# **Electronic Supplementary Information (ESI)**

# Supramolecular Single-Site Photocatalyst Based on Multi-to-One Förster Resonance Energy Transfer

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### 1. Chemicals and materials

Benzonitrile (SafeDry), potassium platinochloride (K<sub>2</sub>PtCl<sub>4</sub>) and 2-(dimethylamino)ethyl methacrylate (DMAEMA), potassium peroxodisulfate and phosphorus pentoxide were purchased from Shanghai Adamas-beta Co. Ltd. 5,10,15,20-Tetrakis(4-hydroxyphenyl) porphyrin (THPP) was purchased from Frontier Scientific. Graphite powder (thickness <6  $\mu$ m) was purchased from Shanghai Lingfeng Chemical Reagent Co. LTD. N,N-dimethylformamide (DMF), tetrahydrofuran (THF), methanol (MeOH), ethyl acetate (EA), n-hexane and hydrogen peroxide were purchased from Greagent. Potassium hydride (KH), calcium hydride (CaH<sub>2</sub>), sulfuric acid (>96.0%) and hydrochloric acid (36.0-38.0%) were purchased from Sinopharm Chemical Reagent Co., Ltd. THF was first purified with CaH<sub>2</sub> and further distilled with sodium wire to remove water. DMAEMA was distilled under reduced pressure to remove polymerization inhibitor before use. KH in mineral oil was washed by distilled THF for 6 times. The deionized water was obtained from the Milli-Q System. All the other materials were used as received without further purification.

# 2. Characterization and measurements

### Nuclear magnetic resonance (NMR) spectroscopy

<sup>1</sup>H NMR spectra were recorded using Bruker AVANCEIII 400 spectrometer with dimethylsulfoxide- $d_6$  as solvents at 298 K. Tetramethylsilane (TMS) was used as the internal standard.

#### Dynamic light scattering (DLS) and Zeta potentials measurements

DLS and Zeta potential measurements were performed on a Malvern Zetasizer Nano ZS90 (Malvern Instruments, Ltd.) equipped with a 4 mW He-Ne laser light. The scattering angle of DLS measurement was 90°.

# Matrix-assisted laser desorption and ionization time-of-flight mass spectrometry (MALDI-TOF-MS)

MALDI-TOF-MS was performed on a SolariX XR 7.0 T hybrid quadrupole-FTICR mass spectrometer equipped with an ESI/APCI/MALDI ion source (Bruker Daltonics, Bremen, Germany). The MS instrument was tuned and calibrated with ESI-L low concentration tuning mix (Aglient Technologies, Santa Clara, CA, USA) and sodium formate. The analytical sample was prepared by mixing the reaction mixture (2.0 mg mL<sup>-1</sup> in MeOH) with the matrix solution (10 mg mL<sup>-1</sup> DCTB in MeOH) in a v/v ratio of 1/5, and then loaded onto the MALDI plate. The sample on the plate was thoroughly dried prior to analysis. The mass spectra were analyzed using Compass Data Analysis 5.0 (Bruker).

## Transmission electron microscopy (TEM)

TEM was carried out on a FEI G2 Spirit BioTwin TEM with an accelerating voltage of 120 kV. Every TEM sample was prepared by depositing one drop of the sample solution onto a carbon-coated copper grid, and the grid was dried in the air at room temperature for 24 h.

#### High resolution transmission electron microscopy (HRTEM)

HRTEM measurements were performed with a JEOL JEM-2100F instrument at a voltage of 200 kV. The samples were prepared using the same method as the TEM measurements.

### Ultraviolet-visible (UV-vis) absorption spectroscopy

The UV-vis absorption spectra were recorded at 298 K in the range of 300-800 nm on a Shimadzu UV 3600 spectrometer. The solutions of the samples were added into a 1 cm quartz cuvette for the measurements.

### Fluorescence and time-resolved fluorescence spectroscopy

The fluorescence spectra and time-resolved fluorescence spectra were recorded on a FLS1000 steadystated & time-resolved fluorescence spectrofluorometer. The solutions of the samples were added into a 1 cm quartz cuvette for the measurements.

