

## Electronic Supplementary Information

*for*

### **H<sub>2</sub>S bubbles-assisted synthesis of hollow Cu<sub>2-x</sub>Se<sub>y</sub>S<sub>1-y</sub>/reduced graphene oxide nanocomposites with tunable compositions and localized surface plasmon resonance**

Wen Long Li,<sup>‡a</sup> Hong Yan Zou,<sup>‡b</sup> Jing Lan,<sup>b</sup> Qiang Wang,<sup>b</sup> Yuan Fang

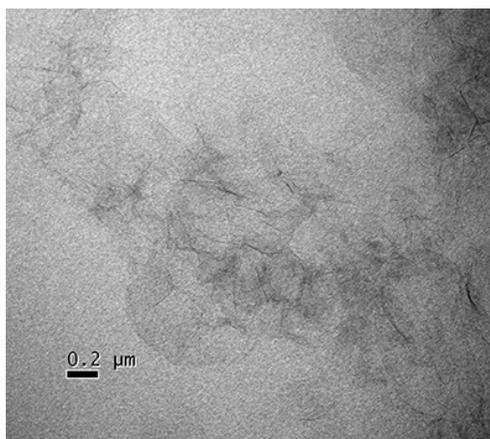
Li<sup>a</sup> and Cheng Zhi Huang<sup>\*ab</sup>

<sup>a</sup> Key Laboratory of Luminescent and Real-Time Analytical Chemistry (Southwest University), Ministry of Education, College of Chemistry and Chemical Engineering, Southwest University, Chongqing 400715, China.

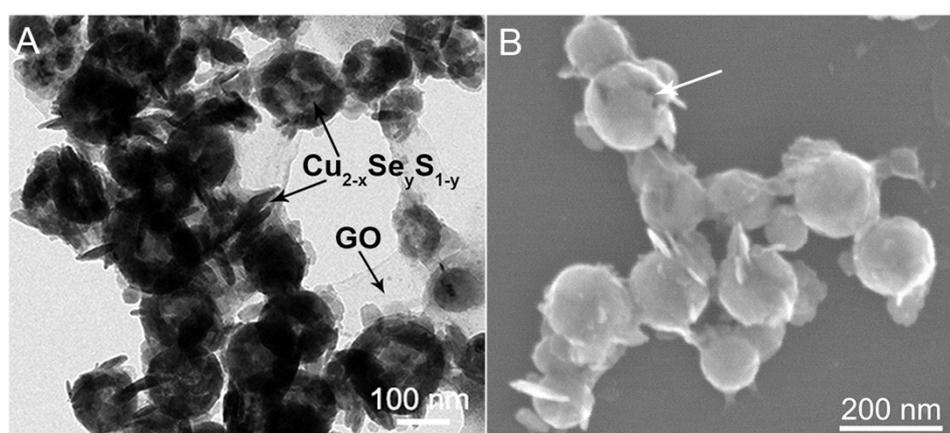
<sup>b</sup> Chongqing Key Laboratory of Biomedical Analysis (Southwest University), Chongqing Science & Technology Commission, College of Pharmaceutical Science, Southwest University, Chongqing 400715, China.

\* Corresponding Author. *E-mail:* [chengzhi@swu.edu.cn](mailto:chengzhi@swu.edu.cn); *Tel:* +86-23-68254659; *Fax:* +86-23-68367257

