# Electronic Supplementary Information

## $CuFeSe_2$-based thermo-responsive multifunctional nanomaterial initiated by a single NIR light for hypoxic cancer therapy

Juan Lu,[a] Jun Yang,*[a] Dan Yang,[b] Shanshan Hu,*[a] Qianqian Sun,[b] Guixin Yang,*[b] Shili Gai,[b] Zhao Wang,[b] and Piaoping Yang*[b]

[a] School of Chemistry and Chemical Engineering, Southwest University, Chongqing 400715, P. R. China

[b] Key Laboratory of Superlight Materials and Surface Technology, College of Materials Science and Chemical Engineering, Ministry of Education, Harbin Engineering University, Harbin 150001, P. R. China

E-mail: jyang@swu.edu.cn; hushan3@swu.edu.cn; yangguixin@hrbeu.edu.cn; yangpiaoping@hrbeu.edu.cn

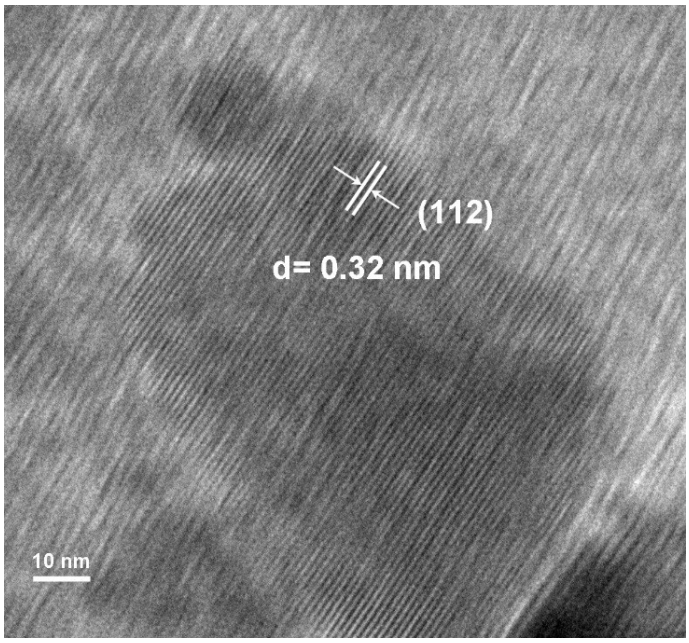


**Fig. S1** HRTEM image of $CuFeSe_2$ nanocrystal.

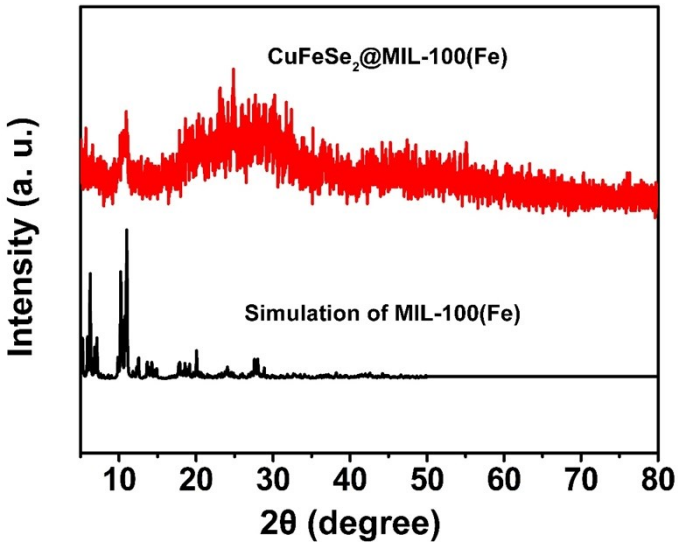


