Supporting Information

Enhanced emission in supramolecular artificial light-harvesting system for photocatalytic thiol-ene reaction

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Table of Contents

1. General information and experimental procedure	3
2. Synthesis of WPP5 and BND	4
3. Absorption spectra of WPP5, BND and ESY	5
4. Tyndall effects of aggregates	5
5. DLS results of supramolecular nanoparticles	6
6. Zeta potential results of supramolecular nanoparticles	6
7. Fluorescence lifetimes of supramolecular nanoparticles	7
8. Energy transfer efficiency calculation	.10
9. Antenna effect (AE) calculation	.11
10. Photocatalytic thiol-ene reaction	.12
11. References	.25

1. General information and experimental procedure

1) General information:

All reactions were performed in air atmosphere unless otherwise stated. The commercially available reagents and solvents were either employed as purchased or dried according to procedures described in the literatures. Column chromatography was performed with silica gel (200 - 300 mesh) produced by Qingdao Marine Chemical Factory, Qingdao (China). All yields were given as isolated yields. NMR spectra were recorded on a Bruker DPX 400 MHz spectrometer with internal standard tetramethylsilane (TMS) and solvent signals as internal references at room temperature, and the chemical shifts (δ) were expressed in ppm and J values were given in Hz. High-resolution electrospray ionization mass spectra (HR-ESI-MS) were recorded on an Agilent 6540Q-TOF LCMS equipped with an electrospray ionization (ESI) probe operating in the positive-ion mode with direct infusion. Scanning electron microscope (SEM) investigations were carried out on a Hitachi SU8060 instrument. Transmission electron microscope (TEM) investigations were carried out on a Talos F200X instrument. Dynamic light scattering (DLS) measurements were carried out on a Brookhaven BI-9000AT system (Brookhaven Instruments Corporation, USA), using a 200-mW polarized laser source ($\lambda = 514$ nm). Zeta-potential measurements were performed at 25 °C on a Zetasizer Nano Z apparatus (Malvern Instruments Ltd., UK) using the Smoluchowski model for the calculation of the Zetapotential from the measured electrophoretic mobility. The UV-Vis absorption spectra were measured on a Perkin Elmer Lambda 35 UV-Vis Spectrometer. The excitation and emission spectra were recorded on a Hitachi F-7000 Fluorescence Spectrometer. The fluorescence lifetimes were measured employing time correlated single photon counting on a FS5 instrument (Edinburg Instruments Ltd., Livingstone, UK). 9 W Philips White LED was used as the light sources in photocatalytic reactions. The quantum yields were investigated by three wavelengths of monochromatic light sources (9 W 460 nm Blue LED, 9 W 520 nm Green LED, and 9 W 595 nm Yellow LED from Shenzhen Boxing Technology Ltd., Shenzhen, China). The unit area photon flux was measured by AE330 photosynthetic radiation detector (AZOVTES, Guangzhou, China).

2) Experimental procedure:

Fabrication of supramolecular nanoparticle solution. Initially, the stock solution of BND

(0.02 M, dissolved in DMSO), WPP5 (4.2×10^{-4} M, dissolved in water), and ESY (2×10^{-4} M, dissolved in DMSO) were prepared, respectively. WPP5-BND nanoparticles were prepared as follows: 10 µL of BND solution and 48 µL of WPP5 solution were added into 3.94 mL of water. WPP5-BND-ESY nanoparticles were prepared as follows: 10 µL of BND solution, X ($0.1 \sim 5$) µL of ESY solution, and 48 µL of WPP5 solution were added into 3.94 - X mL of water.

2. Synthesis of WPP5 and BND

WPP5 and **BND** was synthesized and purified according to our reported procedures.^{S1–S3} ¹H NMR of **WPP5** (D₂O, 400 MHz) δ (ppm): 6.77 (s, 10H), 4.00 – 3.83 (m, 20H), 3.71 (s, 10H), 2.11 – 1.94 (m, 20H).



Fig. S1¹H NMR spectrum (400 MHz, D₂O, 298 K) of compound WPP5.

