

## Supplementary materials

### *Determination of the quantum yield of Ag<sub>2</sub>S-CS-SNO nanospheres*

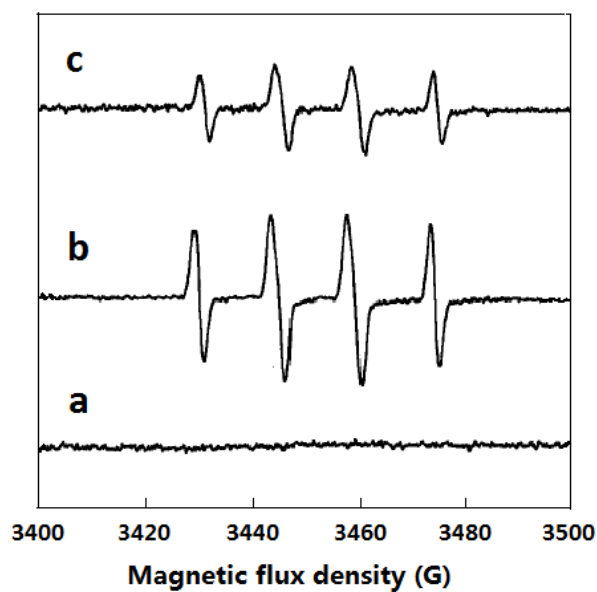
The quantum yield of the as-prepared Ag<sub>2</sub>S-CS-SNO nanospheres were measured using indocyanine green (ICG) as a reference (QY = 13% in DMSO). The absorption spectra of Ag<sub>2</sub>S-CS-SNO nanospheres and ICG solutions at different concentrations were recorded. Then the fluorescence spectra of these samples were recorded under the same conditions. The fluorescence quantum yield was calculated according to the following equation:

$$\phi_{NS} = \phi_{ICG} * \left( \frac{F_{NS}}{F_{ICG}} \right) * \left( \frac{A_{ICG}}{A_{NS}} \right) * \left( \frac{n_{NS}}{n_{ICG}} \right)^2 \quad (1)$$

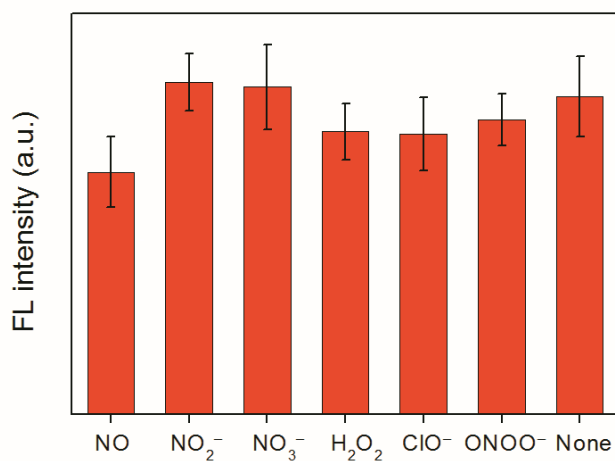
Where  $\phi_{NS}$ ,  $F_{NS}$ ,  $A_{NS}$  and  $n_{NS}$  are the quantum yield, integrated fluorescence intensity, integrated absorption and refractive index of the solvent for the Ag<sub>2</sub>S-CS-SNO nanospheres. The parameters with a subscript of ICG are corresponding quantities of the solvent for ICG.

### *ESR Measurements*

Electron spin resonance (ESR) spectra were recorded at room temperature on an ESP 300 EPR spectrometer (Bruker, Germany) at 37 °C with the microwave frequency of 9.22 GHz and the microwave power of 5 mW. The center magnetic flux density was set at 3450 Gauss (G) and the modulation amplitude was 1 G. The sweep range was 100 G and the sweep rate was 50 G/min. Samples were prepared in a nitrogen glove box and taken up in a capillary, which was sealed at both ends. The capillary was placed in a quartz tube and then placed inside the ESR cavity. The EPR spectra are shown in Fig. 1s. These results confirm that the Ag<sub>2</sub>S-CS-SNO nanospheres are capable of releasing NO in PBS buffer under irradiation.

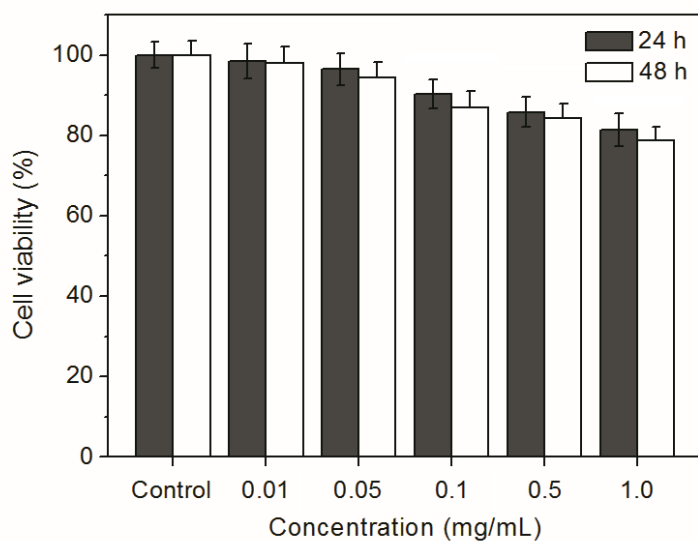


**Fig. S1.** ESR spectra of  $\text{Ag}_2\text{S-CS-SNO}$  nanospheres unirradiated (a), irradiated by 488 nm laser for 10 min (b) and kept in the dark for 10 min after irradiation (c) in a PBS solution (0.2 M, pH=7.4) at 37 °C.



**Fig. S2.** Influences of molecules and anions on the fluorescence emission from  $\text{Ag}_2\text{S-CS-SNO}$  nanospheres in PBS buffer (pH = 7.4, 37 °C). Measurements were performed in a 0.2 M PBS

buffer (pH = 7.4, 37 °C). The concentration of Ag<sub>2</sub>S-CS-SNO nanospheres and each species were 1 μM and 100 μM, respectively. The result of a blank experiment was the control. The Ag<sub>2</sub>S-CS-SNO nanospheres showed largest fluorescence response to NO among the species, ascribed to the fluorescence quenching effect of NO on the Ag<sub>2</sub>S-CS-SNO nanospheres.



**Fig. S3.** Viability of L929 cells incubated with different concentrations of Ag<sub>2</sub>S QDs.