**Fig. S2** The XRD patterns of simulation MIL-100(Fe) and $CuFeSe_2$@MIL-100(Fe) with more layers.

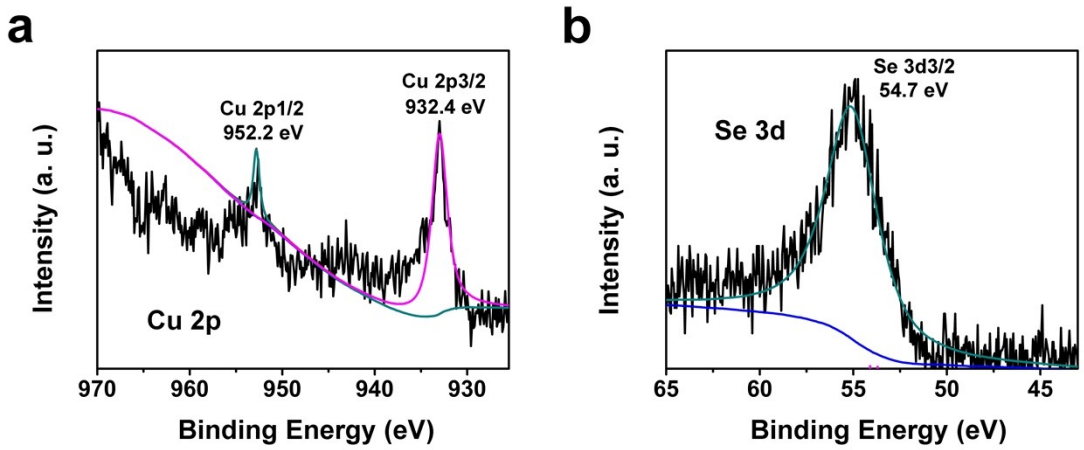


**Fig. S3** XPS spectra of Cu 2p and Se 3d for $CuFeSe_2$@MIL-100(Fe)-AIPH (a,b).

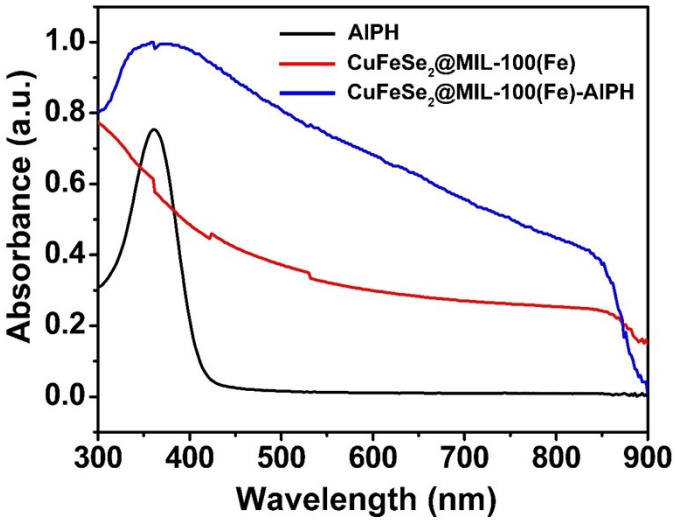


**Fig. S4** UV-vis absorption spectra of AIPH, $CuFeSe_2$@MIL-100(Fe) and $CuFeSe_2$@MIL-100(Fe)-AIPH.

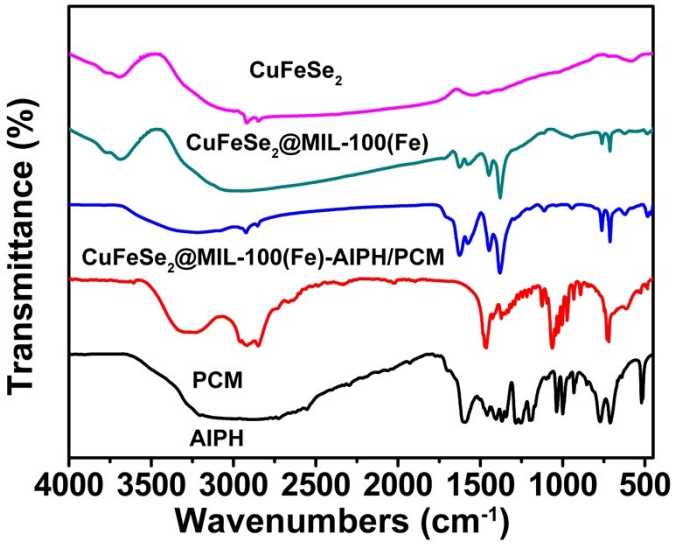


**Fig. S5** FTIR spectra of $CuFeSe_2$, $CuFeSe_2$@MIL-100(Fe), $CuFeSe_2$@MIL-100(Fe)-AIPH, PCM and AIPH.

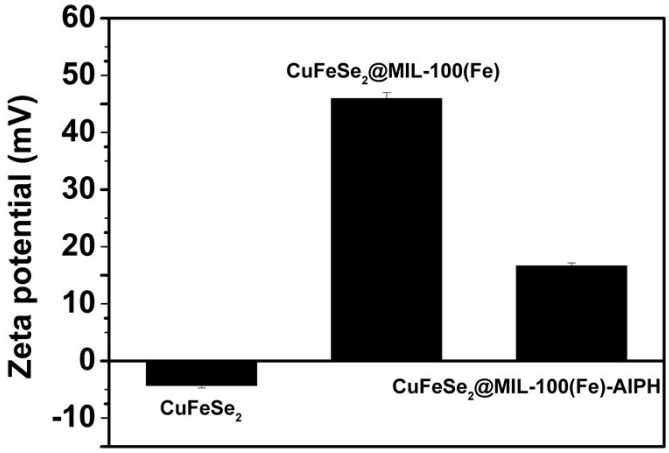


**Fig. S6** The zeta potentials of the $CuFeSe_2$, $CuFeSe_2$@MIL-100(Fe) and $CuFeSe_2$@MIL-100(Fe)-AIPH samples.

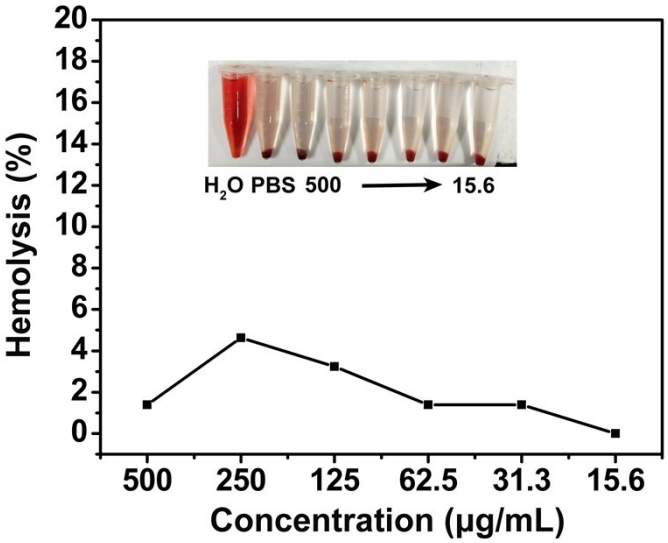


**Fig. S7** Hemolysis ratios of $CuFeSe_2$@MIL-100(Fe)-AIPH solutions with different concentrations treated with red blood cells, PBS as a negative control and deionized water as a positive control respectively. Inset: Hemolysis photographs after centrifugation.

We used the phosphate buffered saline (PBS) and the ultrapure water as the negative and positive control respectively. Firstly, we mixed the purified red blood cells with PBS, ultrapure water and different concentrations of $CuFeSe_2$@MIL-100(Fe)-AIPH solutions respectively. Then we let the mixture stand still at 37 °C for 2 hours and measured the absorbance of the supernatant centrifuged from each group at 490 nm by the ultraviolet-visible spectrophotometer. Finally, we calculated the hemolysis ratio by the following equation: $(A_{sample}\text{-}A_{PBS}) / (A_{water}\text{-}A_{PBS}) \times 100\%$, where $A$ is the absorbance of the UV-vis spectrum. According to the equation, the hemolysis ratios of $CuFeSe_2$@MIL-100(Fe)-AIPH solutions with 250 $\mu$g/mL is calculated to be higher than the other concentrations.

