Electronic Supplementary Material (ESI) for Nanoscale Horizons.

Controlled Functionalization of Carbon Nanodots for Targeted Intracellular Production of Reactive Oxygen Species

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Electronic Supplementary Information

Content list：

S1 Experimental procedures

1. Materials

All chemicals used in the experiments were obtained from commercial sources as analytical reagents without further purification. 2,2'-(ethylenedioxy)bis(ethylamine) was purchased from Sigma-Aldrich. 3-(4-bromophenyl) propanoic acid, 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC·HCl), and *N*-hydroxysuccinimide (NHS) were purchased from Alfa Aesar. 3-thiophene boronic acid and folic acid were purchased from Acros. LC/MS analyses were performed on ThermoFisher Finnigan 6 LCQ Advantage Max. ^{1}H NMR spectra were recorded on Bruker DPX 300 instrument. The peak values were obtained as ppm (δ) and referenced to the solvent. The resonance multiplicity is indicated as s (singlet), d (doublet), t (triplet), dd (doublet of doublet), and m (multiplet). Centrifugation and bath sonication were performed on an Eppendorf centrifuge 5804R and an Elmasonic P sonicator. The dialysis was performed using membranes MWCO 1 kDa from Spectrum Laboratories Inc. Millipore water with a resistivity of 18.2 MΩ was used in the experiments. Inductively coupled plasma atomic emission spectroscopy (ICP–AES) analyses were performed on a Thermo Flash 2000, Thermo Scientific.

2. Instruments for the characterization of materials

HPLC was performed using a Nucleosil 100-5 Waters C18 reverse-phase HPLC column and a Waters Alliance e2695 separation module. The column was used with a 1.2 $mL \cdot min^{-1}$ flow rate of a gradient from 0 to 100% of B (A = H_2O/0.1% trifluoroacetic acid (TFA); B = CH_3CN/0.08% TFA) for 20 min. Fluorescence steady-state spectra were recorded via a Fluorolog FL3-22 (Horiba Jobin Yvon) spectrometer using a swig xenon 450 W lamp. UV-Vis spectra were recorded using a VARIAN 5000 spectrometer. The morphology of the samples was studied by transmission electron microscopy (TEM) (JEM-2010F, JEOL, Japan). Thermogravimetric analyses were performed by using a TGA Q500 TA instrument

with a ramp of 10 °C·min^{-1} under N_2 and a flow rate of 50 mL·min^{-1}. Photodynamic tests were executed with a 660 nm laser (Changchun new industries optoelectronics tech.co., ltd). The dynamic light scattering (DLS) measurements were done with a Zetasizer Nano ZS (Malvern, U.K.). AFM was carried out in tapping mode using a Bruker Multimode V AFM, equipped with nanoscope 5 controller. XPS was performed using Thermo Scientific KAlpha X-ray spectrometer with a mono X-Ray source Al Kα excitation (1486 eV). Binding energy calibration was based on C1s at 284.7 eV. The absolute photoluminescence quantum yield (PLQY) was measured by a Hamamatsu Quantaurus-QY integrating sphere in air-equilibrated condition using an empty quartz tube as a reference.

3. Synthesis of red-emissive carbon nanodots (RCNDs)

Benzyl 3-(4-bromophenyl) propanoate (1): 3-(4-bromophenyl) propanoic acid (1 g, 4.37 mmol) and K_2CO_3 (1.21 g, 8.73 mmol) were added to anhydrous DMF (10 mL) in an ice bath. After 3 h, (bromomethyl)benzene (0.75 g, 8.73 mmol) was added to the mixture. The reaction mixture was stirred at room temperature for another 3 h. The reaction was monitored by TLC. After removing DMF under reduced pressure, water and ethyl acetate were added and the two phases were separated. The organic phase was washed with brine 3 times, dried over Na_2SO_4, filtered, and evaporated under vacuum. The crude was purified by chromatography on silica gel. 1.26 g of **compound 1** was obtained as a colorless oil (yield=91%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 – 7.19 (m, 7H), 7.08 (d, J = 8.3 Hz, 2H), 5.14 (s, 2H), 2.96 (t, J = 7.6 Hz, 2H), 2.69 ppm (t, J = 7.6 Hz, 2H).

Benzyl 3-(4-(thiophen-3-yl) phenyl) propanoate (2): A flask was charged, under an atmosphere of nitrogen, with $Pd(PPh_3)_4$ (230 mg, 0.2 mmol), **1** (400 mg, 1.25 mmol) and toluene (8 mL). 2 M Na_2CO_3 (aq.) (4 mL) and a solution of 3-thiophene boronic acid (300 mg, 2.38 mmol) in EtOH (2 mL) were added to the reaction mixture, which was stirred at 90°C for 5 h. After cooling to rt, the mixture was partitioned between 1

M HCl (aq.) and ethyl acetate. The organic layer was separated and washed with brine 3 times, dried over Na_2SO_4, and concentrated under vacuum. The crude product was purified by chromatography on silica gel using petroleum ether/ethyl acetate (10:1) as eluant affording a white solid (350 mg, yield=87%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, J = 8.1 Hz, 2H), 7.44 (d, J = 2.0 Hz, 1H), 7.42 – 7.30 (m, 7H), 7.24 (d, J = 8.0 Hz, 2H), 5.14 (s, 2H), 3.02 (t, J = 7.7 Hz, 2H), 2.73 ppm (t, J = 7.7 Hz, 2H).