¹H NMR of **BND** (DMSO-*d*₆, 400 MHz) δ (ppm): 8.42 (s, 1H), 8.34 (s, 1H), 8.29 (s, 1H), 8.16 (d, *J* = 8.8 Hz, 1H), 8.09 – 8.06 (m, 2H), 8.03 – 7.92 (m, 4H), 7.63 – 7.58 (m, 2H), 7.41 (s, 1H), 7.26 (dd, *J* = 8.8, 2.4 Hz, 1H), 4.15 (t, *J* = 6.4 Hz, 2H), 3.28 – 3.24 (m, 2H), 3.03 (s, 9H), 1.84 – 1.77 (m, 2H), 1.71 – 1.63 (m, 2H), 1.51 – 1.44 (m, 2H), 1.39 – 1.25 (m, 10H).



Fig. S2 ¹H NMR spectrum (400 MHz, DMSO-*d*₆, 298 K) of compound BND.

3. Absorption spectra of WPP5, BND and ESY



Fig. S3 Absorbance spectra of the various species and associated systems.

4. Tyndall effects of aggregates



Fig. S4 Tyndall effects: (a) BND solution, (b) WPP5 solution, (c) WPP5-BND solution, and (d) WPP5-BND-ESY solution.



5. DLS results of supramolecular nanoparticles





Fig. S6 DLS result of WPP5-BND-ESY solution at 25 °C. [BND] = 5×10^{-5} M, [WPP5] = 5×10^{-6} M, and [ESY] = 2.5×10^{-7} M, respectively.

6. Zeta potential results of supramolecular nanoparticles



Fig. S7 Zeta potential result of WPP5-BND solution at 25 °C. [BND] = 5×10^{-5} M and [WPP5] = 5×10^{-6} M,

respectively.



Fig. S8 Zeta potential result of WPP5-BND-ESY solution at 25 °C. [BND] = 5×10^{-5} M, [WPP5] = 5×10^{-6} M,

and [ESY] = 2.5×10^{-7} M, respectively.

7. Fluorescence lifetimes of supramolecular nanoparticles



Fig. S9 The fluorescence lifetime acquisition of WPP5.

Decay S : Time Scan											Decay5
Counts	10 ¹										
	10 ⁰	10	20	30	40	50 Time/ns	60	70	80	90	
Current Measurement St Peak: 3/1000 Acq	otus 0% .Time: 02:33 mins										

Fig. S10 The fluorescence lifetime acquisition of ESY.



Fig. S11 The fluorescence lifetime acquisition of WPP5-ESY.



Fig. S12 The fluorescence lifetime acquisition of WPP5-BND.



Fig. S13 The fluorescence lifetime acquisition of WPP5-BND-ESY.



Fig. S14 The fluorescence lifetime acquisition of BND.



Fig. S15 The fluorescence lifetime acquisition of BND-ESY.

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Fig. S16 The fitted fluorescence lifetime of WPP5-BND solution at 25 °C. [BND] = 5×10^{-5} M and [WPP5] = 5



 \times 10⁻⁶ M, respectively.

Fig. S17 The fitted fluorescence lifetime of WPP5-BND-ESY solution at 25 °C. [BND] = 5×10^{-5} M, [WPP5] = 5×10^{-6} M, and [ESY] = 2.5×10^{-7} M, respectively.

Table S1. Fluorescence lifetimes of WPP5-BND and WPP5-BND-ESY nanoparticles monitored at 490 nm. $[WPP5] = 5 \times 10^{-6} \text{ M}, [BND] = 5 \times 10^{-5} \text{ M}, \text{ and } [ESY] = 2.5 \times 10^{-7} \text{ M}, \text{ respectively.}$

Sample	τ_1/ns	Rel/%	τ_2/ns	Rel/%	τ/ns	χ^2
WPP5-BND	0.72	67.44	3.64	32.56	1.67	1.156
WPP5-BND-ESY	0.52	80.80	2.85	19.20	0.97	1.005

8. Energy transfer efficiency calculation



Fig. S18 Fluorescence spectra of the various species and associated systems.



Fig. S19 Fluorescence spectra of WPP5-BND and WPP5-BND-ESY ([WPP5] = 5×10^{-6} M, [BND] = 5×10^{-5} M, and [ESY] = 2.5×10^{-7} M, respectively).

The energy-transfer efficiency (Φ_{ET}) was calculated using equation S1:

 $\Phi_{\rm ET} = 1 - I_{\rm DA} / I_{\rm D} (\rm eq. \ S1)$

Where I_{DA} and I_D are the fluorescence intensities of the emission of WPP5-BND-ESY nanoparticles (donor and acceptor) and WPP5-BND nanoparticles (donor), respectively.

