TPE/WP5 was measured as -10.4 mV (Fig. S8d, ESI^{\dagger}), suggesting that the surfaces of the nanoparticles were negatively charged.

Considering the excellent photoluminescence properties of Py-TPE/WP5 and the supramolecular assembly with an interior hydrophobic environment, appropriate organic dye molecules as acceptors could be loaded into supramolecular nanoparticles to avoid self-aggregation caused quenching and have a suitable distance with Py-TPE/WP5 as the donor to achieve efficient energy transfer. Sulforhodamine 101 (SR101), a hydrophobic dye, was selected as an energy acceptor to construct a supramolecular light-harvesting system. Firstly, the absorption band of SR101 overlaps well with the fluorescence emission of the Py-TPE/WP5 assembly (Fig. 2a), which is favorable for the Förster resonance energy transfer (FRET) process. Moreover, the Py-TPE/WP5 assembly with spatially well-organized chromophores by tight π -stacking of Py-TPE is a key parameter in facilitating the efficient energy transfer process, in which SR101 could be loaded into the hydrophobic layer of the assembly and ensure a suitable distance between the energy donor and acceptor. As shown in Fig. 2b, with the addition of SR101 to the Py-TPE/WP5 assembly, the fluorescence emission of



Fig. 2 (a) Normalized emission spectrum of Py-TPE/WP5, absorption and emission spectra of (a) SR101, (d) EY and (g) NiR. Fluorescence spectrum of Py-TPE/WP5 ([Py-TPE] = 20 μ M, [WP5] = 2 μ M) in water with different concentrations of (b) SR101 (inset: photographs of Py-TPE/WP5 and Py-TPE/WP5/SR101), (e) EY (inset: photographs of Py-TPE/WP5 and Py-TPE/WP5/NiR under UV light). The concentrations of SR101 or EY or NiR were 0.00 μ M, 0.01 μ M, 0.02 μ M, 0.04 μ M, 0.06 μ M, 0.08 μ M, 0.1 μ M, and 0.13 μ M. The fluorescence lifetime of (c) Py-TPE/WP5 and Py-TPE/WP5/SR101 or (f) Py-TPE/WP5/EY or (i) Py-TPE/WP5/NiR (λ_{ex} = 350 nm). ([Py-TPE] = 20 μ M, [WP5] = 2 μ M, [SR101] = [EY] = [NiR] = 0.13 μ M).

Py-TPE/WP5 at 480 nm decreased, meanwhile the emission of SR101 at 610 nm increased when excited at 350 nm, and an obvious fluorescence change was clearly observed by the naked eye from blue emission to red emission (donor/acceptor ratio from 2000:1 to 150:1). In a control experiment, free SR101 without Py-TPE/WP5 barely fluoresced when excited at 350 nm (Fig. S9, ESI⁺). The amplified emission of SR101 upon indirect excitation of the donor with 350 nm suggested efficient energy transfer from Py-TPE/WP5 to SR101 in aqueous solution. Subsequently, the fluorescence lifetime experiments were performed to further verify the energy transfer process (Fig. 2c and Fig. S12, ESI[†]). The decay curve of Py-TPE followed a double exponential decay with fluorescence lifetimes $\tau_1 = 0.93$ ns and $\tau_2 = 3.44$ ns at 480 nm (Fig. S13a, ESI⁺). In the Py-TPE/WP5 assembly, the fluorescence lifetimes increased to $\tau_1 = 1.10$ ns and $\tau_2 =$ 3.85 ns (Fig. S13b, ESI[†]). However, in the Py-TPE/WP5/SR101 triad system, the fluorescence lifetimes decreased to τ_1 = 0.86 ns and $\tau_2 = 2.80$ ns (Fig. S13c, ESI[†]). These results both demonstrated that the energy is successfully transfer from donor Py-TPE/WP5 to acceptor SR101. According to the fluorescence titration spectrum, the energy transfer efficiency was calculated to be 64% (Fig. S14, ESI[†]) and the antenna effect was calculated to be 13.0 (Fig. S17, ESI[†]) at a donor/acceptor ratio of 150:1.

Moreover, we further loaded other hydrophobic dyes (Eosin Y or Nile Red) as acceptors into the Py-TPE/WP5 assembly. The absorption spectra of Eosin Y (EY) and Nile Red (NiR) well overlapped with the emission of Py-TPE/WP5 (Fig. 2d and g, ESI†). As a result, efficient energy transfer from Py-TPE/WP5 to EY or NiR was realized in aqueous solution through fluorescence titration experiments (Fig. 2e and h). With gradual



Fig. 3 (a) Normalized emission spectrum of Py-TPE/WP5 and SR101 and absorption and emission spectra of AlPcS₄. (b) Fluorescence spectrum of Py-TPE/WP5/SR101 ([Py-TPE] = 20 μ M, [WP5] = 2 μ M, [SR101] = 0.13 μ M) in water with different concentrations of AlPcS₄.

