

## Electronic Supplementary Information

### Enhanced radiosensitization of ternary $\text{Cu}_3\text{BiSe}_3$ nanoparticles by photo-induced hyperthermia at the second near-infrared biological window

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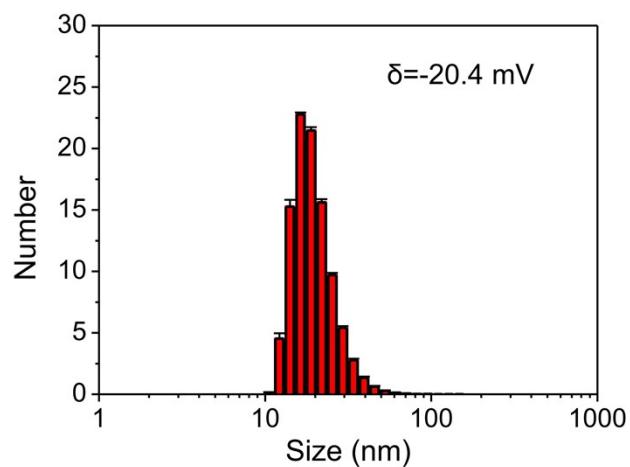
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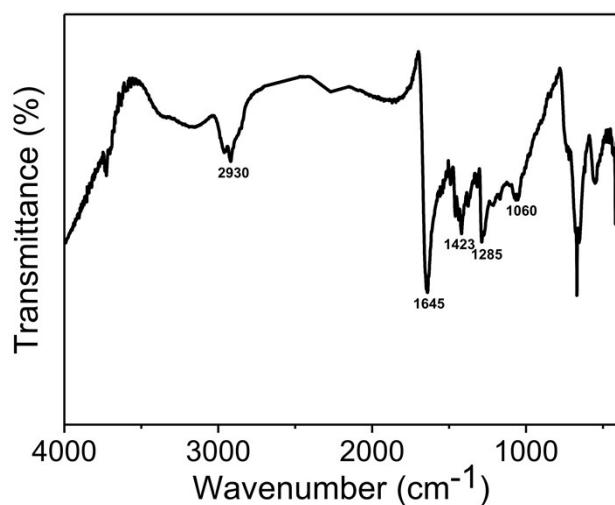
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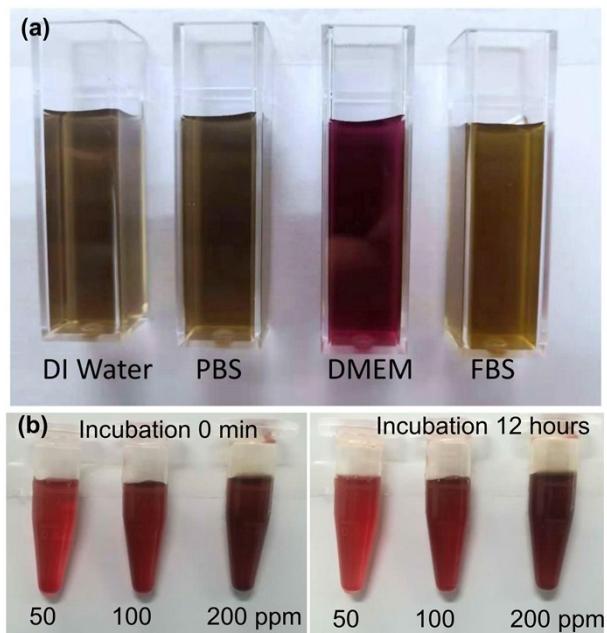
<sup>‡</sup> These authors contributed equally.



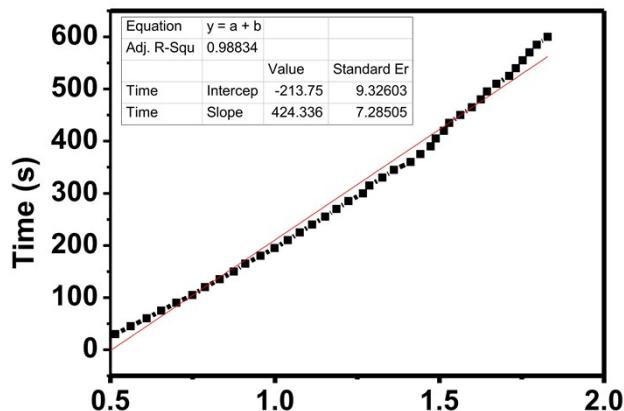
**Fig. S1** Size distribution and Zeta potential of PVP-Cu<sub>3</sub>BiSe<sub>3</sub> NPs.