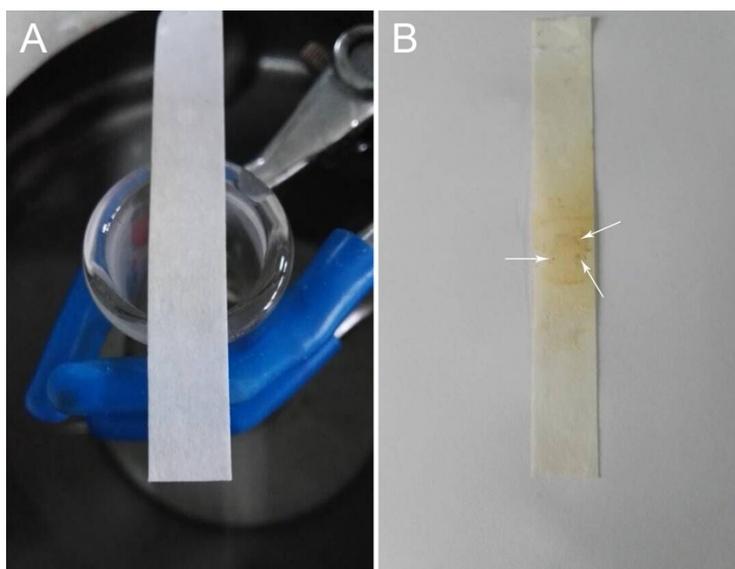
<sup>‡</sup> These people contributed equally to this work.



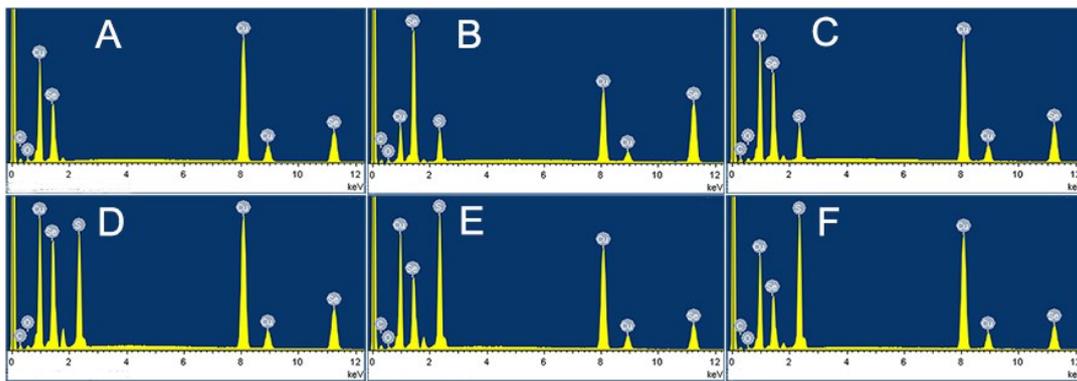
**Fig. S1** TEM image of GO sheets.



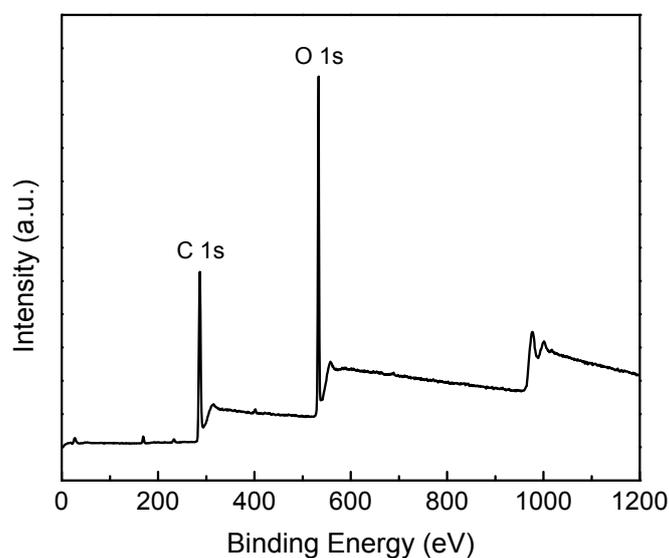
**Fig. S2** (A) TEM image and (B) SEM image of Cu<sub>2-x</sub>Se<sub>y</sub>S<sub>1-y</sub>/rGO nanocomposites prepared with a reactant S/Se molar ratio of 3:1. The white arrow suggests the hollow opening.



