Journal Name

Supporting Information

## 808 nm Photocontrolled UCL Imaging Guided Chemo/Photothermal Synergistic Therapy with Single UCNPs-CuS@PAA Nanocomposite

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Figure S1. The zeta-potentials of UCNPs-CTAB, UCNPs@mSiO<sub>2</sub>, UCNPs@mSiO<sub>2</sub>-NH<sub>2</sub> and CuS nanoparticles.



Figure S2. TEM image (a) and dynamic light scatter measurement (b) of CuS nanoparticles.

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**Figure S3.** (a-c) TEM images of UCNPs@mSiO<sub>2</sub>@CuS nanoparticles with different amounts of CuS nanoparticles attached on the surface of UCNPs@mSiO<sub>2</sub>. The white arrow in (a) refers to the redundant CuS nanoparticles. The scale bar in each image is 100 nm. (d) High angle annular dark field scanning transmission electron microscopy (HAADF-STEM) images and HAADF-STEM-EDS mapping images of UCNPs@mSiO<sub>2</sub>@CuS nanoparticles.



**Figure S4.** TEM images of UCNPs@mSiO<sub>2</sub>@CuS nanoparticles with different reaction times: (a) 30 min, (b) 1 h and (c) 2 h.



**Figure S5.** Temperature changes of UCP aqueous solutions with different concentrations over five ON/OFF cycles of 808 nm laser irradiation (2 min NIR irradiation followed by a 10 min cooling period). The concentrations of Cu were determined by ICP measurement.