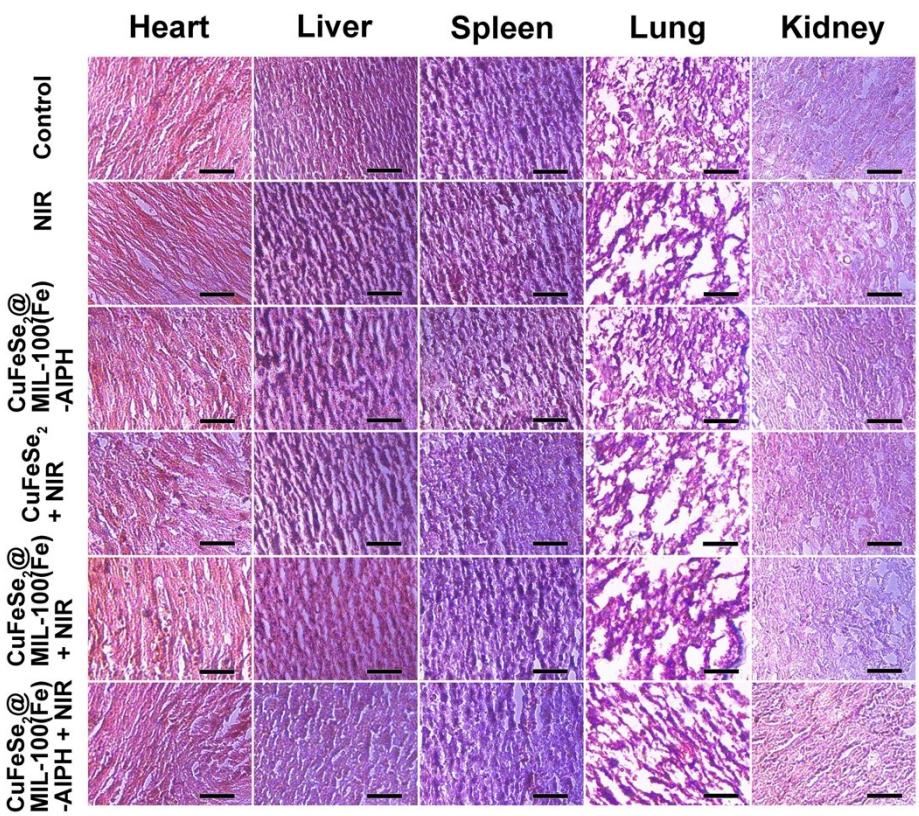


**Fig. S8** H&E stained images of representative organs (heart, liver, spleen, lung and kidney) excised from tumor-bearing mice in various groups after 14 days of therapy. Scale bar: 50 $\mu$m.

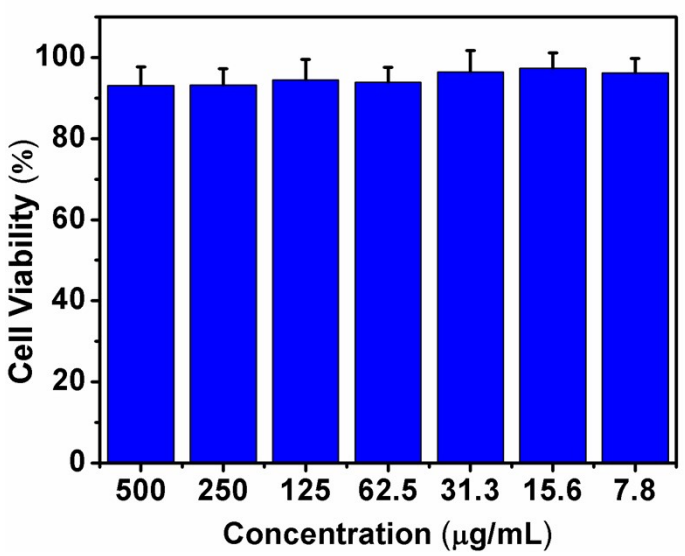


**Fig. S9** *In vitro* cell viabilities of L929 fibroblast cells after incubated with various concentrations of $CuFeSe_2$.