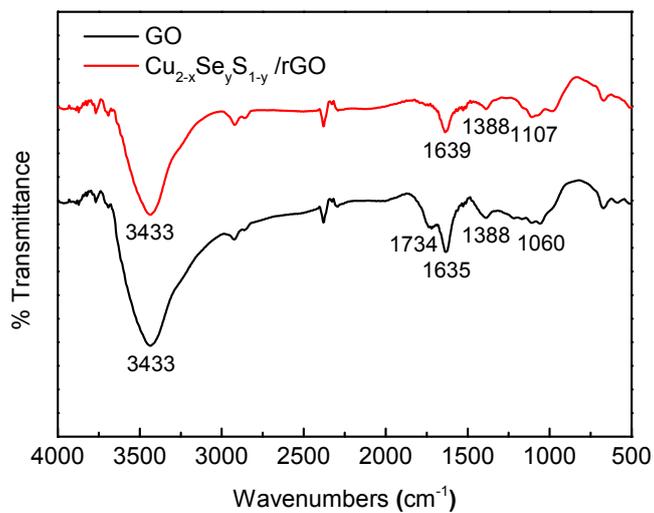
**Fig. S3** (A) Detection of H<sub>2</sub>S gas bubble by wet lead acetate test paper on the bottle. (B) The color of lead acetate test paper turns yellow-black, which confirms the release of H<sub>2</sub>S gas bubble from the solvent. The white arrows suggest the PbS black precipitate.



**Fig. S4** EDX spectra of  $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}/\text{rGO}$  nanocomposites synthesized with various reactant S/Se molar ratios. (A) 0:1; (B) 1:1; (C) 2:1; (D) 3:1; (E) 4:1; (F) 5:1.