3-(4-(Thiophen-3-yl) phenyl) propanoic acid (3): A solution of LiOH (120 mg, 4.84 mmol) in water (1 mL) was added into a stirred solution of **2** (390 mg, 1.21 mmol) in MeOH (20 mL). After stirring for 18 h, the mixture was partitioned between 2 M HCl (aq.) and EtOAc. The organic layer was separated, washed with brine, dried over Na_2SO_4, and concentrated under vacuum. After recrystallization using EtOAc, an off-white solid (240 mg, 85%) was obtained. ^{1}H NMR (400 MHz, DMSO-d_6): δ 12.16 (s, 1H), 7.81 (s, 1H), 7.62 (d, J = 7.1 Hz, 3H), 7.53 (d, J = 4.8 Hz, 1H), 7.27 (d, J = 7.7 Hz, 2H), 2.84 (t, J = 7.3 Hz, 2H), 2.56 ppm (t, J = 7.5 Hz, 2H). The ^{1}H NMR spectrum is in agreement with the literature.[1]

Polythiophene phenylpropionic acid (PPA): 480 mg anhydrous $FeCl_3$ were dissolved into 5 mL anhydrous $CHCl_3$ under nitrogen, and the solution was stirred for 0.5 h. A solution of **3** (100 mg) dissolved in 5 mL $CHCl_3$ was added dropwise. After stirring for 2 days, the resulting precipitate was collected by filtration, washed with methanol until $FeCl_3$ was completely removed, and dried under vacuum to give PPA as a dark red solid (80 mg, 80%).

Synthesis of RCNDs: 20 mg of **PPA** was dispersed in 40 mL of NaOH solution (1 mM). The mixture was treated ultrasonically for 3 h and then transferred into an autoclave and heated at 220°C for a period of 48 h. After cooling to rt, the RCNDs were collected through filtering with 0.22 μm membranes (Millipore, PTFE) for 3 times. The filtrate was dialyzed (1 kDa membrane) against deionized (DI) water for 5 days to remove the residual NaOH. After lyophilization, a red powder (7 mg) was obtained.

4. Synthesis of FA-TEG-NH_2

***Tert*-butyl (2-(2-(2-aminoethoxy) ethoxy) ethyl) carbamate (4):** To a cooled solution (0°C) of 2'2-(ethylenedioxy)bis(ethylamine) (30.0 g, 203 mmol) in 200 mL chloroform, a solution of di-*tert*-butyl dicarbonate (4.425 g, 20.3 mmol) in 200 mL chloroform was dropwise added under inert atmosphere (N_2) over 2 h. After complete addition, the mixture was allowed to warm to rt and then stirred for 24 h. The organic solvent was removed and the liquid was dissolved in 100 mL deionized (DI) water. The aqueous phase was extracted 3 times with 100 mL methylene chloride. The organic layer was separated, washed with brine, dried over Na_2SO_4, and concentrated under vacuum, resulting in 4.5 g of a colorless oil (yield= 90 %). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.13 (s, 1H), 3.57 (s, 4H), 3.48 (m, 4H), 3.26 (m, 2H), 2.82 (t, J = 5.2 Hz, 2H), 1.39 ppm (s, 9H). The ^{1}H NMR spectrum is in agreement with literature.[2]

(*tert*-Butyl *N*-(2-[2-(2-aminoethoxy)ethoxy]ethyl)carbamate) folic acid (5): Folic acid (440 mg, 1 mmol) was suspended in 10 mL of anhydrous DMSO under argon atmosphere and sonicated for 0.5 h. EDC·HCl (383 mg, 2 mmol) and NHS (230 mg, 2 mmol) were added and the resulting suspension was stirred in an ice bath for 4 h before **4** (298 mg, 1.2 mmol) was added. The mixture was stirred below 0°C for 18 h. After removing the ice bath to reach rt, the mixture was poured into 200 mL of cold diethyl ether, filtered, and the resulting precipitate was thoroughly washed with cold diethyl ether until trace DMSO was removed. The solid was dissolved in acetonitrile and precipitated again by pouring in cold diethyl ether. After filtration and drying under vacuum, the crude product was purified by chromatography on silica gel using chloroform/EtOH/acetone/NH_3·H_2O (30%) = 2:2:2:1 as eluant. After drying under vacuum, a yellow solid (180 mg, yield=36%) was obtained. ^{1}H NMR (400 MHz, DMSO-d6): δ 8.64 (s, 1H), 8.42 – 8.04 (m, 3H), 7.93 (dd, J = 7.0, 3.0 Hz, 2H), 7.63 (d, J = 7.6 Hz, 2H), 7.15 (s, 2H), 6.98 – 6.86 (m, 1H), 6.74 (s, 1H), 6.64 (t, J = 11.1 Hz, 1H), 4.48 (d, J = 4.1 Hz, 2H), 4.19 (dd, J = 11.9, 8.4 Hz, 1H), 3.61 (t, J = 6.3 Hz, 1H),

3.47 (s, 4H), 3.23 – 3.13 (m, 2H), 3.06 (d, J = 5.4 Hz, 2H), 2.84 (s, 1H), 2.34 (t, J = 6.3 Hz, 1H), 2.20 – 2.09 (m, 2H), 2.06 – 1.80 (m, 2H), 1.37 (s, 6H), 1.28 ppm (s, 3H) (see spectrum in part S3). LC-MS (ESI): *m/z* calculated for $C_{30}H_{41}N_9O_9$: 671.30, found 672.13 $[M + H]^+$. HPLC (t_R=8.5 min over 20 min of 0.6 mL·min^{-1} mobile phase (90% acetonitrile and 10% water)).