addition of EY to Py-TPE/WP5 assembly solution, the energy transfer efficiency was calculated to be 83.5% (Fig. S15, ESI[†]) and the antenna effect was calculated to be 7.0 (Fig. S18, ESI[†]) at a donor/acceptor ratio of 150:1. Similarly, when NiR was used as an energy acceptor, the energy transfer efficiency was calculated to be 74.3% (Fig. S16, ESI[†]) and the antenna effect was calculated to be 74.3% (Fig. S19, ESI[†]). The term efficiency was calculated to be 5.6 (Fig. S19, ESI[†]). Free EY or NiR without Py-TPE/WP5 barely fluoresced when excited at 350 nm (Fig. S10 and S11, ESI[†]). The time-resolved fluorescence was also measured (Fig. S12 and S13, ESI[†]). The decreased fluorescence lifetime indicated that the energy transferred from Py-TPE/WP5 to EY or NiR. Importantly, these dyes also demonstrated the practicality and universality of the Py-TPE/WP5 assembly as a donor to fabricate light-harvesting systems.

As mentioned above, we chose a NIR dye sulfonated aluminum phthalocyanine (AlPcS₄) as the second acceptor to achieve the twostep sequential energy transfer process. Because the absorption of AlPcS₄ overlaps with the emission of SR101 (Fig. 3a), there is a possible sequential energy transfer from Py-TPE/WP5 to SR101 and subsequently from Py-TPE/WP5/SR101 to AlPcS₄. When the second acceptor AlPcS4 was gradually added into Py-TPE/WP5/ SR101 assembly solution, the fluorescence intensity at 610 nm decreased and a new emission band at 690 nm appeared simultaneously under 350 nm excitation (Fig. 3b), and the Stokes shift reached 340 nm. The energy transfer efficiency and antenna effect were calculated to be 84.2% and 11.6 respectively (Fig. S20 and S21, ESI[†]) at a Py-TPE/WP5:SR101:AlPcS₄ molar ratio of 20 000:133:15. Furthermore, time-resolved fluorescence was also measured to further investigate the energy transfer process from Py-TPE/WP5/SR101 to AlPcS₄. The fluorescence lifetime of Py-TPE/ WP5/SR101 assembly was $\tau = 4.03$ ns, with the addition of AlPcS₄, the fluorescence lifetime was decreased to τ = 2.99 ns (Fig. S22, ESI[†]). The fluorescence quantum yield of the Py-TPE/WP5/SR101 assembly and Py-TPE/WP5/SR101/AlPcS4 assembly was 7.3% and 5.0% (Fig. S23, ESI⁺). In contrast, the fluorescence intensity barely changed upon the addition of AlPcS4 to the Py-TPE/WP5 assembly in the absence of SR101 (Fig. S24, ESI⁺), indicating that the donor cannot directly transfer energy to AlPcS4 due to the marginal overlap between the absorption of AlPcS4 and the emission of Py-TPE/WP5. Moreover, only free AlPcS₄ yielded negligible fluorescence under the same conditions (Fig. S27, ESI†), which ruled out the possibility for direct excitation. These results jointly suggested that a two-step sequential energy transfer



Fig. 4 (a) The CIE chromaticity diagram of photoluminescence color changes by varying the ratios of chromophores. The fluorescence spectra of Py-TPE/WP5 with different concentrations of (b) SR101, (c) EY, and (d) NiR. (e) The fluorescent inks based on a light-harvesting system: photographs under natural light and (f) under UV light of the painting. (g) Photograph of writing "N" with SR101 merely and rewriting "N" with Py-TPE/WP5 solution under UV light.

occurred from Py-TPE/WP5 to SR101 and then to AlPcS4. Similarly, the fluorescence titration experiments were also performed with Py-TPE/WP5/EY or Py-TPE/WP5/NiR upon the addition of $AlPcS_4$ (Fig. S25 and S26, ESI†). However, they both exhibited low energy transfer efficiency. The above results confirmed that SR101 could act as a crucial bridge acceptor to achieve sequential energy transfer in aqueous solution.

Based on the abovementioned points, the efficient lightharvesting system could be used as fluorescence ink. Upon painting the pre-prepared solutions on filter paper, no apparent color was observed under natural light (Fig. 4e), but obvious different colors were observed under UV light (Fig. 4f). On the basis of efficient energy transfer from Py-TPE/WP5 to SR101, fluorescent security ink was fabricated. As illustrated in Fig. 4g, when writing "N" with SR101 merely, no appreciable emission was observed. However, rewriting "N" with Py-TPE/WP5 solution gives prominent red emission. Therefore, we achieved data encryption and anti-counterfeiting by utilization of efficient energy transfer.

In conclusion, we have developed a highly efficient artificial light-harvesting system based on a supramolecular assembly. WP5 induced aggregation of Py-TPE to form supramolecular nanoparticles, which exhibited an excellent aggregation induced emission enhancement effect. The AIEE-active supramolecular assembly as an antenna donor could load a hydrophobic dye (SR101 or EY or NiR) to achieve highly efficient energy transfer respectively. Furthermore, we chose SR101 as the first energy acceptor and AlPcS₄ as the second energy acceptor to achieve two-step sequential energy transfer from Py-TPE/WP5/SR101 to AlPcS4. Most importantly, the highly efficient aqueous light-harvesting system provides great potential application to mimic the natural photosynthesis.

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Conflicts of interest

There are no conflicts to declare.

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