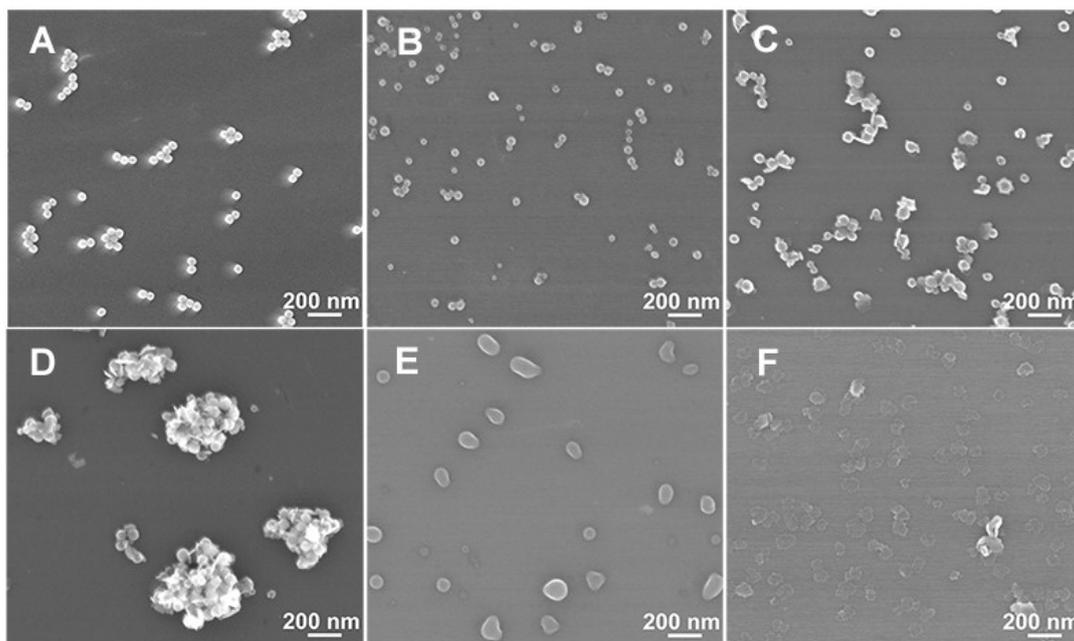


**Fig. S5** The survey XPS spectrum of GO.

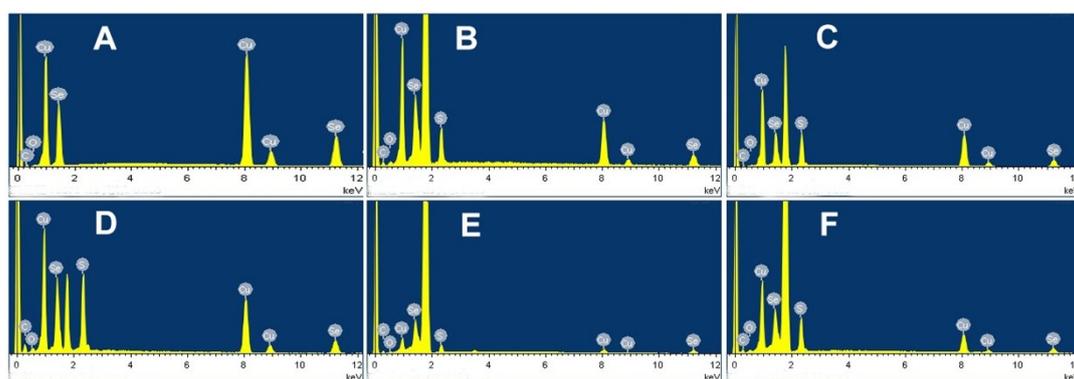


**Fig. S6** FTIR spectra of GO and  $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}/\text{rGO}$  nanocomposites (synthesized with a reactant S/Se molar ratio of 3:1). The oxygen-containing functional groups of GO afforded bands at 3433,

1734, 1388 and 1060  $\text{cm}^{-1}$ , which were associated with the -OH vibration, the C=O stretching vibration, the C-O-H deformation and the alkoxy C-O stretching vibration peaks, respectively. The peak at 1631  $\text{cm}^{-1}$  (aromatic C=C) could be assigned to the skeletal vibrations of unoxidized graphite domains. In the spectrum of  $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}/\text{rGO}$  nanocomposites, the difference from that of GO was evidenced by the entire disappearance of the C=O stretching vibration peak and the dramatic decrease of other oxygen-containing functional group peaks, which confirmed that GO had been reduced to rGO.



**Fig. S7** SEM images of  $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}$  NPs synthesized with PSS as capping agent with different S/Se molar ratios, while the other experiment conditions were kept same as those used for preparing  $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}/\text{rGO}$  nanocomposites. (A) 0:1; (B) 1:1; (C) 2:1; (D) 3:1; (E) 4:1; (F) 5:1.



**Fig. S8** EDX spectra of  $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}$  NPs synthesized with PSS as capping agent with different S/Se molar ratios. (A) 0:1; (B) 1:1; (C) 2:1; (D) 3:1; (E) 4:1; (F) 5:1.

**Table S1** EDX analysis of  $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}$ @PSS NPs prepared with reactants of various S/Se ratios.

S/Se molar ratios in reactants	EDX analysis (mean atomic percent)			Compositions of $\text{Cu}_{2-x}\text{Se}_y\text{S}_{1-y}$ @PSS NPs
	Cu	Se	S	
1:1	30.32	12.34	10.10	$\text{Cu}_{1.35}\text{Se}_{0.55}\text{S}_{0.45}$
2:1	40.03	13.34	17.19	$\text{Cu}_{1.31}\text{Se}_{0.44}\text{S}_{0.56}$
3:1	23.11	8.94	12.93	$\text{Cu}_{1.06}\text{Se}_{0.40}\text{S}_{0.60}$
4:1	11.73	15.67	9.13	$\text{Cu}_{0.47}\text{Se}_{0.63}\text{S}_{0.37}$
5:1	17.58	9.01	12.43	$\text{Cu}_{0.82}\text{Se}_{0.42}\text{S}_{0.58}$