***N*-(2-[2-(2-Aminoethoxy)ethoxy]ethyl) folic acid (6):** In a round-bottom flask, 50 mg of compound **1** was mixed with 1 mL of trifluoracetic acid (TFA) and stirred for 2 h at rt. TFA was removed under reduced pressure and the crude product was dissolved in 2 mL DMF. Yellow precipitate came out after adding the mixture to cold Et_2O (100 mL). After filtering, the precipitate was washed with Et_2O 3 times. After drying under reduced pressure, a light yellow solid was obtained (35 mg, yield=82%). ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.65 (s, 1H), 8.22 – 8.01 (m, 1H), 7.97 – 7.78 (m, 1H), 7.65 (d, J = 7.8 Hz, 2H), 7.17 – 6.82 (m, 3H), 6.65 (d, J = 7.8 Hz, 2H), 4.50 (s, 2H), 4.35 – 3.97 (m, 1H), 3.80 – 3.45 (m, 6H), 3.20 (d, J = 4.5 Hz, 4H), 3.10 – 2.91 (m, 3H), 2.87 – 2.70 (m, 1H), 2.59 (s, 1H), 2.35 – 2.11 (m, 2H), 2.11 – 1.85 ppm (m, 2H) (see spectrum in part S3). LC-MS (ESI): *m/z* calculated for $C_{25}H_{23}N_9O_7$: 571.25, found 572.18 $[M + H]^+$. HPLC (t_R=6 min over 20 min of 0.6 mL·min^{-1} mobile phase (90% acetonitrile and 10% water)).

The ^{1}H NMR spectra of FA, compounds **5** and **6** are shown below (Section S3).

5. Synthesis of RCND-TEG-FA

Conjugation of FA-TEG-NH_2 to RCNDs: A suspension of RCNDs (10 mg) in DI water (10 mL) was sonicated at an ice bath for 10 min. NHS (40 mg, mmol), EDC·HCl (128 mg), and triethylamine (96 μL) were added, and the mixture was stirred for 4 h. Afterwards, a solution of **6** (30 mg) in DMSO (1 mL) was carefully added and then stirred for another 20 h at rt. The mixture was dialyzed (1 kDa membrane) against DI water for 3 days. After lyophilization, a red solid (9.5 mg) was obtained.

6. Dispersibility of RCND-TEG-FA and RCNDs

RCNDs or RCND-TEG-FA at a concentration of 100 μg·mL^{-1} was added to 1 mL DI water, 0.01 M pH=7.4 PBS (without Ca^{2+} and Mg^{2+}), saline (0.9%), or cell medium. After sonication for 10 min, the samples were allowed to stay for 4 days.

7. Calculation of reactive oxygen species (ROS)

Dihydrorhodamine-123 (DHR123) purchased from Sigma-Aldrich was used to measure ROS generation in solution. Oxidation of DHR123 by ROS results in the formation of fluorescent Rhodamine 123. In a typical assay, RCNDs (25 μg·mL^{-1}) or RCND-TEG-FA (25 μg·mL^{-1} of RCNDs) were added to an aqueous solution of DHR123 (20 nM). Then, the mixture was irradiated using a 660 nm laser (0.1 W·cm^{-2}) for 0-10 min, and the emission intensity at 530 nm was measured upon excitation at 485 nm.

8. ROS generation mechanism

DHR 123 was used to detect the radicals generated with/without the relevant radical scavengers. Thiourea and glutathione (GSH) were chosen as inhibitors $^{\cdot}OH$ and $O_2^{\cdot-}$, respectively. RCND-TEG-FA and DHR123 were added to 1 mL DI water. The final concentration of RCND-TEG-FA was 25 μg·mL^{-1} and the final concentration of DHR was 2 μM. For inhibition experiments, 2 μM thiourea or 2 μM GSH was added to the mixture. The sample was irradiated under a 660 nm laser (0.1 W·cm^{-2}) for 15 min, and the emission intensity at 520 nm was measured (excitation 485 nm) before and after the irradiation.

For the oxygen exclusion assay, 1 mL DI water was firstly flushed with argon for 15 min. Then RCND-TEG-FA and DHR123 were added to the DI water. The final concentration of RCND-TEG-FA was 25 μg·mL^{-1} and the final concentration of DHR123 was 2 μM. The mixture was irradiated under argon atmosphere using a 660

nm laser (0.1 W·cm^{-2}) for 15 min, and the emission intensity at 520 nm were measured with excitation of 485 nm before and after the irradiation.

9. Calculation of singlet oxygen

ABDA (9,10-anthracenediyl-bis dimalonic acid) purchased from Sigma-Aldrich was used to measure $^1O_2$ generation in solution. ABDA can react with $^1O_2$ undergoing a Diels-Alder cycloaddition, showing a decrease of the absorption intensity at 360, 378, and 400 nm. RCND-TEG-FA (25 μg·mL^{-1}) was added to an aqueous solution of ABDA (125 μM) with different conditions. Then, the UV-Vis spectra were collected after laser irradiation at 660 nm (0.1 W·cm^{-2}) for 0-60 min.