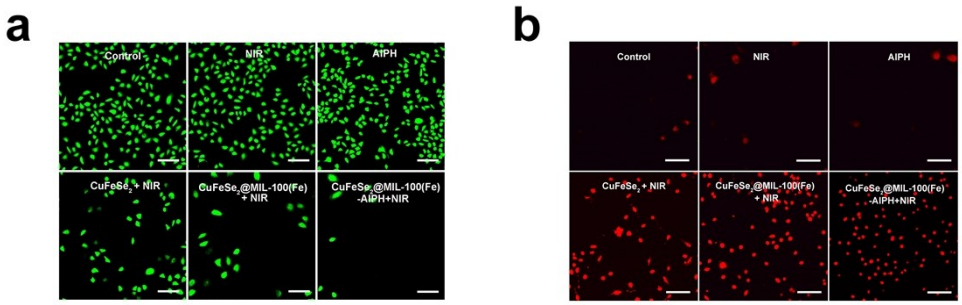


**Fig. S10** CLSM images of U14 cells after different treatments, dyed with AM (living cells, green) and PI (dead cells, red). Scale bar: 100 $\mu$m.

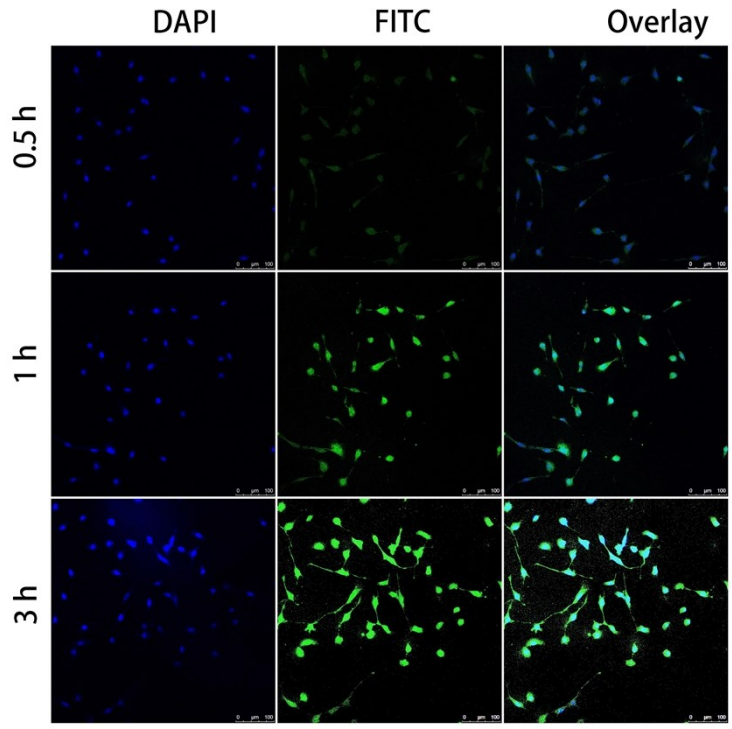


**Fig. S11** CLSM images of U14 cells after culture with FITC-modified $CuFeSe_2$@MIL-100(Fe)-AIPH nanoparticles for 0.5, 1, 3 h.

