

Fig. S1 A TEM image of Pd-Fe.

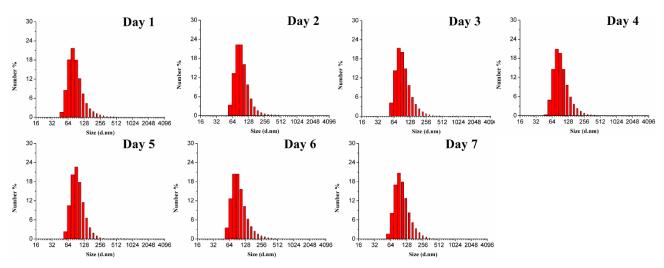


Fig. S2 Stability of HSA-Pd-Fe-Ce6 NAs in DI water.

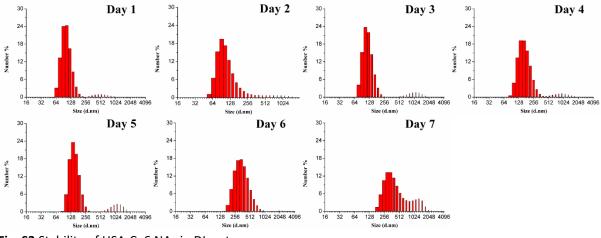


Fig. S3 Stability of HSA-Ce6 NAs in DI water.

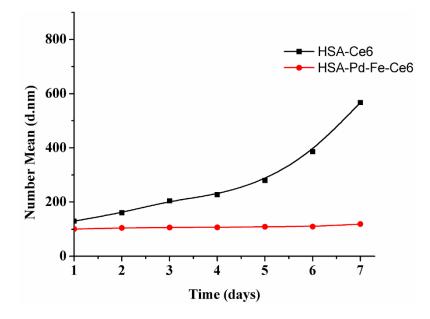
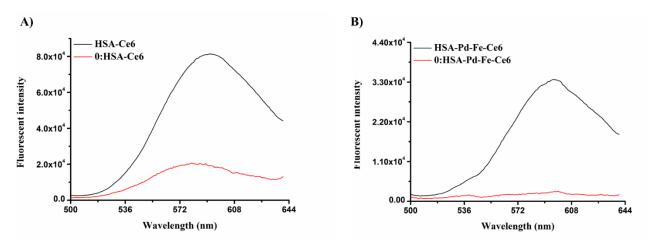
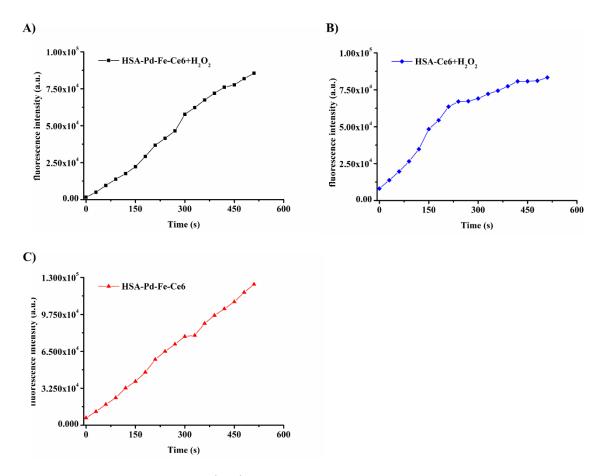


Fig. S4 size change curve of S2 and S3.



**Fig. S5** The fluorescence intensity of dihydroethidium (DHE) of **A)** HSA-Ce6 NPs and **B)** HSA-Pd-Fe-Ce6 NAs after mixed with  $H_2O_2$  (100  $\mu$ M), DNA (1 mg/ml) and DHE (4 mg/ml) in PBS (pH 5.5). 0: refers to the fluorescence intensity before mixed with DHE.



**Fig. S6** The fluorescence intensity of 2', 7' -dichlorofluorescein diacetate (DCFH-DA) **A**) group HSA-Pd-Fe-Ce6 +H<sub>2</sub>O<sub>2</sub>, **B**) group HSA-Pd-Fe-Ce6 and **C**) group HSA-Ce6+H<sub>2</sub>O<sub>2</sub> NPs under 660 nm laser (0.5 w/cm<sup>2</sup>, 30 s) irradiation per 30 s.

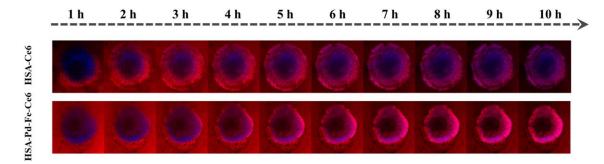


Fig. S7 Images of the 3D spheroids of MCF-7 cells incubated with HSA-Pd-Fe-Ce6 or HSA-Ce6 per hour.

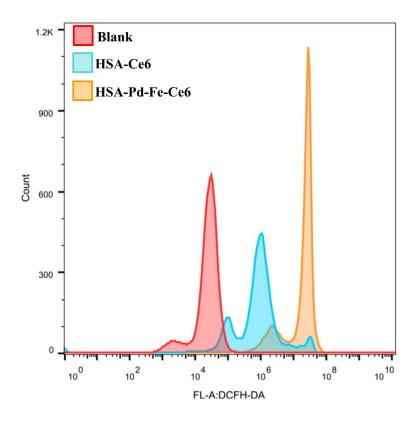


Fig. S8 The generation of ROS determined by the DCFH-DA fluorescence of MCF-7 cells after coincubated with PBS or HSA-Ce6 nanoparticles (20  $\mu$ g/ml) or HSA-Pd-Fe-Ce6 NAs (20  $\mu$ g/ml) with H<sub>2</sub>O<sub>2</sub> (50  $\mu$ M) for 2 h and further 20 minutes laser irradiation.( 660 nm,0.5 w/cm<sup>2</sup>).

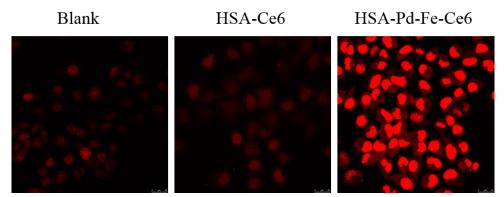
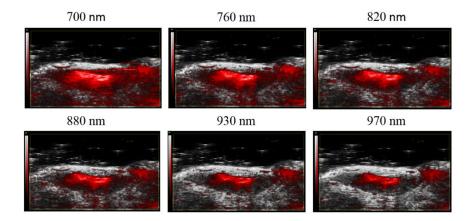


Fig. S9 Use of a free radical indicator to evaluate the production of ROS (  $\cdot$  OH,  $\cdot$  O<sub>2</sub><sup>-</sup>) after incubation of

MCF-7 cells with dihydroethidium and  $H_2O_2$  (50  $\mu$ M).



**Fig. S10** Photoacoustic signals of HSA-Pd-Fe-Ce6 nanoparticles at 700 nm, 760 nm, 820 nm, 880 nm, 930 nm and 970 nm.