10. Calculation of singlet oxygen quantum yield

To calculate the singlet oxygen quantum yield, ABDA was used as the $^1O_2$ indicator, and methylene blue was used as the standard reference. In brief, RCND-TEG-FA (25 μg·mL^{-1}) was added to an aqueous solution of ABDA (125 μM). The mixture was exposed to 660 nm laser with a power density of 0.1 W cm^{-2}. To avoid the inner-filter effect, the absorption of MB and RCND-TEG-FA at 660 nm was regulated to approximately 0.1 OD. The absorption of ABDA at 400 nm were collected and plotted at different irradiation time. The times for the decay of ABDA were calculated by applying a first-order exponential fitting to the plot curves.[3,4] The singlet oxygen quantum yield of RCND-TEG-FA in water was calculated according to:

$$\eta_{CNDs} = \eta_{MB} \times \frac{t_{CNDs}}{t_{MB}} \times \frac{I_{MB}}{I_{CNDs}}$$

Where I_{MB} is the absorption of MB at 660 nm, I_{CNDs} is the absorption of RCNDs-TEG-FA at 660 nm, t_{MB} is the time for the decay of ABDA in MB solution, t_{CNDs} is the time for the decay of ABDA in RCND-TEG-FA solution, η_{MB} refers the $^1O_2$ quantum yield of MB in water (η_{MB}=0.52). From the Figure S7, t_{MB} is 582,

t_{CNDs} is 330, I_{MB} is 0.097, I_{CNDs} is 0.072. Therefore, the $^1O_2$ quantum yield of CNDs η_{CNDs} was calculated to be 0.4.

11. Cell culture

HeLa (epithelial, human cervical adenocarcinoma) cells were cultured as monolayers in Dulbecco's modified Eagle medium (DMEM) supplemented with 10 μg·mL^{-1} gentamycin, 10 mM *N*-(2-hydroxyethyl)-piperazine-*N*-ethanesulfonic acid, 0.05 mM β-mercaptoethanol, and 10% fetal bovine serum (FBS) at 37°C in a 5% CO_2 incubator.

12. Confocal imaging

Confocal images were obtained with a Zeiss Axio Observer Z1 spinning disk confocal microscope equipped with a 63 or 100 X oil objective. z-Stacking was recorded with 0.3 μm interplanar distance. The fluorescence signal from CellMask (Sigma-Aldrich) was obtained using a 488 nm laser excitation and recorded in the green channel (505-555 nm), whereas RCNDs and RCND-TEG-FA were recorded using a 405 nm laser excitation in the far-red (FR) channel (665-715 nm). Images were then treated with ImageJ software.

13. Cytotoxicity assay

The cytotoxicity of RCND-TEG-FA was evaluated by alamarBlue® assay. Briefly, HeLa cells were seeded in 96-well plates at a density of 2000 cells per well and incubated at 37°C for 24 h. Then RCND-TEG-FA at varying concentrations (0, 1, 10, 25, 50, 100 mg·mL^{-1}) were added to cells and incubated for another 24 h. The culture media were discarded, and 0.1 mL of the alamarBlue solution (10% in cell medium) was added to each well, followed by incubation at 37°C for 4 h. Absorbance values of the wells were read with a microplate reader (Thermo Scientific, Multiskan FC) at 570 nm and 620 nm.

14. Cell imaging and assessment of particle endocytosis

For fluorescence imaging, the adherent cells grown on glass-bottom culture dishes containing 0.5 mL of culture medium were first incubated with CellMask for 2 h. After washing with cell culture medium 2 times, the cell lines were incubated with RCNDs or RCND-TEG-FA (20 μg·mL^{-1} or 100 μg·mL^{-1}) for 24 h at 37℃. The fluorescence imaging was taken at 0, 4, 8, and 24 h. To confirm the receptor-mediated uptake, competition experiments were conducted where the cell culture was pre-treated with 50 μL of saturated FA solution for 1.5 h prior to RCND-TEG-FA treatment. Confocal images were obtained. The saturated FA solution was prepared according to the following procedure: 10 mg of FA was added to 1 mL PBS and the suspension was sonicated for 10 min. After centrifugation at 10000 rpm for 20 min, the supernatant was collected.

15. Intracellular ROS formation

HeLa cells were seeded in a 8-well chamber slide (Thermo Scientific) (approximately 1×10^4 cells·cm^{-2}, 500 μL/well), cultured for 24 h, and then cultured for 24 h in RCND-TEG-FA solution (0.5 mL, 100 μg·mL^{-1}). The culture media were discarded, then CellROX reagent (Life Technologies, Inc.) at a final concentration of 5 μM was added to the cells and incubated for 30 min. The culture media were discarded, and the HeLa cells were washed with cell culture medium 2 times. Irradiation was performed using a 660 nm laser at 0.1 W·cm^{-2} for 10 min and the fluorescence imaging experiments were carried out.

16. *In vitro* PDT experiments

HeLa cells (2.0 × 10^3/well) were seeded on a 96-well microplate with optically clear bottom (Greiner bio-one, Germany) overnight. The cells were preincubated with RCND-TEG-FA at different concentrations (0, 20, 50, 100 μg·mL^{-1}) for 24 h. Then, cells were gently washed with cell medium twice. Samples (n = 6) with a total volume

of 100 µL in 96-well plates were irradiated for 10 min with a 660 nm laser (0.1 $W \cdot cm^{-2}$). After 24 h incubation, the culture media were discarded, and 0.1 mL of the alamarBlue solution (10% in cell medium) was added to each well, followed by incubation at 37℃ for 1 h. Absorbance values of the wells were read with a microplate reader (Multiskan FC, Thermo scientific) at 570 nm and 620 nm.

