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Artificial Light-harvesting Supramolecular Polymeric Nanoparticles Formed by Pillar[5]arene-based Host–guest Interaction

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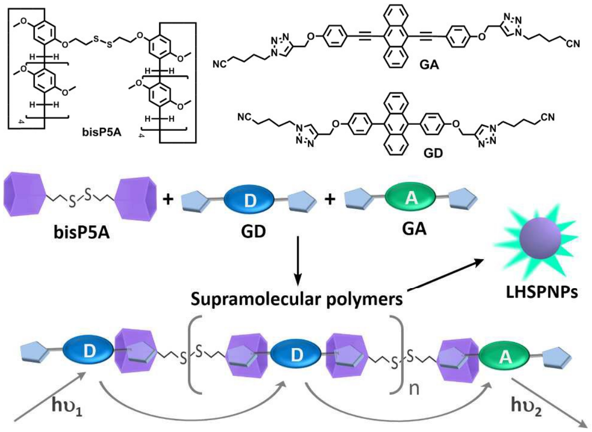
Artificial light-harvesting nanoparticles were prepared from supramolecular polymers in which pillar[5]arene linked anthracene-derived donors and acceptors by host-guest interactions. The resulting water-dispersible nanoparticles displayed efficient energy transfer and excellent light harvesting ability in part because the steric bulk of pillar[5]arene suppressed self-quenching of the chromophores.

Photosynthesis starts with efficient absorption of sunlight by the light-harvesting system. In it numerous peripheral antenna chromophores are assembled together at well-defined distance and orientation to guarantee that the excitation energy is funneled directionally and rapidly to the acceptor of the reaction centre.[1] More than 200 organized chromophores funnel energy into one acceptor, endowing the natural light-harvesting system with considerable capacity to utilize solar energy efficiently.[1a] Reported synthetic analogs of the light-harvesting system used various scaffolds including dendrimers,[2] porphyrin arrays,[3] organo or hydrogels,[4] biopolymer assemblies[5] and nanomaterials[6]. The development and studies of these analogs have been motivated by the fundamental importance of understanding natural photosynthesis and potential applications in photocatalysis, optoelectronic devices, and luminescent materials.[7] However, traditional chromophores are vulnerable to photoluminescence quenching in these concentrated states. Identifying strategies to maximize the density of chromophores while minimizing self-quenching remain a critical but highly challenging task in the construction of artificial light-harvesting system.[7a, 8]

A promising strategy relies on host–guest interactions to maintain short separations of chromophores while suppressing excitation energy quenching. Host–guest interaction using bulky host, such as cucurbit[n]uril (n= 7, or 8), or pillar[n]arene (n=5, or 6), has proved to be an effective approach to enhance fluorescence intensity by encapsulating guest group of chromophoric molecules while hindering chromophores from stacking.[9] For example, Zhang and co-workers took advantage of bulky CB[7] to suppress self-quenching of porphyrins and constructed supramolecular photosensitizers with enhanced antibacterial efficiency.[9b] However, to our knowledge only rare examples of light-harvesting systems utilizing bulky hosts have been reported to date.[2b, 10] Below, we report a novel light-harvesting system based on supramolecular polymeric nanoparticles comprised of chromophoric guest molecules (**GD** and **GA**) and disulfide-bridged bispillar[5]arene (**bisP5A**) (Scheme 1). These water-dispersible supramolecular polymeric



Scheme 1 Chemical structures of disulfide-bridged bispillar[5]arene (**bisP5A**), and the guest molecules (energy donor: **GD** and energy acceptor: **GA**); cartoon representations of **bisP5A**, **GD**, **GA**, their supramolecular polymers and the light-harvesting supramolecular polymeric nanoparticles (**LHSPNPs**); the schematic light-harvesting paths in the **LHSPNPs**.

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Electronic Supplementary Information (ESI) available: Experimental details including the synthesis and characterization of **GD** and **GA**, copies of NMR spectra, HR-ESI-MS, COSY, ROESY, DOSY, viscosity, SEM, DLS and supporting absorption and fluorescence measurements and calculations. See DOI: 10.1039/x0xx00000x

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nanoparticles (**SPNPs**) were prepared by the microemulsion method from supramolecular polymers formed by host–guest interaction of **GD**/**GA** and **bisP5A**. The formation of **SPNPs** maintains the chromophores in close proximity of each other, maximizing energy transfer and bulky pillar[5]arene minimizes the self-quenching of excitation energy, leading to the excellent light-harvesting ability.

