## **Supporting Information**

## Ultra Small Gold Nanoclusters Supported on Two-dimension Bismuth Selenium Nanosheets for Synergistic Photothermal and Photodynamic Tumor Therapy

Chenxi Li †<sup>a,b</sup>, Xueyang Fang †<sup>\*b</sup>, Qingdong Zeng <sup>a,b</sup>, Li Zeng <sup>\*b</sup>, Bin Zhang <sup>\*b</sup>, Guohui Nie <sup>\*b</sup>

a Graduate Collaborative Training base of Shenzhen Second People's Hospital, Heng Yang Medical School, University of South China, Hengyang, Hunan, 421001, China

b Shenzhen Key Laboratory of Nanozymes and Translational Cancer Research, Institute of Translational Medicine Department of Otolaryngology Shenzhen Second People's Hospital, the First Affiliated Hospital of Shenzhen University, Health Science Center, Shenzhen 518035, China †Chenxi Li and Xueyang Fang contributed equally to this work.

\*Corresponding authors: Bin Zhang, E-mail: binzhang@email.szu.edu.cn; Guohui Nie, E-mail: nghui@21cn.com; Li Zeng, E-mail: zengli0298@163.com; Xueyang Fang, E-mail: FANGXueyang0210@163.com



Fig. S2 AFM images and corresponding height analysis of AuNC@Bi<sub>2</sub>Se<sub>3</sub>



Fig. S1 The morphology of AuNC@Bi<sub>2</sub>Se<sub>3</sub> nanocomposites across different batches.



Fig. S3 XRD patterns and energy-dispersive X-ray spectroscopy (EDX) of AuNC@Bi<sub>2</sub>Se<sub>3</sub>.



Fig. S6 The photothermal heating curve of AuNC@Bi<sub>2</sub>Se<sub>3</sub> and Bi<sub>2</sub>Se<sub>3</sub> dispersions at 50 ppm under 808 nm and 660 nm laser irradiation  $(1W/cm^2)$ , respectively.



Fig. S7 ESR spectra of AuNC and AuNC + Laser group to detect the production of  $O_2^-$  production.



Fig. S8 Temperature curves of 4T1 tumor bearing mice after injection of PBS and AuNC@Bi<sub>2</sub>Se<sub>3</sub> respectively.



Fig. S9. H&E staining of major organs including Heart, Liver, Lung, Spleen and Kidney after different treatments.



Fig. S10. Serum biochemical indices, including ALT, AST, BUN, TP, ALB, ALP, TBIL and CREA of 4T1 tumor-bearing mice after the treatment process.