

## Supplementary Material

### Iron oxide nanoflowers @ CuS hybrids for cancer tri-therapy: interplay of photothermal therapy, magnetic hyperthermia and photodynamic therapy

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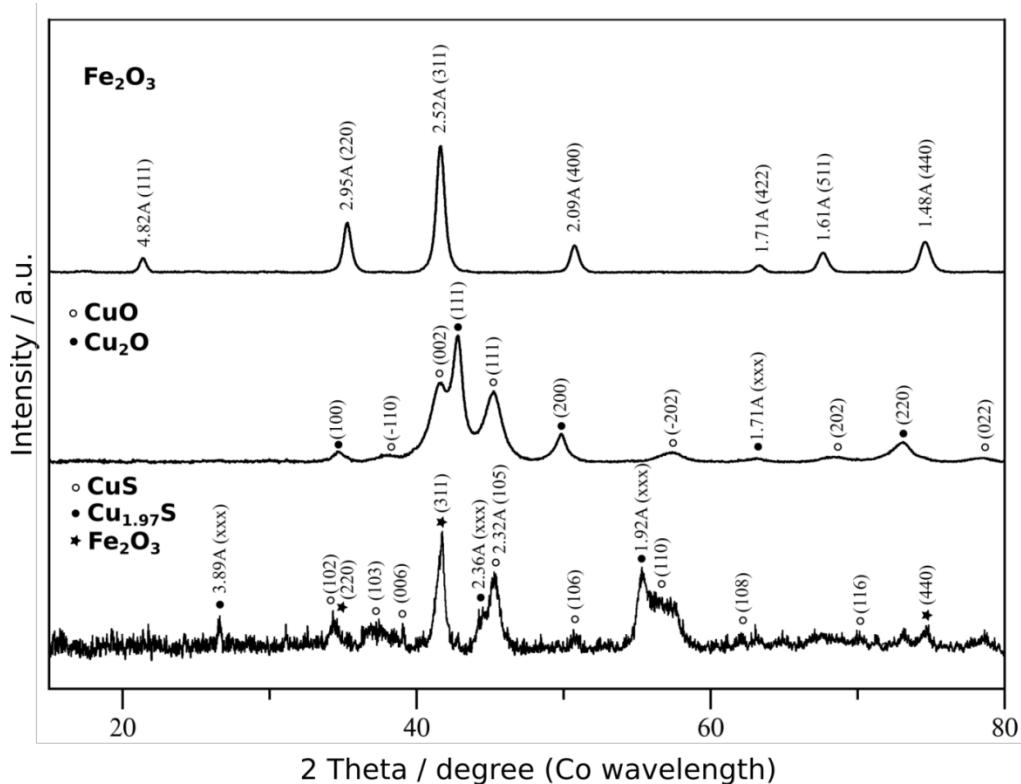
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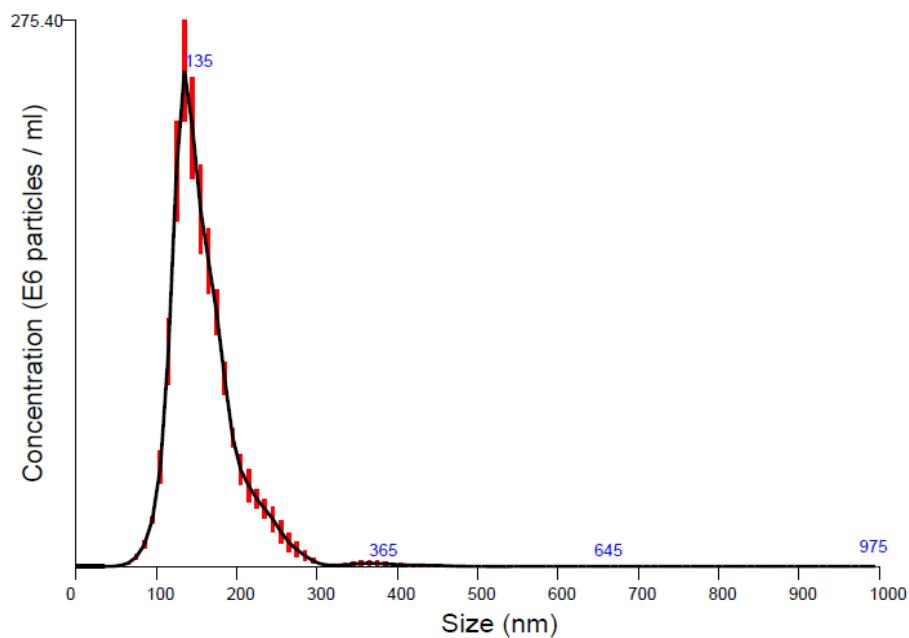
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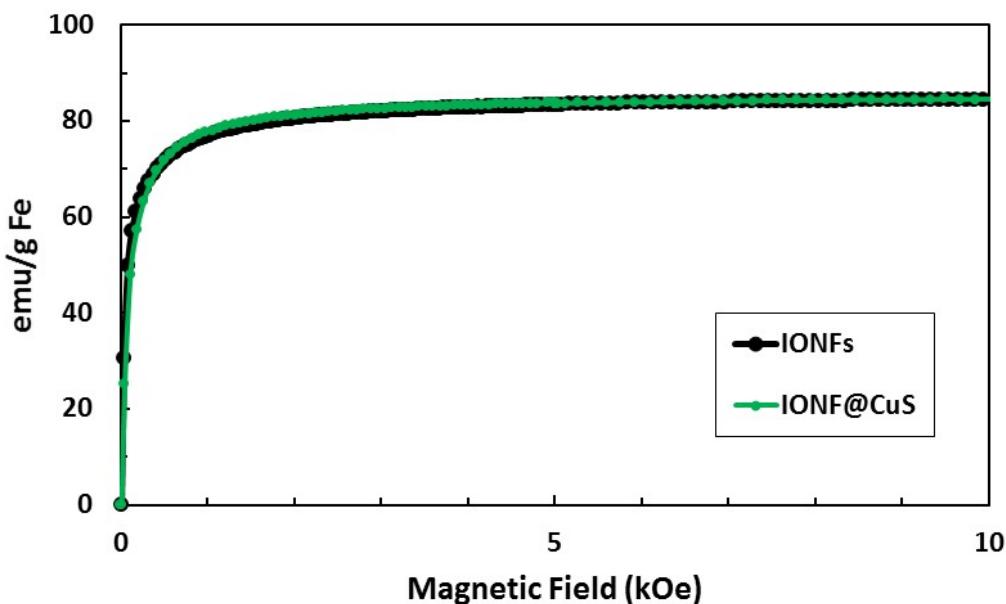
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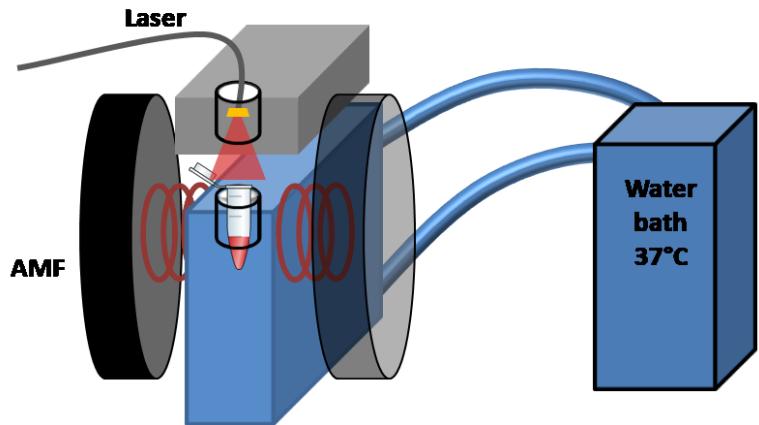
**Figure S1.** X-ray diffraction diagrams of the spiky IONF@CuS using a Co  $K\alpha$  radiation showing the presence of maghemite  $\text{Fe}_2\text{O}_3$  (PDF2 card no. 00-039-1346), tenorite  $\text{CuO}$  (PDF2 card no. 00-041-0254), cuprite  $\text{Cu}_2\text{O}$  (PDF2 card no. 01-071-4310), covellite  $\text{CuS}$  (PDF2 card no. 00-006-0464), and  $\text{Cu}_{2-x}\text{S}$  (consistent with PDF2 card of djurleite no. 01-071-1383).



