## **Supporting Information**

Synthesis of CoSnS<sub>2</sub> hollow nanocubes with NIR-enhanced chemodynamic therapy and glutathione depletion for synergistic cancer therapy

Xuerui Zhu<sup>a,b</sup>, Zhaoyou Chu<sup>b</sup>, Benjin Chen<sup>b</sup>, Qianqian Jin<sup>a</sup>, Xuke Ma<sup>a</sup>, Juan Yang<sup>b</sup>, Yongxin Jiang<sup>b</sup>, Wanni Wang<sup>b,\*</sup>, Zhengbao Zha<sup>a,\*</sup>, Haisheng Qian<sup>b,c,\*</sup>

<sup>a</sup> School of Food and Biological Engineering, Hefei University of Technology, Hefei 230009, P. R. China.

<sup>b</sup> School of Biomedical Engineering, Research and Engineering Center of Biomedical Materials, School of Basic Medical Sciences Anhui Medical University, Hefei 230032, P. R. China.

<sup>c</sup> Anhui Provincial Institute of Translational Medicine, Anhui Medical University, Hefei, Anhui 230032, P. R. China

### **Experimental Section**

### 1. Photothermal Effect of CSS.

2.0 mL aqueous solutions of CSS NPs with different concentrations were exposed upon an 808 nm laser (2.0 W cm<sup>-2</sup>) for 10 min. Then, CSS NPs (125  $\mu$ g mL<sup>-1</sup>) aqueous solutions was exposed to different power of 808 nm laser. The photothermal stability of CSS NPs was further evaluated by repeating the laser on/off cycles for five times. The temperature change of CSS NPs solution was recorded at 10 s intervals by an IR thermal camera (Testo 865).

## 2. Animal model.

Tumor models were established by injecting 4T1 cells subcutaneously into the back of female BALB/c mice (4 and 5 weeks of age). The CSS was injected when the tumor volume reached 50 ~ 100 mm<sup>3</sup>. Animal experiments were approved by the ethics committee of Anhui Medical University (approval number: LLSC20210077). All animal-related experimental protocols were performed in accordance with the guidelines of the Association for Laboratory Animal Science and the Center for Laboratory Animal Science of Anhui Medical University.

#### 3. Characterization.

The surface morphologies, phase, fluorescence, optical properties, X-ray diffraction (XRD) and X-ray photoelectron spectra (XPS) of these products were investigated carefully according to our previous protocol or instruments. Photothermal performance, ROS, Hydroxyl radical ( $\cdot$ OH) and singlet oxygen ( $^{1}O_{2}$ ) detection were studied via our previously reported protocol. All animal experimental protocols were investigated carefully according to our previous protocol or instruments.

# **Supporting Figures**



Fig. S1 (a) TEM images of  $CoSn(OH)_6$  hollow nanocubes and (b) XRD patterns of as-obtained  $CoSn(OH)_6$ .



Fig. S2 Energy dispersive X-ray spectra of the as-prepared CSS hollow nanocubes.



Fig. S3 Zeta potential analyses of CSS dispersed in PBS and DMEM.



Fig. S4 (a) XRD patterns of as-obtained  $(CoSn)S_2$  and (b) TEM images for the single  $(CoSn)S_2$  NPs.



Fig. S5 S XPS spectra of as-obtained CSS.



Fig. S6 Thermal images of CSS solutions at various concentrations upon 808 nm laser irradiation (2 W cm<sup>-2</sup>).



**Fig. S7** GSH depletion by CSS at different concentrations characterized by the absorbance of 5,5'dithiobis-(2-nitrobenzoic acid) (DTNB).



**Fig. S8** Fenton catalytic effect of CSSs at different temperature (25°C/55°C) via OPDA and TMB probes.



Fig. S9 CLSM images of 4T1 cells after 4 h incubation with CSS at different concentration (0, 25, 50, 75  $\mu$ g mL<sup>-1</sup>).



Fig. S10 Results of different experimental groups of the scratching experiment and statistical histogram.



Fig. S11 Results of different experimental groups of plate cell clone formation experiments and statistical histograms.



**Fig. S12** CLSM images of 4T1 cells for GSH analysis stained by Thiol-Tracker<sup>TM</sup> Violet after ncubation with different groups.



Fig. S13 Flow cytometric analysis of 4T1 cells apoptosis induced by different treatments with Annexin V-FITC/PI staining.



Fig. S14 Representative mice from different treatment groups.

	Control	PBS+NIR	CSS	CSS+NIR
DAPI	200 µ <u>m</u>			
TUNEL	200 μ <u>m</u>		_	
Merge	200 µ <u>m</u>			

Fig. S15 TUNEL staining images of excised tumors of different treatments group on the 14th day.



Fig. S16 8-OH-dG staining images of excised tumors of different treatments group on the 14th

day.



**Fig. S17** Hematoxylin and eosin (H&E) stained images of major organs (heart, liver, spleen, kidney, and lung) of mice post-injection of CSS nanoparticles.



**Fig. S18** *In vivo* toxicology assays of the CSS. (a) blood panel analysis and blood biochemistry test of healthy mice after intravenous injection of CSS NPs (10 mg kg<sup>-1</sup>) at different days. (b) H&E staining images of major organs (heart, liver, spleen, kidney, lung) of the mice after injection of CSS at different time.



**Fig.S19** Ion distribution in the main organs (heart, liver, spleen, lung and kidney) on 0, 1, 7 and 14 days (a) *In vivo* distribution of tin ions (b) *In vivo* distribution of cobalt ions.

Element	Weight %	Atomic %
S K	30.24	54.55
Co K	23.20	22.77
Sn K	46.56	22.69
Total	100.00	100.00

 Table S1 Element scale in CSS by energy dispersive X-ray spectra.