## Redox responsive UCNPs-DPA conjugated NGO-PEG-BPEI for cancer theranostic

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Fig. S1 XRD pattern of UCNPs. The standard pattern of hexagonal phases NaGdF<sub>4</sub>:Yb<sup>3+</sup>,  $Er^{3+}$ @NaGdF<sub>4</sub> is given for reference (JCPDS No.27-0699).



**Fig. S2** FT-IR spectra of NGO-PEG and NGO. The strong stretching vibration peak of C–H ( $\sim$ 2880 cm<sup>-1</sup>) demonstrated the presence of PEG in the NGO-PEG. The appearance of the new absorption at  $\sim$ 1649 cm<sup>-1</sup> for –CONH– further indicated that PEG has been covalently boned on the surface of NGO successfully.



**Fig. S3** Temperature variation curves of the NGO-PEG solution subjected to the 980 nm laser at a power density of 0.72 W/cm<sup>2</sup>.



**Fig. S4** LSUCLM images of HeLa cells when incubating with UCNPs-DPA-NGO-PEG-BPEI-DOX for 0.5 h, 1 h, and 3 h.



**Fig. S5** Hemolytic assay of UCNPs-DPA-NGO-PEG-BPEI-DOX by human red blood cells.



**Fig. S6** (A) The photothermal response of the UCNPs-DPA-NGO-PEG-BPEI-DOX aqueous solution (200  $\mu$ g/ mL) radiated with 980 nm laser (0.72 W/cm<sup>2</sup>) and then the laser was shut off. (B) Linear time data *versus* –ln  $\theta$  obtained from the cooling period of Fig. S6A. (C) Temperature change of UCNPs-DPA-NGO-PEG-BPEI-DOX under three irradiation/cooling cycles (0.72 W/cm<sup>2</sup>).