S2 Additional figures S1-S8

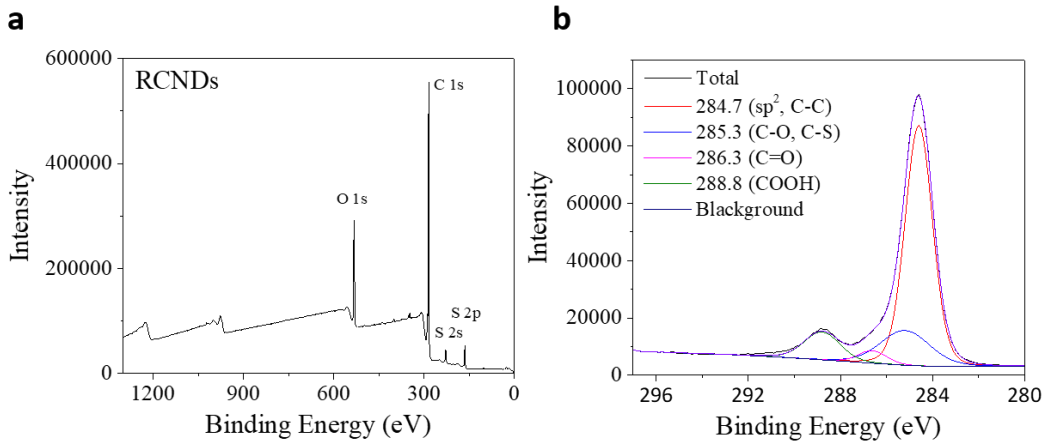


Figure S1. XPS spectra of survey (a) and high resolution C1s (b) for RCNDs.

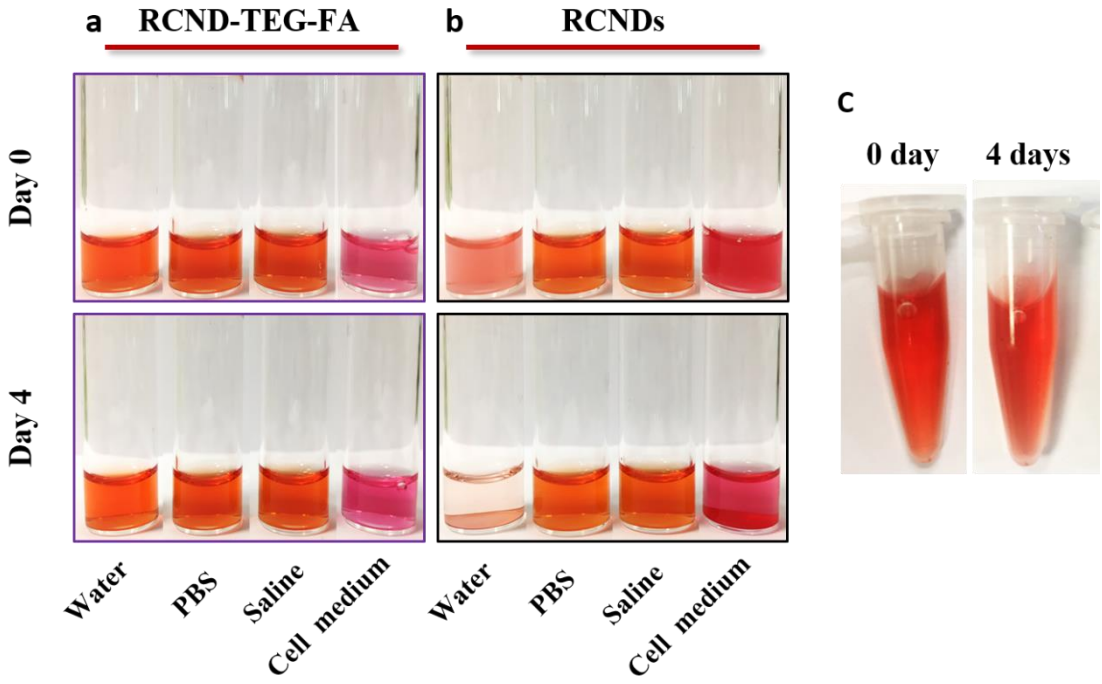


Figure S2. Digital photos of a) RCND-TEG-FA and b) RCNDs (both at 100 μg·mL^{-1}), dispersed in DI water, PBS (0.01 M), saline solution, and cell culture medium, respectively; c) Digital photos of RCND-TEG-FA (1 mg·mL^{-1}) stored in cell culture medium for 4 days.

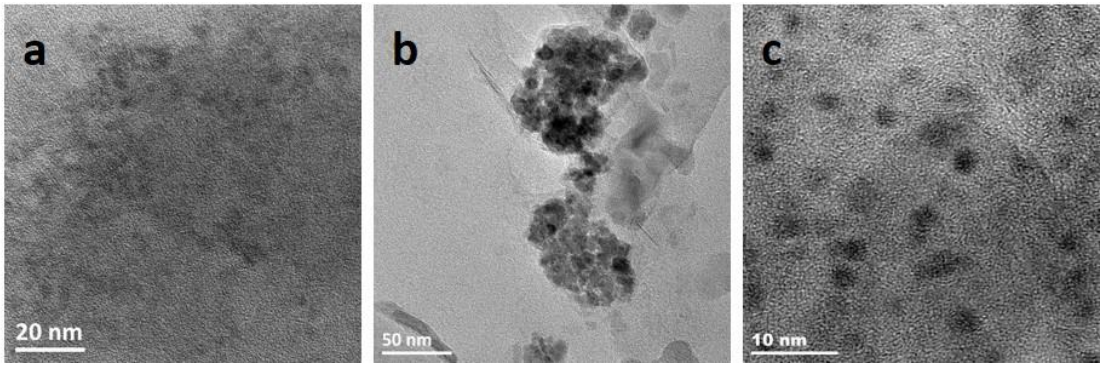


Figure S3. HRTEM of a) RCNDs in alkaline, b) RCNDs in DI water and c) RCND-TEG-FA in DI water.