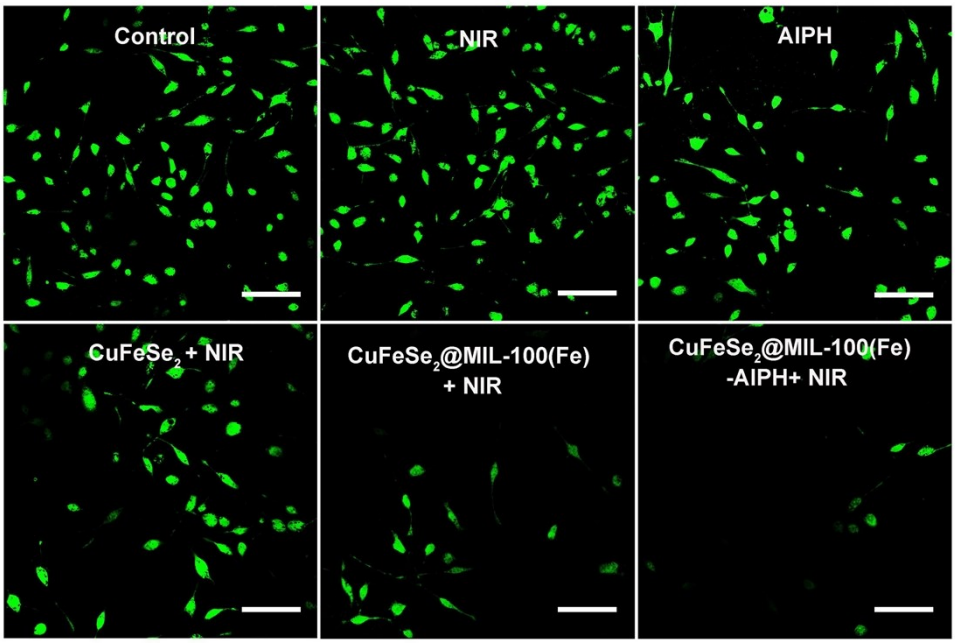


**Fig. S12** CLSM images of HeLa cells after different treatments, dyed with AM (living cells, green). Scale bar: 100 $\mu$m.

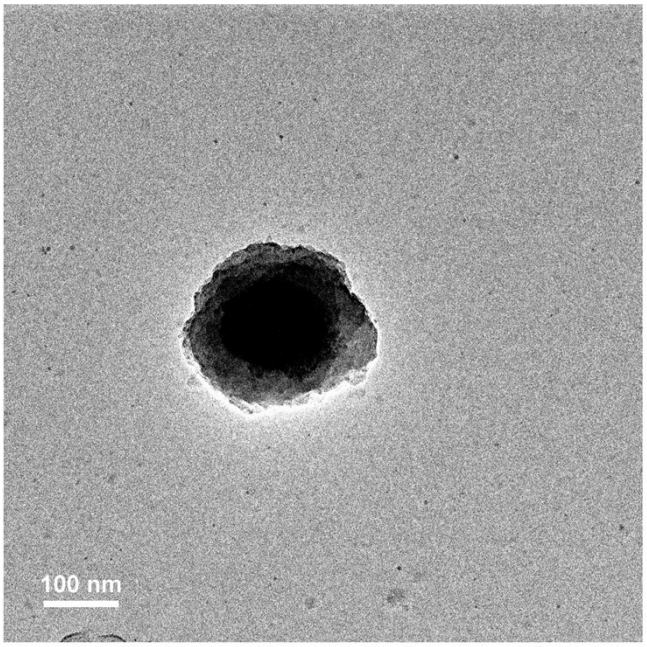


**Fig. S13** TEM images of $CuFeSe_2$@MIL-100(Fe) coated with six layers.

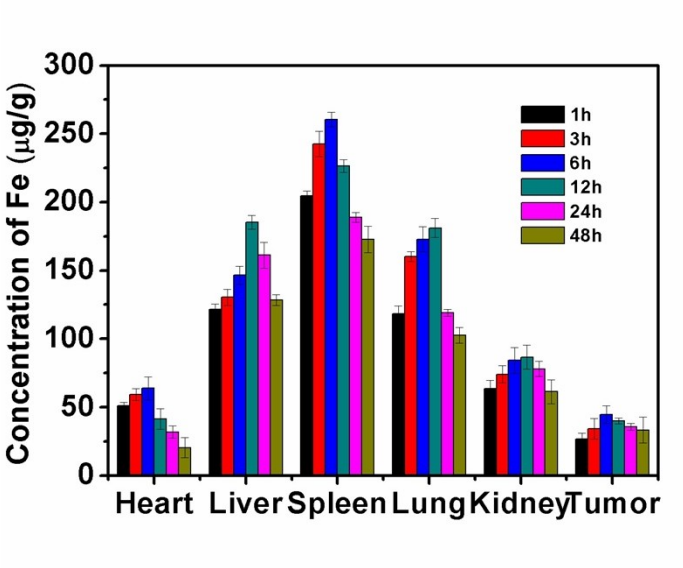


**Fig. S14** The biodistribution of iron element in major organs and tumor of tumor-bearing mice after the injection of $CuFeSe_2$@MIL-100(Fe)-AIPH at different time points. Error bars indicate standard deviations, $N = 3$.