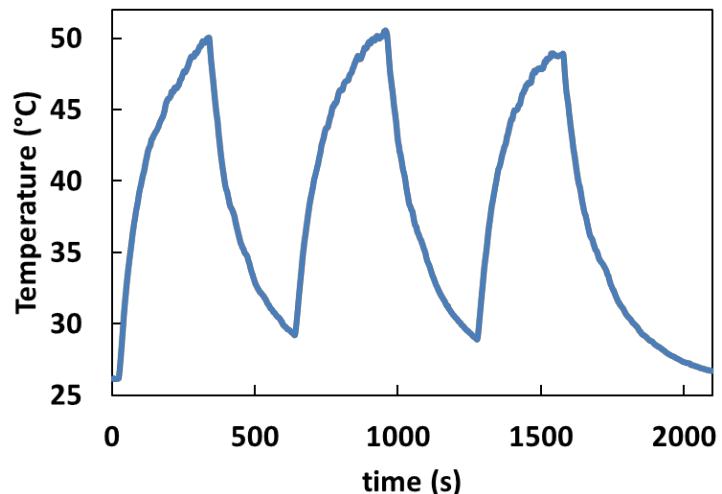
**Figure S2.** Nanoparticle Tracking Analysis (NTA) of spiky IONF@CuS in water having the main diameter peak at 135 nm and with a mean diameter of 154.6 nm.