# X-ray photoelectron spectroscopy (XPS)

XPS was performed on a Kratos Axis UltraDLD with a monochromated Al K $\alpha$  X-ray bean as the excitation source (1486.6 eV). Binding energies were corrected by referring to the C 1s peak at 284.8 eV. GQDs aqueous were dropped on the aluminum foil and then dried in room temperature.

# Fourier transform infrared spectroscopy (FTIR)

FTIR spectrum was recorded on a Perkin Elmer Spectrum 100 FTIR Spectrometer at 298 K. The samples were coated onto KBr crystal wafers and carefully dried before measurements.

#### Photocatalytic hydrogen production

The activity of water splitting was evaluated in a 160 mL optical reaction vessel. A 300 W Xe-lamp equipped with  $\lambda \ge 420$  nm cutoff filter was used to gain visible-light illumination at ambient temperature. Typically, 2.0 mg of the photocatalyst and 50 mL deionized water containing 0.1 M ascorbic acid (AA) as sacrificial reagents were added to the reaction vessel. The pH value of the reaction system was adjusted by 0.5 M HCl/NaOH aqueous solutions. The reactions were conducted by a photocatalytic H<sub>2</sub> evolution system furnished by PerfectLight, Beijing Co., Ltd. The testing temperature was kept at 5 °C utilizing a homeothermic cooling circulation pump. A gas chromatograph (Shimadzu GC-2014C, argon as a carrier gas) containing a thermal-conductivity detector (TCD) was used to detect online the formation of H<sub>2</sub> every hour.

# 3. Synthetic procedures

#### Synthesis of graphene oxide (GO)

GO was synthesized according to the modified Hummers method.<sup>1</sup> Typically, 8 g graphite powder,  $K_2S_2O_8$  (10 g, 37.0 mmol) and  $P_2O_5$  (10 g, 70.5 mmol) were added to 30 mL concentrated  $H_2SO_4$  at 80 °C for 12 h. After cooling to the room temperature, the mixture was diluted with deionized water, then filtered and washed with deionized water several times until the filtrate became neutral. The product was dried at ambient condition over a day to obtain preoxidized graphite. The preoxidized graphite was added to a solution of concentrated  $H_2SO_4$  (184 mL) and NaNO<sub>3</sub> (4 g, 47.1 mmol) at 0 °C. Then, KMnO<sub>4</sub> (24 g, 151.9 mmol) was slowly added under vigorous stir, keeping the temperature of the mixture below 10 °C. The mixture was stirred for another hour. Afterwards, the mixture was reacted at 35 °C for 6 h under vigorous stir and stayed overnight. 368 mL deionized water and 20 mL 30%  $H_2O_2$  were added to the mixture to terminate the reaction. The color of the mixture turned to bright yellow. Metal ions were removed by filtering and washing the mixture with 1000 mL 1:10 HCl solution followed by large amounts of deionized water. In order to thoroughly remove metal ions and acids, the product was obtained by freeze-drying in vacuum. Yield = 62.5%.

#### Synthesis of graphene quantum dots (GQDs)

GQDs were synthesized according to a top-down approach reported previously.<sup>1</sup> 250 mg GO was dispersed in 10 mL DMF and ultrasonicated for 30 minutes (120 W, 100 kHz). Then, the mixed solutions were transferred to a 30 mL poly(tetrafluoroethylene) (Teflon)-lined autoclave and heated at 200 °C for 8 hours. After cooling to the room temperature naturally, the product comprising a transparent brown suspension and black precipitates was obtained. The suspension was further purified on silica gel by gradient column chromatography (mobile phase: A and B were CH<sub>2</sub>Cl<sub>2</sub>: MeOH = 2:1 (v/v) and H<sub>2</sub>O, respectively). The B phase elution gave the desired GQDs solution. The concentration of the GQDs solutions could be adjusted by adding deionized water or distilled under reduced pressure. Yield = 15.0%.