We designed blue emissive diphenylanthracene derivative (**GD**) as energy donor and green emissive diphenylethynylanthracene derivative (**GA**) as energy acceptor (Scheme 1). **GD** and **GA** were synthesized in 5 steps with overall yield of 47% and 37%, respectively (Scheme S1 and Scheme S2 in ESI†). The large extinction coefficients (2.54×10^4 M^{-1} cm^{-1} for **GD** and 3.42×10^4 M^{-1} cm^{-1} for **GA**, Fig. S1a in ESI†), well-separated absorption maxima (378 nm for **GD** and 450 nm for **GA**, Fig. S1b in ESI†), and significant spectral overlap between the emission band of **GD** and the absorption band of **GA** (Fig. S2 in ESI†, $J = 9.99 \times 10^{14}$ M^{-1} cm^{-1} nm^4) all favour Förster resonance energy transfer.[7a] We modified both diphenylanthracene and diphenylethynylanthracene with a pair of cyanoalkyl triazoles to afford **GD** and **GA** respectively. The motif of cyanoalkyl triazole has strong affinity towards pillar[5]arene with the binding constant of 1.2×10^4 M^{-1} in $CHCl_3$, driving the formation of the supramolecular polymers of **GD**/**GA** and **bisP5A**.[11] Pillararenes are emerging macrocyclic host with facile synthesis, unique rigid structure, versatile functionalization, and interesting host–guest properties.[12] We here used disulfide-bridged bispillar[5]arene (**bisP5A**) because of its convenient synthesis.[13]

The host–guest interactions were demonstrated by ^1H NMR and 2D ROESY spectroscopy. ^1H NMR spectra of a mixture of **bisP5A** and **GD** suggested the formation of **GD⊂bisP5A**, as evidenced by upfield shifts of methylene protons ($\Delta\delta_1$= -3.47 ppm, $\Delta\delta_2$= -2.90 ppm, $\Delta\delta_3$= -3.19 ppm, $\Delta\delta_4$= -2.07 ppm, Fig. S3 in ESI†). The proton chemical shifts described here were assigned on the basis of the ^1H–^1H COSY spectrum (Fig. S4 in ESI†). The 2D ROESY spectrum showed clear correlations between methylene protons of **GD** and aromatic and methyl protons of **bisP5A**, further confirming this inclusion complex (Fig. S5 in ESI†). The formation of supramolecular polymers was probed by diffusion-ordered ^1H NMR spectroscopy (DOSY) and viscosity measurements. The diffusion coefficients decreased from 4.99×10^{-10} to 1.28×10^{-11} m^2 s^{-1} as concentration of **bisP5A** and **GD** increased from 5 mM to 150 mM; the slope of double logarithmic plots of specific viscosity vs. concentration increased from 1.09 to 1.99 as their concentration exceeded 12 mM (Fig. S6 in ESI†). Both DOSY and viscosity measurements support the formation of polymeric aggregates.

We prepared **SPNPs** from **bisP5A** and **GD** by the microemulsion method.[14] A 200 μL solution of **bisP5A** and **GD** (both at 20 mM) in $CHCl_3$ was injected into an aqueous solution of cetyltrimethyl ammonium bromide (CTAB) followed by ultrasonication for 25 min to afford nanospheres. Scanning electron microscopy (SEM) revealed nanoparticles of regular spherical shape and uniform size (Fig. 1a). Dynamic light scattering (DLS) yielded an average hydro-dynamic diameter of 73 nm (Fig. 1c). Interestingly, under identical experimental conditions **GD** alone formed nanosheets (**NSs**) as observed by SEM (Fig. 1b). We speculate that the formation of these nanosheets is driven by π–π stacking of diphenylanthracene aromatic rings and hydrophobic interactions while host–guest interactions are responsible for the supramolecular polymerization of **GD** and **bisP5A** and the formation of **SPNPs.**

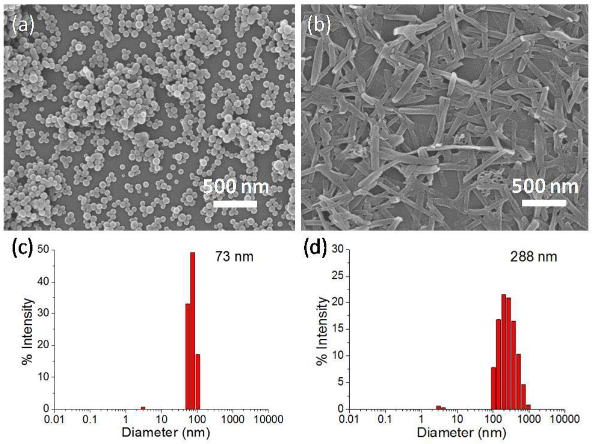

Fig. 1 SEM images and hydrodynamic diameter distributions from DLS of nanoparticles prepared from supramolecular polymers of **GD** and **bisP5A** (**SPNPs**, a, c) and nanosheets prepared from **GD** (**NSs**, b, d).