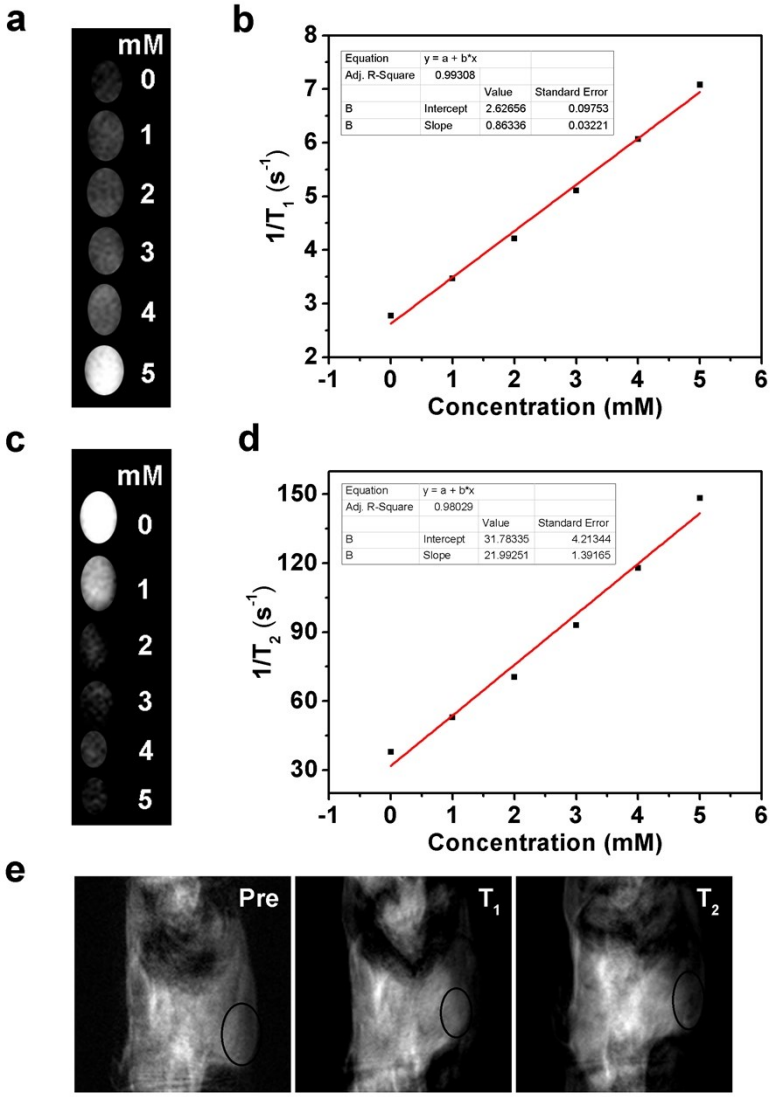


**Fig. S15** *In vitro* $T_1/T_2$-weighted MR images of $CuFeSe_2$@MIL-100(Fe)-AIPH when incubated with PBS (a, c) and corresponding relaxation rate $r_1/r_2$ versus the concentration of Fe (b, d); *In vivo* $T_1/T_2$-weighted MR images of tumor-bearing mice before and after injection of $CuFeSe_2$@MIL-100(Fe)-AIPH (e).