# Synthesis of 5,10,15,20-Tetrakis (4-hydroxyphenyl) platinum porphyrin (PtTHPP)

PtTHPP was synthesized according to literature.<sup>2</sup> K<sub>2</sub>PtCl<sub>4</sub> (498.1 mg, 1.2 mmol) and 5,10,15,20-Tetrakis(4-hydroxyphenyl) porphyrin (200.0 mg, 0.3 mmol) were dissolved in 60 mL of anhydrous benzonitrile and degassed with a stream of nitrogen for 30 minutes. The solution was vigorously stirred and refluxed under nitrogen atmosphere at 180 °C for 48 h. After distilling off the solvent, the residue was purified by column chromatography using silica gel with ethyl acetate: n-hexane (3:2) as the eluent. Dark red solid was obtained after removing the solvent under vacuum and dried. Yield = 50.7%. <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>, δ): 10.01 (s, 1H), 8.79 (s, 2H), 7.95 (d, *J* = 8.3 Hz, 2H), 7.19 (d, *J* = 8.3 Hz, 2H). MALDI-TOF MS Calcd for C<sub>44</sub>H<sub>28</sub>N<sub>4</sub>O<sub>4</sub>Pt 871.1718, found 871.1718.

#### Synthesis of single-Pt-site porphyrin star polymer (PtTHPD)

PtTHPD was synthesized according to the procedures reported in literature.<sup>2c,3</sup> PtTHPP (50.0 mg,  $5.7 \times 10^{-2}$  mmol) and 18-crown-6 ether (66.3 mg,  $2.5 \times 10^{-1}$  mmol) were dissolved in 20 mL of anhydrous THF in the glove box. KH (10.0 mg,  $2.5 \times 10^{-1}$  mmol) was added under vigorous stir at 50 °C for 1 h and subsequently the temperature was increased to 65 °C. DMAEMA (716.8 mg, 4.6 mmol) in 5 mL of anhydrous THF was dropwise added to the mixture within 24 hours. Deionized water was used to terminate the reaction. Afterwards, the mixed solution was precipitated in n-hexane for 3 times. The sediment was dialyzed against (MWCO: 3500 Da) DMF for 3 days and further dialyzed against (MWCO: 3500 Da) deionized water for 1 day. Finally, PtTHPD polymers were obtained by freezedrying in vacuum. Yield = 42.5%.

## Preparation of PtTHPD unimolecular micelles (PtTHPD-UMs)

PtTHPD (2.0 mg) was dissolved in 2.5 mL DMF and then 10 mL deionized water (pH=6.0) was dropwise added to the solution under vigorous stir within 12 h. The mixture was dialyzed against (MWCO: 1000 Da) deionized water (pH=6.0) for 3 days. 0.5 M HCl/NaOH aqueous solutions were used to adjust the pH value. The final concentration of the PtTHPD solution was calculated based on the weight of polymer and the volume of the solution after dialysis. Solutions of PtTHPD-UMs with different concentration were prepared by diluting with deionized water.

# Construction of the GQDs-PtTHPD photocatalytic system

GQDs-PtTHPD photocatalytic systems with different GQD/PtTHPD ratios were constructed by electrostatic self-assembly. We prepared six GQD/PtTHPD-UM aqueous solutions in the work. For these solutions, PtTHPD-UM aqueous solutions (pH=6.0) with a same volume but different concentrations were dropwise added into 0.3 mL GQD solutions (pH=6.0) with the same concentration of  $2.8 \times 10^{-4}$  M, respectively, under vigorous stirring. The final concentrations of GQDs in all of the mixed solutions were  $1.4 \times 10^{-5}$  M, while the concentrations of PtTHPD-UMs ranged from  $1.4 \times 10^{-7} \sim 1.4 \times 10^{-6}$  M (GQDs /PtTHPD-UM molar ratios ranging from 100:1 to 10:1). Afterwards, the mixed solutions were incubated for 30 minutes to produce stable GQDs-PtTHPD nanoparticles. These GQDs-PtTHPD nanoparticle solutions were subsequently used to photocatalysis and stationary state fluorescence measurements.