To further confirm the effect of pillar[5]arene and prove that incorporation of **GD** into the bulky host suppresses self-quenching, we compared the absorption and emission spectra of **GD** in solution, in **SPNPs** and in **NSs**. In the first two systems, individual **GD** molecules are isolated, whereas they are assumed to be aggregated in **NSs**. As shown in Fig. 2a, the absorption spectra of **SPNPs** and free **GD** in $CHCl_3$ solution are very similar, with the maximum absorption at 378 nm. In contrast, the absorption spectrum of **GD** in **NSs** is bathochromically shifted, with the maximum absorption at 410 nm, suggesting π–π stacking of the chromophores that is absent in **SPNPs**. A similar effect was observed in fluorescence spectra. Excitation of aqueous dispersions of **SPNPs** and **NSs** of identical optical density resulted in ~4-fold higher emission intensity from **SPNPs** compared to **NSs** (Fig. 2b). Their absolute fluorescence quantum yields also demonstrate the difference: 4.3% for **NSs**, vs. 17.9% for **SPNPs**. Previously, **SPNPs** of the

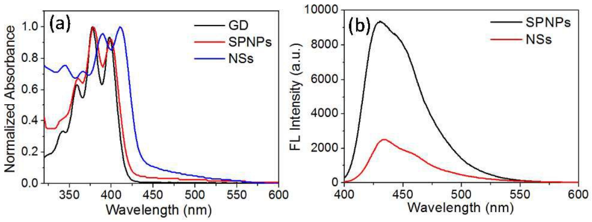

Fig. 2 (a) Normalized absorption spectra of solution of **GD** in $CHCl_3$ and aqueous dispersion of **SPNPs**, **NSs**; (b) Fluorescence spectra of aqueous dispersion of **SPNPs**, **NSs** (λ_{exc} = 378 nm).

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same fluorophore linked by quadruple hydrogen bond (Scheme S3 in ESI†) were reported to manifest fluorescence quantum yield of 11.9%.[15] We attribute the increased quantum yield in these pillar[5]arene-based **SPNPs** to the steric bulk of pillar[5]arene, whose outer diameter of 13.5 Å (according to the minimized energy model of 1,4-dimethoxypillar[5]arene (Fig. S7 in ESI†) and the X-ray crystal structure of 1,4-dipropoxypillar[5]arene).[16] In **SPNPs**, these bulks disrupt π–π interactions, suppressing self-quenching and enhancing emission.

Considering the advantage of **SPNPs** in preventing self-quenching of aromatic chromophores, we constructed light-harvesting supramolecular polymeric nanoparticles (**LHSPNPs**) by co-polymerization of **bisP5A**, **GD**, and **GA**. **GA** carried the sample cyanoalkyl triazole complexing moiety and manifested the same complexation with **bisP5A** as **GD** (Fig. S8 in ESI†). The solubility of **GA** in $CHCl_3$ was enhanced after the addition of **bisP5A**, suggesting complexation of **GA** and **bisP5A**. **BisP5A**, **GA**, and **GD** were first employed with ratio of 100: 2.0: 98.0 to form ternary supramolecular polymers, which then assembled into **LHSPNPs**. The participation of **GA** merely altered the assembly behaviour and the ternary copolymers formed spherical nanoparticles with a diameter similar to that of **GD** and **bisP5A** pairs (~82 nm vs. ~73 nm, Fig. S9d and Fig. S9d' in ESI†).

In **LHSPNPs** the fluorescence quenching of **GD** and amplification of **GA** emission suggested the energy transfer from donor **GD** to acceptor **GA**. When an aqueous dispersion **LHSPNPs** was irradiated at 378 nm the emission of ternary **LHSPNPs** at 430 nm decreased 69% compared to the emission from a dispersion of binary **SPNPs** of the same optical density. **LHSPNPs** also emitted at 494 nm, which corresponded to fluorescence of **GA** (the green line in Fig. 3a). The 378 nm excitation wavelength corresponds to the maximum extinction coefficients of **GD** and a negligible extinction coefficient of **GA**. In contrast, irradiating **LHSPNPs** at 450 nm, corresponding to the maximum absorption of **GA**, produced emission intensity at 494 nm 15-fold lower than that under 387 nm irradiation (Fig. 3b). This difference in emission intensities from the same fluorophore at different excitation wavelengths reflects two factors: (a) a higher fraction of incident irradiation at 378 nm is absorbed by **LHSPNPs** compared to 450 nm and (b) efficient transfer of excitation energy from **GD** to **GA**. The value of 15 was determined by $I_{494\ nm\ (\lambda\ exc\ =\ 378\ nm)}/I_{494\ nm\ (\lambda\ exc\ =\ 450\ nm)}$ and defined as antenna effect representing the degree of the amplified **GA** emission assisted by the excitation of **GD**. In addition, the excitation spectrum of **LHSPNPs** for 494 nm was nearly identical to the absorption spectrum of **GD** in **SPNPs**, proving that the excited energy of **GA** derives from absorption of **GD** (Fig. S10 in ESI†).