The energy-transfer efficiency (Φ_{ET}) was calculated as 60.6% in water, measured under the condition of [**WPP5**] = 5 × 10⁻⁶ M, [**BND**] = 5 × 10⁻⁵ M, [**ESY**] = 2.5 × 10⁻⁷ M, and λ_{ex} = 365 nm.

9. Antenna effect (AE) calculation



Fig. S20 Fluorescence spectra of **WPP5-BND-ESY** in water (red line), blue line (acceptor emission, $\lambda_{ex} = 500$ nm) ([**WPP5**] = 5 × 10⁻⁶ M, [**BND**] = 5 × 10⁻⁵ M, and [**ESY**] = 2.5 × 10⁻⁷ M, respectively). The black line represents the fluorescence spectrum of **WPP5-BND**, which was normalized according to the fluorescence intensity at 490 nm of the red line.

The antenna effect (AE) was calculated based on the excitation spectra using equation S2.

 $\mathbf{AE} = (\mathbf{I}_{\text{DA},365} - \mathbf{I}_{\text{D},365}) / \mathbf{I}_{\text{DA},500} \text{ (eq. S2)}$

Where $I_{DA,365}$ and $I_{DA,500}$ are the fluorescence intensities at 551 nm with the excitation of the donor at 365 nm and the direct excitation of the acceptor at 500 nm, respectively. $I_{D,365}$ is the fluorescence intensity at 551 nm of the **WPP5-BND** assembly, which was normalized with the **WPP5-BND-ESY** assembly at 490 nm.

The antenna effect value was calculated as 29.3 in water, measured under the condition of $[WPP5] = 5 \times 10^{-6} \text{ M}, [BND] = 5 \times 10^{-5} \text{ M}, \text{ and } [ESY] = 2.5 \times 10^{-7} \text{ M}, \text{ respectively.}$

10. Photocatalytic thiol-ene reaction



Scheme S1. Photocatalytic thiol-ene reaction.

According to previous literatures,^{S4-S6} supramolecular nanoparticles were prepared according to the above experimental procedures, respectively. Thiophenol-R₁ (40 μ mol, 1 equiv.) and ethenylbenzene-R₂ (40 μ mol, 1 equiv.) were added in the freshly prepared LHS solution (4 mL) in a 10 mL glass vial. The mixture was cooled by liquid nitrogen and degassed with nitrogen for three times, and then irradiated by 9 W White LED at room temperature for corresponding time. After that, the mixture was extracted with 20 mL ethyl acetate and the combined organic layer was washed with saturated NaHCO₃ solution and brine. The solvent was removed to afford product 1-11. The crystal structure of product **8** was successfully obtained (Fig. S21 and S22).



Fig. S21 X-ray Structure of the photocatalytic product 8. Thermal ellipsoids were shown at 50% probability level.



Fig. S22 The crystal packing of product 8 viewed along a, b, and c axes.



Fig. S23 The plausible mechanism for the photocatalytic thiol-ene click reaction of thiophenol and styrene derivatives.



Fig. S24 Fluorescence spectra of ESY and ESY with 4-methoxythiophenol.

According to reported procedure,^{S7,S8} a classic radical-trapping experiment between 2,2,6,6-tetramethylpiperidinooxy (TEMPO) and 4-methoxythiophenol was carried out. 4-Methoxythiophenol (40 µmol, 1 equiv.) and 2,2,6,6-tetramethylpiperidinooxy (TEMPO) (80 µmol, 2 equiv.) were added in LHS for photocatalysis. The photocatalytic product was successfully obtained by column chromatography (PE:EA = 5:1). ¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.59 (d, J = 8.4 Hz, 2H), 6.97 (d, J = 8.4 Hz, 2H), 3.84 (s, 3H), 1.65 – 1.49 (m, 18H). HR-ESI-MS: m/z [M + H]⁺ calcd for [C₁₆H₂₆NO₂S]⁺ 296.1679, found 296.1672.



Fig. S25 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of the photocatalytic product of 4-methoxythiophenol and TEMPO.



Fig. S26 HR-ESI-MS spectrum of the photocatalytic product of 4-methoxythiophenol and TEMPO.