# 4. Supporting figures S1-S22



Fig. S1 Schematic illustration of the synthesis of GQDs.<sup>1</sup>



**Fig. S2** The 3D molecular simulation of single-layered GQDs (328 C atoms) based on the statistical average diameter  $(3.0 \pm 0.2 \text{ nm})$  measured through the TEM image. According to literature,<sup>1</sup> GQDs yielded by this method are single-layered or double-layered with an average thickness of 1.0 nm. based on this dimension, the molecular weight of GQDs is estimated to be 3.9 kD.



Fig. S3 A typical UV-vis absorption spectrum of GQDs.



Fig. S4 The up-conversion photoluminescence spectra of GQDs.



Fig. S5 (a) The XPS survey spectrum of GQDs. (b) The C 1s XPS survey spectrum of GQDs.



Fig. S6 The FTIR spectrum of GQDs.



Fig. S7 The Zeta potential result of GQDs at pH=6.0.



Fig. S8 Schematic illustration of the synthesis of PtTHPP.<sup>2</sup>



**Fig. S9** The <sup>1</sup>H NMR spectra of THPP and PtTHPP. The peak at -2.88 ppm, which is ascribed to the proton in the -NH of porphyrin core in THPP, disappears in the <sup>1</sup>H NMR spectrum of PtTHPP. This result indicates the successful Pt-N coordination.



**Fig. S10** The UV-vis absorption spectra of THPP and PtTHPP. The UV-vis absorption spectrum of THPP exhibits a strong B band and four weak Q bands. However, the number of Q bands decreases from four to two after the Pt coordination with porphyrin, indicating the successful synthesis of PtTHPP.



**Fig. S11** The MALDI-TOF-MS spectra of PtTHPP. The molecular weight of PtTHPP found by MALDI-TOF-MS is 871.1718, which agrees well with the calculated value 871.1719.



**Fig. S12** (a) The N 1s XPS survey spectrum of THPP. (b) The N 1s XPS survey spectrum of PtTHPP. The N 1s peaks of THPP can be fitted into two peaks at 400.1 eV and 397.9 eV, attributed to =N- and -NH groups, respectively. While PtTHPP shows only a single peak at 399.8 eV, which is caused by Pt-N coordination.



Fig. S13 Schematic illustration of the synthesis of the star polymer (PtTHPD).<sup>2c,3</sup>



**Fig. S14** <sup>1</sup>H NMR spectrum of PtTHPD. The proton peak at 10.01 ppm attributed to phenolic hydroxyl groups of PtTHPP core disappears while new peaks ascribed to PDMAEMA arms appear after oxyanionic polymerization, confirming the successful synthesis of PtTHPD. The average degree of polymerization of single PDMAEMA arm is calculated to be 20 via the equation  $DP_{arm}=S_a/S_A$ , where  $S_a$  and  $S_A$  represent the integrated area of peak a and A, respectively. The molecular weight of PtTHPD is thus determined to be about 14 kD.



**Fig. S15** (a) XPS survey spectra of THPP, PtTHPP and PtTHPD. Pt characteristic peaks are clearly observed in the XPS survey pectra of PtTHPP and PtTHPD. (b) O 1s XPS survey spectra of PtTHPP and PtTHPD. The peaks at 533.1 eV are ascribed to the O 1s of C-O groups of PtTHPP and PtTHPD, respectively. A new peak at 531.9 eV attributed to the O 1s of C=O groups appears after grafting PDMAEMA chains, which further confirms the successful synthesis of PtTHPD.



**Fig. S16** The Pt 4f XPS survey spectrum of THPD. Two peaks at 72.7 eV and 76.0 eV are attributed to Pt  $4f_{7/2}$  and Pt  $4f_{5/2}$ , respectively, which are between those of Pt(II) and Pt(0), indicating that the Pt species are partially in an oxidized form.