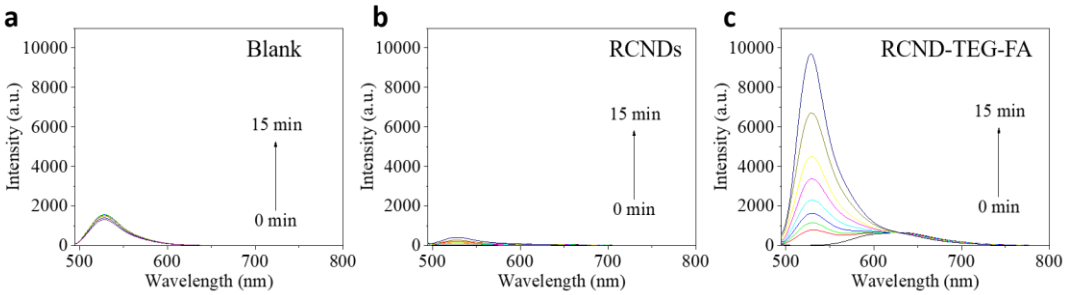


Figure S4. (a) Time-dependent fluorescence titration of DHR123 (20 nM) without (a) or with (b) RCNDs (25 μg·mL^{-1}) and (c) RCND-TEG-FA (25 μg·mL^{-1} of RCNDs). All measurements were carried out in DI water with an excitation of 485 nm.

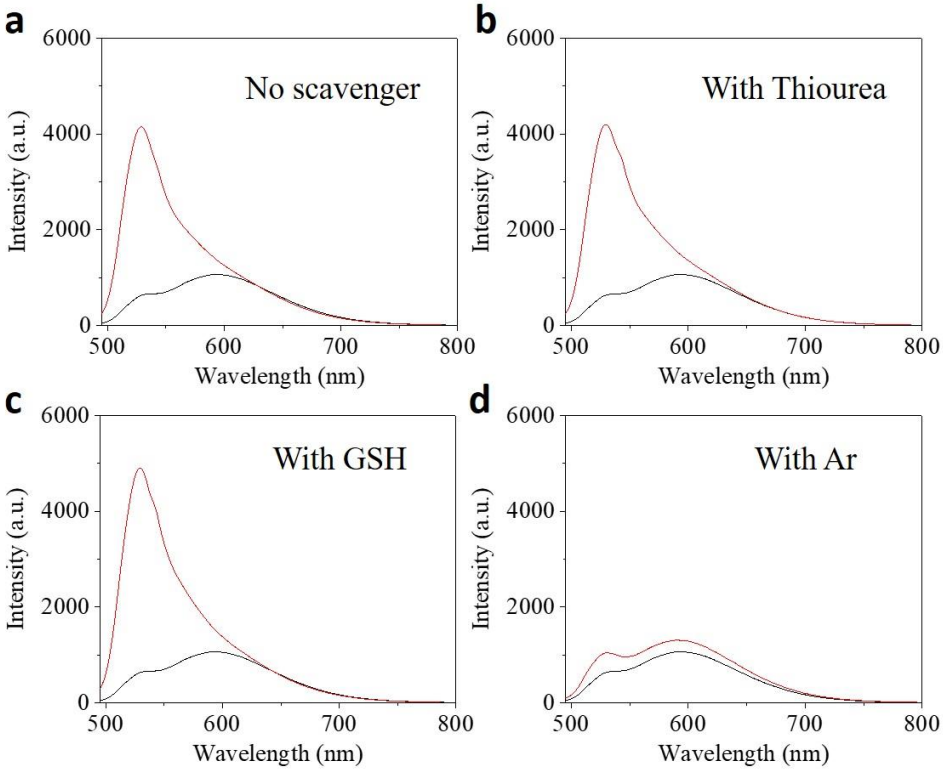


Figure S5. Fluorescence differences of DHR123 with RCND-TEG-FA (25 μg·mL^{-1}) before (black line) or after (red line) 660 nm laser irradiation (0.1 W·cm^{-2}), without scavenger a), or with b) thiourea, c) GSH, and under inert atmosphere d) Ar. All measurements were carried out in DI water with an excitation at 485 nm.

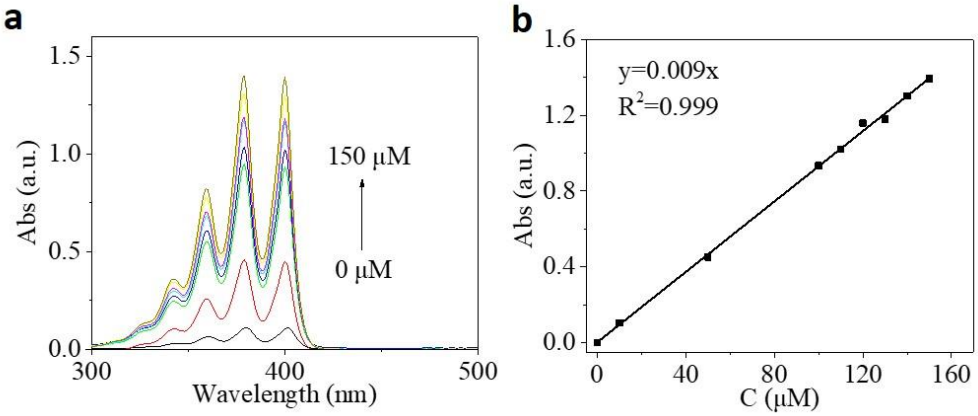


Figure S6. a) The absorption spectra of ABDA with different concentrations (0-150 μM); b) The standard curve of ABDA based on its absorption at 400 nm.