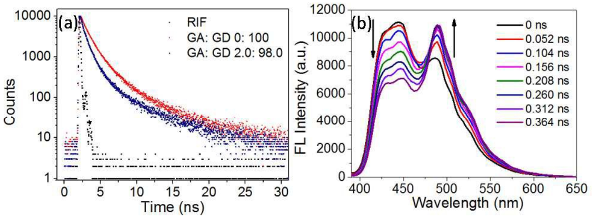

Fig. 4 (a) Fluorescence decay profiles of aqueous dispersion of **SPNPs** of **GD** and **bisP5A** and **LHSPNPs** (**GA**: **GD** = 2.0:98.0, λ_{exc} = 375 nm, $\lambda_{monitor}$ = 430 nm, RIF = instrument response function); (b) Time-resolved fluorescence spectra of aqueous dispersion of **LHSPNPs** (**GA**: **GD** = 1.0:99.0, λ_{exc} = 375 nm). The time when emission peak of **GD** reached the maximum value was set as 0 ns in (b).

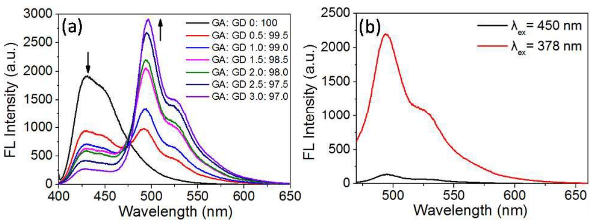

Fig. 3 (a) Fluorescence spectra of aqueous dispersion of **SPNPs** of **GD** and **bisP5A** and **LHSPNPs** with various **GA**/**GD** molar ratio (λ_{exc} = 378 nm); (b) Fluorescence spectra of aqueous dispersion of **LHSPNPs** excited by 378 nm and 450 nm (**GA**: **GD** = 2.0:98.0).

Additional evidence for the energy transfer from **GD** to **GA** was obtained from time-resolved fluorescence measurements. We measured the lifetimes of the excited state of **GD** in binary **SPNPs** (**GD** + **bisP5A**) and ternary **LHSPNPs** (**GD** + **GA** + **bisP5A**) of 1.3 ns and 0.8 ns, respectively (Fig. 4a). Such a prominent shortening of the donor lifetime confirmed energy transfer from **GD** to **GA** in the **LHSPNPs**. We also recorded time-resolved fluorescence spectra of **GA** and **GD** in **LHSPNPs** with a **GA/GD** ratio of 1.0:99.0 upon 375 nm excitation. We observed a rapid increase in the fluorescence intensity of **GD**, followed by the slower growth of **GA** emission (Fig. S12 in ESI†). Once the emission peak of **GD** reached the maximum value (set as 0 ns), it began to decay while the emissive intensity of **GA** kept increasing for 0.36 ns. (Fig. 4b). The spectral changes indicated the energy transfer from excited donor **GD** to acceptor **GA**. The obtained spectrum at 0.36 ns was similar with that of **LHSPNPs** in the steady-state fluorescence measurements (the blue line in Fig. 3a).

Increasing the **GA**/**GD** molar ratio from 0.5:99.5 to 3.0:97.0 increased the intensity of **GA** emission at 494 nm, while decreasing the emission of **GD** at 430 nm (Fig. 3a). The resulting energy transfer efficiency reaches the maximum of 85% at the **GA**/**GD** molar ratio of 3.0:97.0 (Table S2 and Table S3 in ESI†). In contrast, the antenna effect increased with the decrease of the **GA**/**GD** molar ratio (Fig. S13 and Table S2 in ESI†) as more **GD** molecules transferred the excitation energy to per **GA** chromophore reaching the maximum of 22 at the **GA**/**GD** molar ratio of 0.5:99.5.

In summary, we constructed novel light-harvesting supramolecular polymeric nanoparticles, which were formed by host–guest interactions between pillar[5]arene and anthracene-derived energy donor (**GA**) and acceptor (**GD**). Bulky pillar[5]arene inhibited self-quenching of these chromophores while maximizing energy transfer from **GD** to **GA** to afford highly emissive material. This strategy may inspire

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the exploration of new light-harvesting systems and luminescent materials.

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