**Fig. S17** The DLS result of PtTHPD-UMs in the aqueous solution with pH=6.0 and a concentration of 0.05 mg mL<sup>-1</sup>.



Fig. S18 The Zeta potential result of PtTHPD-UMs in the aqueous solution with pH=6.0 and a concentration of 0.05 mg mL<sup>-1</sup>.



**Fig. S19** (a) Proposed models of GQDs-PtTHPD, where yellow circles represent GQDs and the blue circle denotes the PtTHPD-UM.  $d_1$  and  $d_2$  denote the diameters of GQDs and PtTHPD-UM, respectively. Thus, the diameter of GQDs-PtTHPD equals  $d=d_1+d_2+d_1=\sim 11$  nm. (b) The DLS result of GQDs-PtTHPD in the aqueous solution of pH=6.0.



**Fig. S20** Normalized PL spectrum ( $\lambda_{ex}$ =420 nm) and UV-vis absorption spectrum of GQDs and PtTHPD-UMs in aqueous solutions.



Fig. S21 (a) PL spectra ( $\lambda_{ex}$ =420 nm) of GQDs-PtTHPD NPs and pristine PtTHPD-UMs with the same concentration of PtTHPD-UMs from  $1.4 \times 10^{-7}$  M to  $1.4 \times 10^{-6}$  M. (b) The magnified PL spectra ( $\lambda_{ex}$ =420 nm) of pristine PtTHPD-UMs with increased concentration from  $1.4 \times 10^{-7}$  M to  $1.4 \times 10^{-6}$  M.



**Fig. S22** UV-vis absorption spectra of GQDs-PtTHPD (10:1) and pristine PtTHPD-UMs with the same concentration of PtTHPD-UMs.

#### **5** Calculation methods

#### Calculation of the fluorescence quantum yield of GQDs in water

The fluorescence quantum yield of GQDs in the aqueous solution of pH=6.0 is calculated by utilizing quinine sulfate in 0.05 M H<sub>2</sub>SO<sub>4</sub> as a standard based on the Equation S1.<sup>4</sup>

$$\boldsymbol{\Phi}_{\mathrm{x}} = \boldsymbol{\Phi}_{\mathrm{st}} \left( \frac{I_{\mathrm{x}}}{I_{\mathrm{st}}} \right) \left( \frac{\eta_{\mathrm{x}}^2}{\eta_{\mathrm{st}}^2} \right) \left( \frac{A_{\mathrm{st}}}{A_{\mathrm{x}}} \right)$$
(S1)

where  $\Phi$  is the quantum yield, *I* is the integrate area of the fluorescence emission peak, *A* is the absorption value determined by UV-vis spectroscopy and  $\eta$  is the refractive index of the solvent. The subscript '*st*' refers to the standard sample and '*x*' refers to the sample. Quinine sulfate is excited at 343 nm. The quantum yield of GQDs is calculated to be 13.3%.

Sample	Ι	A	η	$\Phi$
Quinine sulfate	7594157	0.029	1.33	0.550 (known)
GQDs	1180050	0.045	1.33	0.133

Table S1 Quantum yield of GQDs using quinine sulfate in 0.05 M H<sub>2</sub>SO<sub>4</sub> solution as a standard

# Calculation of the integral of the spectral overlapped area (J)

The parameter of the spectral overlap integral J is calculated by the Equation S2.<sup>3</sup>

$$J = \int_{0}^{\infty} f_{\rm D}(\lambda) \varepsilon_{\rm A}(\lambda) \lambda^4 \mathrm{d}\lambda$$
(S2)

where  $\lambda$  denotes the wavelength (nm);  $\varepsilon_A(\lambda)$  denotes the molar extinction coefficient of PtTHPD-UMs at the wavelength  $\lambda$ ;  $f_D(\lambda)$  denotes the fraction of the fluorescence intensity of GQDs. In this work, the overlap integral *J* is calculated to be 2.1×10<sup>12</sup> M<sup>-1</sup> cm<sup>-1</sup> nm<sup>4</sup> for light energy transfer from GQDs to the PtTHPD-UM.