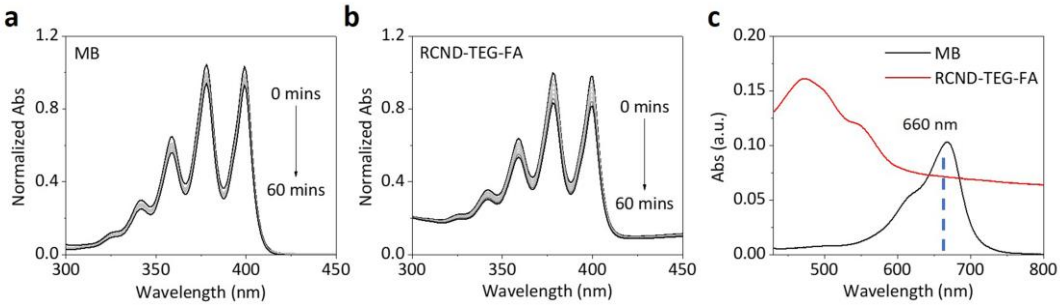


Figure S7. Photodegradation of ABDA with a) MB and b) RCND-TEG-FA under 660 nm laser irradiation (0.1 W cm^{-2}); c) The absorbance of RCND-TEG-FA and MB at 660 nm.

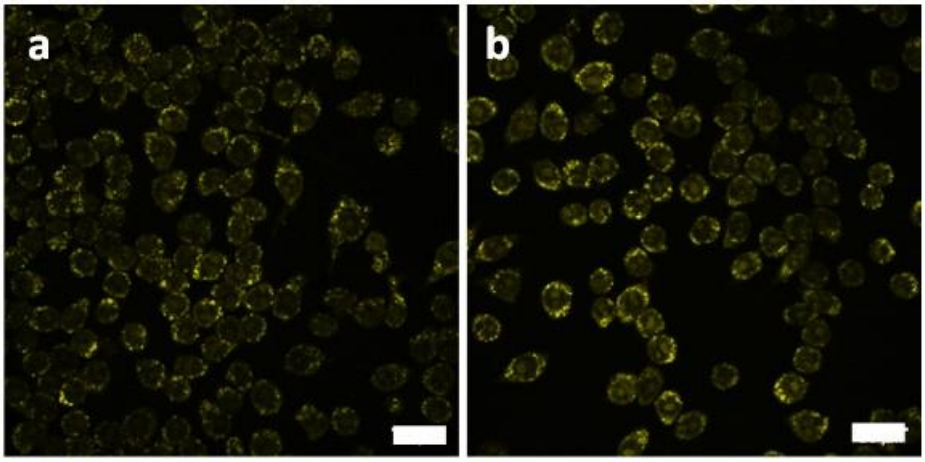
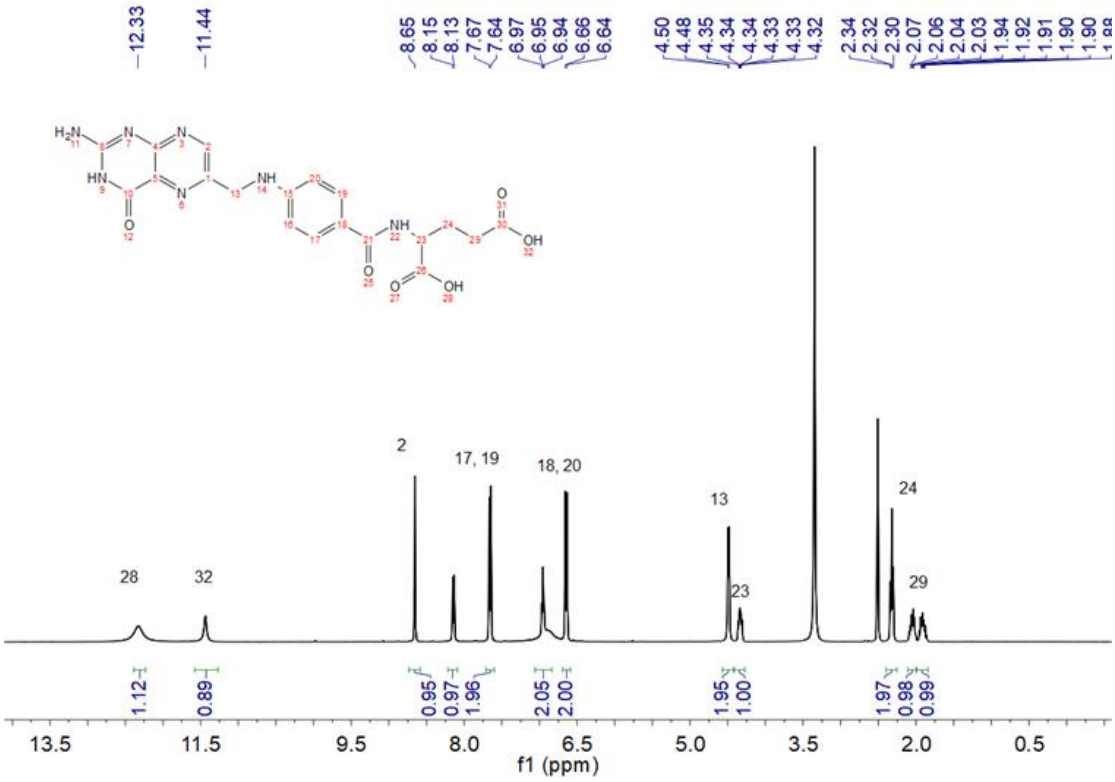