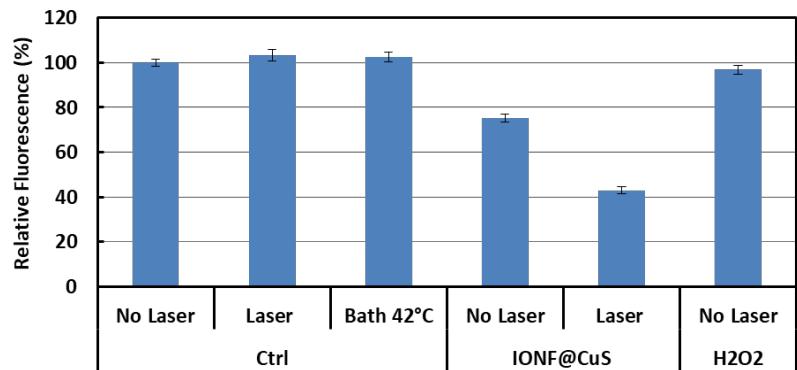
**Figure S3.** Magnetic properties of the magnetic iron oxide nanoflowers (IONFs) core alone, and after coating with the CuS shell. The shell does not impact neither the saturation magnetization ( $84 \text{ emu g}^{-1}$  of Fe) nor the initial magnetic susceptibility.



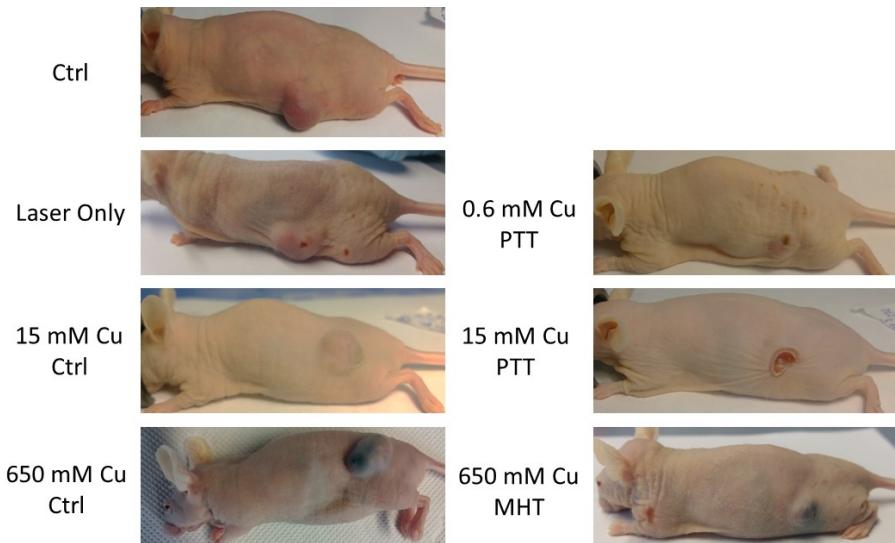
**Figure S5:** Sketch drawing of the thermostated support used to apply simultaneously AMF and Laser.



**Figure S5.** Multiples heating cycles of the IONF@CuS water dispersion at the concentration of 80 mM Cu illuminated by 1064 nm laser at  $0.3 \text{ W cm}^{-2}$  for 5 min on and 5 min off, showing the nanoparticles stability during PTT.



**Figure S6:** Selective detection of the superoxide radical ( $\cdot\text{O}_2^-$ ) in presence of IONF@CuS nanohybrids ( $[\text{Cu}] = 130 \mu\text{M}$ ) and an applied 1064 nm laser (5 min exposure at  $1 \text{ W cm}^{-2}$ ) using the specific probe 1,3-diphenylisobenzofuran (DPBF,  $\lambda_{\text{ex}} = 410 \text{ nm}$ ;  $\lambda_{\text{em}} = 477 \text{ nm}$ ).  $\text{H}_2\text{O}_2$  at the concentration of 1.8 mM have been used to prove the specificity of the analysis toward the superoxide.



**Figure S7:** Representative photographs of tumor-bearing mice 13 days after treatment.