¹H NMR data of product 1-11

Product 1. (4-methoxyphenyl)(phenethyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.59 (d, J = 8.8 Hz, 2H), 7.31 (t, J = 7.2 Hz, 2H), 7.23 (d, J = 7.2 Hz, 1H), 7.18 (d, J = 6.8 Hz, 2H), 7.04 (d, J = 8.8 Hz, 2H), 3.86 (s, 3H), 3.08 – 3.01 (m, 3H), 2.94 – 2.86 (m, 1H).



Fig. S27 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 1.

Product 2. (4-methylphenethyl)(p-tolyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.28 (d, J = 8 Hz, 2H), 7.11 – 7.05 (m, 6H), 3.11 (t, J = 8 Hz, 2H), 2.87 (t, J = 8.4 Hz, 2H), 2.31 (s, 6H).



Fig. S28 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 2.

Product 3. 4-(2-((4-methoxyphenyl)thio)ethyl)benzonitrile

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.59 (t, J = 8.4 Hz, 4H), 7.30 (d, J = 8 Hz, 2H), 7.05 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H), 3.18 – 3.11 (m, 1H), 3.05 – 3.01 (m, 2H), 2.98 – 2.91 (m, 1H).



Fig. S29 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 3.

Product 4. (4-bromophenyl)(phenethyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.40 (d, J = 6.4 Hz, 2H), 7.31 (t, J = 6.8 Hz, 2H), 7.23 (d, J = 7.2 Hz, 1H), 7.19 – 7.16 (m, 4H), 3.15 (t, J = 7.6 Hz, 2H), 2.92 (t, J = 8.4 Hz, 2H).



Fig. S30 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 4.

Product 5. (2,4-dimethylphenyl)(4-fluorophenethyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.22 (d, *J* = 8 Hz, 1H), 7.13 (dd, *J* = 8.4, 5.6 Hz, 2H), 7.0 (s, 1H), 6.97 (t, *J* = 8.4 Hz, 3H), 3.06 (t, *J* = 7.2 Hz, 2H), 2.87 (t, *J* = 8.4 Hz, 2H), 2.34 (s, 3H), 2.28 (s, 3H).





Fig. S31 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 5.

Product 6. (4-bromophenyl)(2-(naphthalen-2-yl)ethyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.82 (d, *J* = 9.6 Hz, 3H), 7.62 (s, 1H), 7.49 – 7.44 (m, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4, 1.2 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 3.25 (t, *J* = 6.8 Hz, 2H), 3.10 (t, *J* = 8.4 Hz, 2H).



Fig. S32 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 6.

Product 7. phenethyl(p-tolyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.30 – 7.26 (m, 4H), 7.22 (d, J = 7.2 Hz, 1H), 7.18 (d, J = 7.2 Hz, 2H), 7.11 (d, J = 8 Hz, 2H), 3.13 (t, J = 7.6 Hz, 2H), 2.91 (t, J = 8.4 Hz, 2H), 2.31 (s, 3H).



Fig. S33 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 7.

Product 8. 4-(2-((4-bromophenyl)thio)ethyl)benzonitrile

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.60 (d, *J* = 8 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 3.17 (t, *J* = 7.2 Hz, 2H), 2.98 (t, *J* = 8 Hz, 2H).





Fig. S34 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 8.

Product 9. (2,4-dimethylphenyl)(2-(naphthalen-2-yl)ethyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.82 – 7.77 (m, 3H), 7.63 (s, 1H), 7.45 – 7.41 (m, 2H), 7.34 (dd, *J* = 8, 1.2 Hz, 1H), 7.28 (d, *J* = 8 Hz, 1H), 7.03 (s, 1H), 7.01 (d, *J* = 8 Hz, 1H), 3.20 – 3.16 (m, 2H), 3.09 – 3.05 (m, 2H), 2.37 (s, 3H), 2.30 (s, 3H).



Fig. S35 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 9.

Product 10. (2,4-dimethylphenyl)(4-methylphenethyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.23 (d, *J* = 7.6 Hz, 1H), 7.10 – 7.05 (m, 4H), 7.00 (s, 1H), 6.97 (d, *J* = 7.6 Hz, 1H), 3.08 (t, *J* = 7.6 Hz, 2H), 2.87 (t, *J* = 8.4 Hz, 2H), 2.35 (s, 3H), 2.30 (s, 3H), 2.28 (s, 3H).