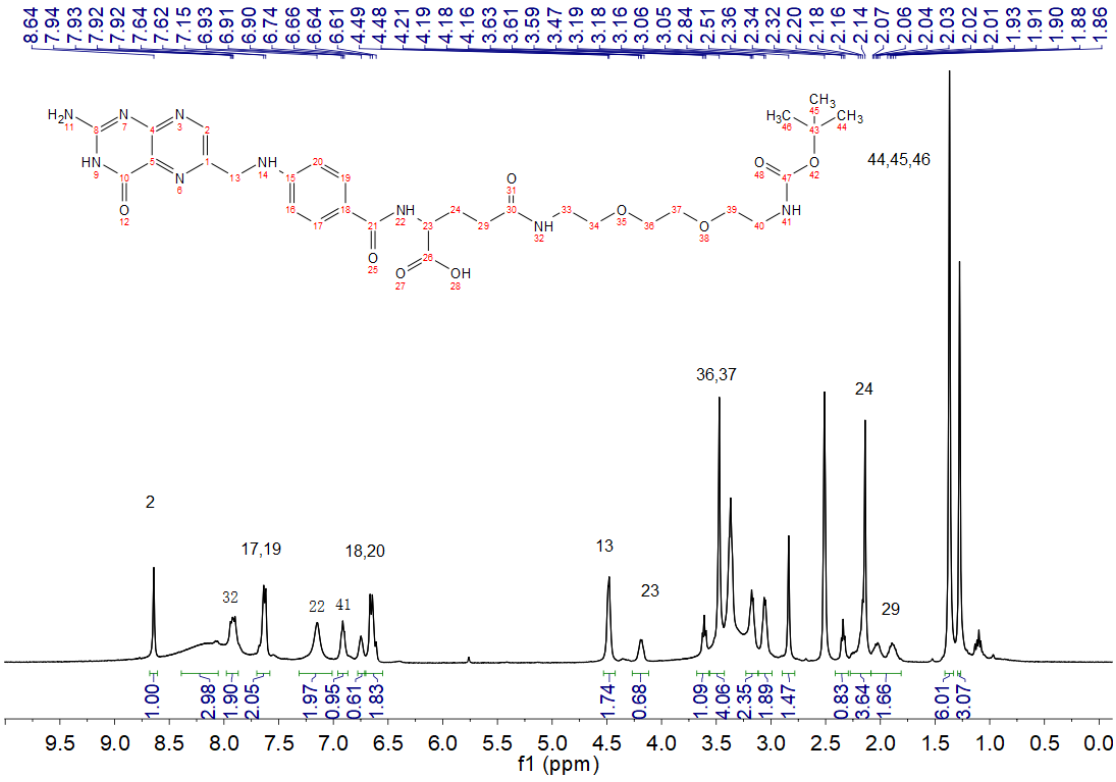
Figure S8. HeLa cells images with CellROX reagent for 30 min incubation without (a) or with (b) a low-dose 660 nm laser irradiation (0.1 $W \cdot cm^{-2}$) for 10 min. Scale bar is 20 μm.

S3 NMR spectra of FA and FA ligands

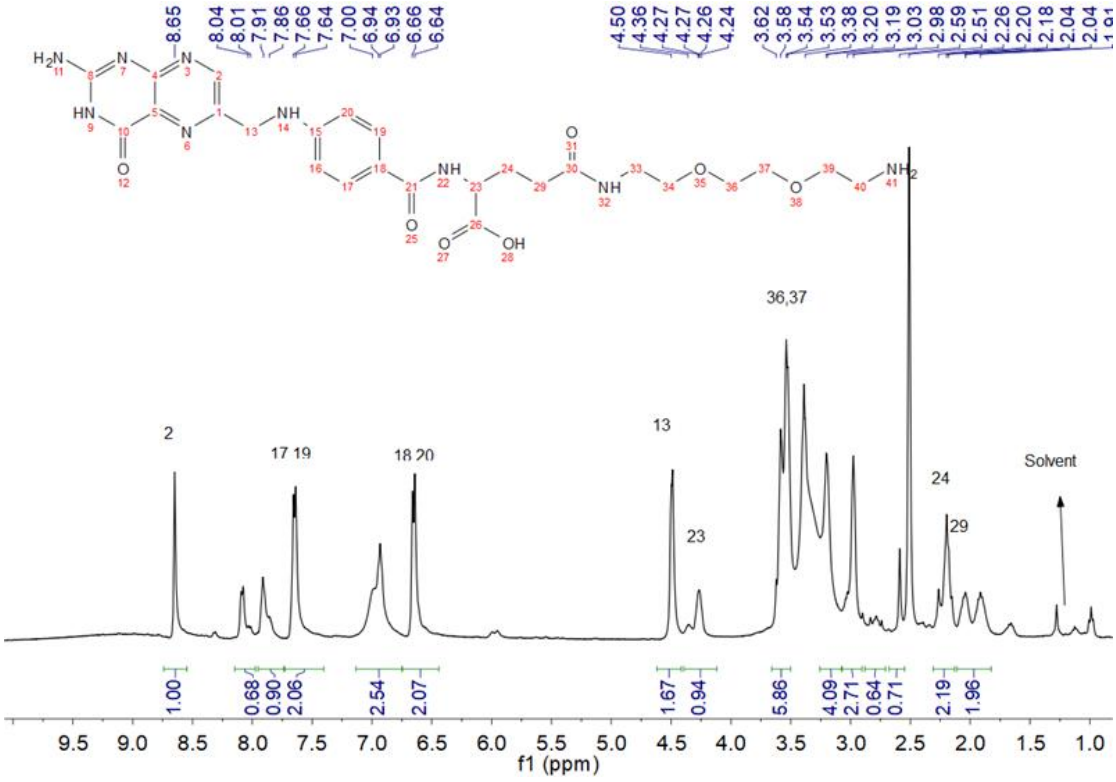
NMR spectrum of folic acid



NMR spectrum of compound **5**



NMR spectrum of compound **6**



Reference:

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