#### Calculation of the energy-transfer efficiency ( $\Phi_{ET}$ ) of GQDs-PtTHPD

Energy-transfer efficiency ( $\Phi_{ET}$ ) is defined as the fraction of the absorbed energy transferred from donors to acceptors. It is experimentally measured as the ratio of the fluorescence intensities of donors in the absence and presence of acceptors (Equation S3).<sup>5</sup>

$$\Phi_{ET} = 1 - \frac{I_{DA}}{I_D} \tag{S3}$$

where  $I_{DA}$  and  $I_{D}$  are the fluorescence emission intensity of GQD antennas with and without the PtTHPD-UM catalytic center, respectively. The excitation wavelength is 420 nm.

Molar ratio of GQDs/PtTHPD	Energy transfer efficiency
100:1	6.6%
50:1	12.3%
25:1	17.7%
16:1	30.3%
12:1	43.1%
10:1	54.5%

**Table S2** Energy transfer efficiency of GQDs-PtTHPD light harvesting nanosystem under differentGQDs/PtTHPD mole ratios

# Calculation of the energy transfer rate constant $(k_{ET})$ of GQDs-PtTHPD

The energy transfer rate constant ( $k_{\rm ET}$ ) is calculated according to the Equation S4.<sup>3</sup>

$$\Phi_{\rm ET} = \frac{k_{\rm ET}}{k_{\rm ET} + \tau_{\rm D}^{-1}}$$
(S4)

where  $\Phi_{\text{ET}}$  is the energy-transfer efficiency of GQDs-PtTHPD,  $k_{\text{ET}}$  is the energy transfer rate constant and  $\tau_{\text{D}}$  is the singlet state fluorescence lifetime of GQDs-PtTHPD. The  $k_{\text{ET}}$  between GQDs and the PtTHPD-UM is calculated to be  $1.23 \times 10^8 \text{ s}^{-1}$ .

#### Calculation of the center to center distance $(R_{D-A})$ between the donor and acceptor

The center to center distance ( $R_{D-A}$ ) between GQD antennas and the PtTHPD-UM catalytic center is obtained according to the Equation S5.<sup>3</sup>

$$k_{\rm ET} = \frac{9000I\eta 10k^2 \Psi J}{128\pi^5 \eta^4 N \tau_{\rm D} R^6}$$
(S5)

where  $k_{\text{ET}}$  is the energy transfer rate constant, k is the orientation parameter ( $k^2 = 2/3$  for random orientation),  $\Psi$  is the fluorescence quantum yield of GQDs, J is the spectral overlap integral,  $\eta$  is the refractive index of water ( $\eta = 1.33$ ), N is the Avogadro constant,  $\tau_D$  is the fluorescence singlet lifetime of GQDs and  $R_{D-A}$  is the center to center distance between GQDs and the PtTHPD-UM. The center to center distance in GQDs-PtTHPD is estimated to be 4.0 nm.

#### Calculation of the average number of donors quenched by single acceptor $(K_{SV})$

The number of donors quenched by single acceptor ( $K_{SV}$ ) is calculated by the Stern-Volmer equation (Equation S6).<sup>6</sup>

$$\frac{F_0}{F} = I + K_{\rm SV}[A] \tag{S6}$$

Where  $F_0$  and F are the emission intensity of GQD antennas with and without the PtTHPD-UM catalytic center, respectively ( $\lambda$ =420 nm),  $K_{SV}$  is the Stern-Volmer constant, [A] is the concentration of the PtTHPD-UMs. When [A] denotes the molar ratio of GQDs/PtTHPD,  $K_{SV}$  denotes the number of GQDs antennas quenched by a single PtTHPD-UM catalytic center. In this work, the number of GQDs quenched by a single PtTHPD-UM ( $K_{SV}$ ) is estimated to be 7, which further evidences that solar energy is transferred from multiple GQD antennas to the single PtTHPD-UM catalytic center in GQDs-PtTHPD.

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