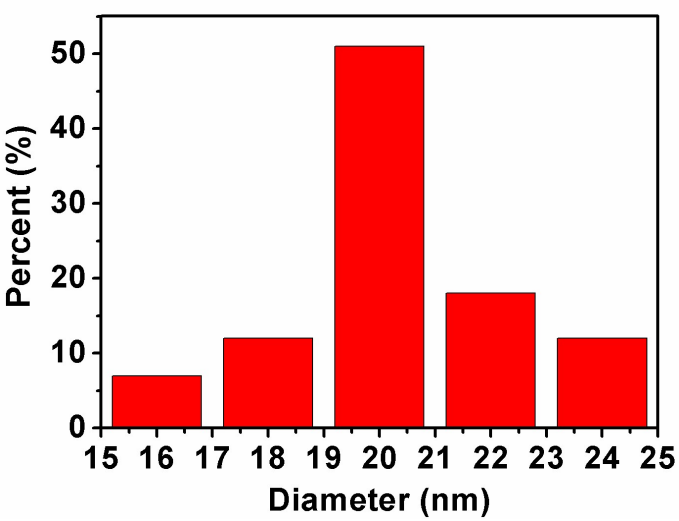


**Fig. S16** The size distribution diagram of $CuFeSe_2$ nanoparticles from the TEM imaging.

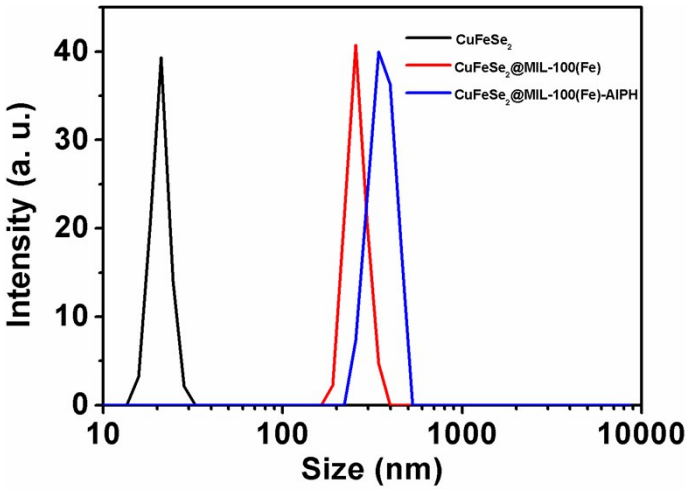


**Fig. S17** The DLS measurements of $CuFeSe_2$, $CuFeSe_2$@MIL-100(Fe) and $CuFeSe_2$@MIL-100(Fe)-AIPH.

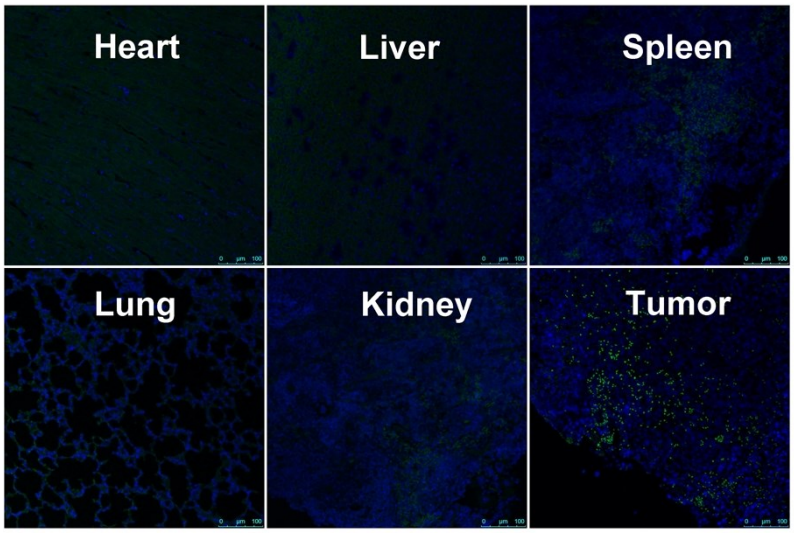


**Fig. S18** The HIF-1α expression (green fluresence) in different tissue sections..