Fig. S36 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 10.

Product 11. (4-methoxyphenyl)(2-(naphthalen-2-yl)ethyl)sulfane

¹H NMR (CDCl₃, 400 MHz) δ (ppm): 7.81 – 7.75 (m, 3H), 7.62 (t, J = 8.8 Hz, 3H), 7.47 – 7.42 (m, 2H), 7.30 (dd, J = 8.4, 1.2 Hz, 1H), 7.04 (d, J = 8.8 Hz, 2H), 3.85 (s, 3H), 3.25 – 3.19 (m, 1H), 3.14 (t, J = 6.8 Hz, 2H), 3.09 – 3.02 (m, 1H).





Fig. S37 ¹H NMR spectrum (400 MHz, CDCl₃, 298 K) of product 11.

Time/h	Yield/%
8	
8	3 ^a , 4 ^b , 4 ^c , 15 ^d
8	3ª, 4 ^b , 4 ^c , 18 ^d
8	3 ^a , 3 ^b , 3 ^c , 16 ^d
8	$5^{a}, 6^{b}, 6^{c}, 23^{d}$
8	4ª, 5 ^b , 4 ^c , 19 ^d
8	6 ^a , 6 ^b , 8 ^c , 27 ^d
8	5ª, 4 ^b , 5 ^c , 20 ^d
8	6 ^a , 5 ^b , 7 ^c , 28 ^d
8	4ª, 5 ^b , 4 ^c , 17 ^d
8	3 ^a , 4 ^b , 5 ^c , 15 ^d
8	3ª, 4 ^b , 4 ^c , 15 ^d
	8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8

Table S2. The photocatalytic results of WPP5, BND, WPP5-BND, and ESY groups, respectively.

^aWPP5 group's photocatalytic yield, ^bBND group's photocatalytic yield, ^cWPP5-BND group's photocatalytic

yield, and ${}^{d}ESY$ group's photocatalytic yield.

Sample	Time/h	Yield/%
Product 1	8	3
Product 2	8	4
Product 3	8	3
Product 4	8	4
Product 5	8	3
Product 6	8	4
Product 7	8	4
Product 8	8	5
Product 9	8	3
Product 10	8	3
Product 11	8	3

Table S3. The photocatalytic results of blank experiment (only reactant substrate).

Based on previous literatures,^{\$9,\$10} three wavelengths of monochromatic light sources (9 W 460 nm Blue LED, 9 W 520 nm Green LED, and 9 W 595 nm Yellow LED from Shenzhen Boxing Technology Ltd., Shenzhen, China) were utilized to investigate the quantum yields. 4-Methoxythiophenol (0.2 mmol, 1 equiv.) and ethenylbenzene (0.2 mmol, 1 equiv.) were added in the freshly prepared LHS solution (20 mL) in a 50 mL round-bottom flask. The reaction mixture was stirred and irradiated by three wavelengths of monochromatic light sources (460 nm, 520 nm, and 595 nm) for 2 h. After irradiation, the solution was measured the unit area photon flux (AE330 photosynthetic radiation detector, AZOVTES). And the yield of product formed was isolated. The quantum yield is calculated using the following equation S3:

 $\Phi = \text{mol product} / (\text{flux} \cdot \text{S} \cdot \text{t}) (\text{eq. S3})$

Where, Φ is quantum yield, S (m²) is the irradiation area, and t (s) is the photoreaction time.

As shown in **Table S4**, the unit photon flux was 752 μ mol·s⁻¹·m⁻² (for 460 nm LED), 980 μ mol·s⁻¹·m⁻² (for 520 nm LED), 242 μ mol·s⁻¹·m⁻² (for 595 nm LED) (average of three experiments), the irradiation area was 1.14×10^{-3} m², and the product yields were 4%, 5%, 0% after 2.0 h (7200 s). Thus the quantum yields were 0.0013, 0.0012 and 0, confirming the reaction mechanism.

Table S4. The photocatalytic results of three wavelengths of monochromatic light sources.

Sample	Unit photon flux/µmol·s ⁻¹ ·m ⁻²	Yield/%	Quantum yield
460 nm LED	752	4	0.0013
520 nm LED	980	5	0.0012
595 nm LED	242	0	0



Fig. S38 Three wavelengths of monochromatic light sources and photosynthetic radiation detector.

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