

Scheme S1.Schematic illustration of the synthetic approach to the
APTES-functionalizedUVlight-cleavable4-(2-carboxy-ethylsulfanylmethyl)-3-nitro-benzoic acid (CNBA) linker

Synthesis of 4-(2-carboxy-ethylsulfanylmethyl)-3-nitro-benzoic acid (CNBA). 3-

Mercaptopropionic acid (0.5415 g, 5 mmol) was dissolved in 10 mL of deionized water and then neutralized by triethylamine (665 μ L, 4.6 mmol). The solution was cooled to 0 °C, then 4-Bromomethyl-3-nitrobenzoic acid (1.233 g, 4.6 mmol in 10 mL of methanol) was added dropwise over 15 min. The reaction solution was stirred vigorously at 0 °C for 30 min and then at room temperature for 60 min. Next, the solution was rotated evaporation to remove methanol at 44 °C. The obtained yellow sticky substance was dissolved with a small amount of water and the pH was adjusted to 9 with saturated sodium carbonate at 0 °C. Ethylacetate was added, and then the lower solution was collected and adjusted the value of pH to 1-2. Added ethylacetate to the solution again and collected upper solution and rotated evaporation. Finally, by recrystallizing the crude product in hot water, the pale-yellow precipitate was washed

sequentially with ethyl acetate $(3 \times 20 \text{ mL})$ and deionized water $(3 \times 20 \text{ mL})$. ¹HNMR of CNBA (Acetone): $\delta = 2.56-2.60$ (t, 2H), 2.71-2.74 (t, 2H), 4.21-4.23 (d, 2H), 7.82-7.84 (d, 1H), 8.26-8.28 (m, 1H), 8.55-8.56 (d, 1H) (see Figure S1).



Fig. S1 The NMR spectrum of as-synthesized CNBA in acetone.

Synthesis of (H₅C₂O)₃Si(CH₂)₃NH-CNBA-NH(CH₂)₃Si(OC₂H₅)₃. 285 mg (1mmol) of as-synthesized CNBA linker, 191.7 mg (1 mmol) of EDC, 115.1 mg (1 mmol) of NHS and 442.7 mg (2 mmol) of APTES were added to 2 mL of dimethyl sulfoxide, the reaction mixture was reacted for 24 h under stirring at room temperature.



Fig. S2 Low-angle XRD patterns of (a) UCNP@mSiO₂/HA, (b) UCNP@mSiO₂/HA-DOX@CNBA1, (c) UCNP@mSiO₂/HA-DOX@CNBA2 and (d) UCNP@mSiO₂/HA-DOX@CNBA3.

All the samples reveal only one diffraction peak at about 2θ = 2.26, suggesting they possesses the mesoporous structure. Furthermore, the relative intensity of the diffraction collected from UCNP@mSiO₂/HA-DOX@CNBA is obviously reduced compared to that of UCNP@mSiO₂/HA owing to the drug loading and CNBA modification.



Fig. S3 Emission spectra of UCNP@mSiO₂ under 980 nm excitation with different excitation intensities.



Fig. S4 The digital photos under bright and UV light irradiation of (a) UCNP@mSiO2,(b)UCNP@mSiO2/HA,(c)UCNP@mSiO2/HA-DOXand(d)UCNP@mSiO2/HA-DOX@CNBA.

UCNP@mSiO₂ does not reveal fluorescence. The color of UCNP@mSiO₂/HA becomes pink and the red fluorescence is derived from HA. And UCNP@mSiO₂/HA-DOX shows the enhanced red fluorescence owing to the loading of DOX. The change of color and fluorescence further testify the HA graft and drug loading.



Fig. S5 (A) MTT cell viability assay of (UCNP@mSiO₂/HA@CNBA on 293T cells (blue) and MCF-7 (pink) cells for 24 h incubation in dark. (B) MTT cell viability assay of UCNP@mSiO₂/HA-DOX@CNBA on MCF-7 cells without (pink) and with (yellow) 980 nm (0.15 W cm⁻²) irradiation for 20 min.