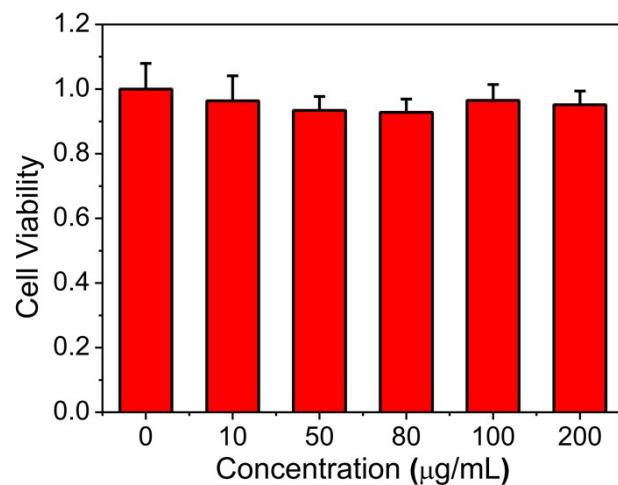
**Fig. S2** FT-IR spectrum of PVP-Cu<sub>3</sub>BiSe<sub>3</sub> NPs.



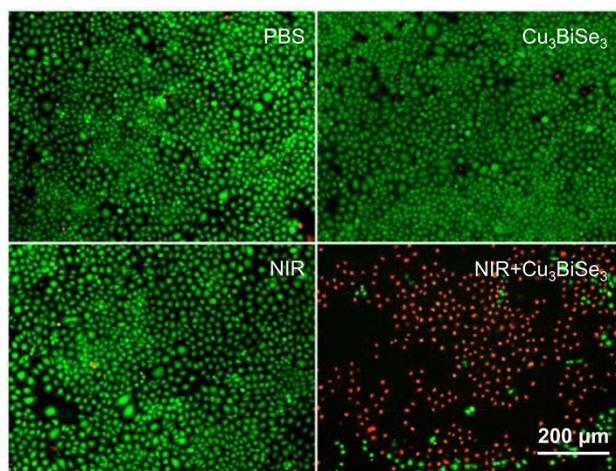
**Fig. S3** (a) Photographs of the dispersion of PVP- $\text{Cu}_3\text{BiSe}_3$  NPs in deionized (DI) water, PBS, DMEM, and FBS (200  $\mu\text{g}/\text{mL}$ ). (b) Photographs of the dispersion of PVP- $\text{Cu}_3\text{BiSe}_3$  NPs in the blood.



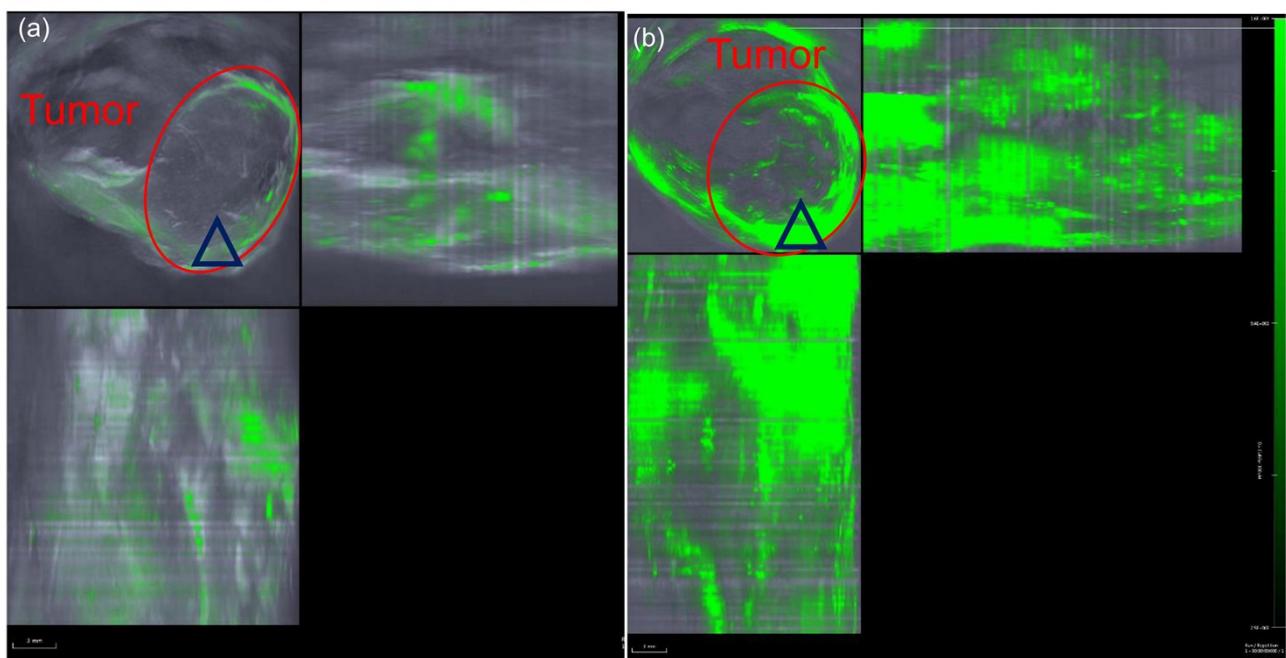
**Fig. S4** Plot of cooling time versus negative natural logarithm of the temperature driving force which is obtained from the cooling stage. Time constant for heat transfer from the system is determined to be  $\tau_s = 424.336$



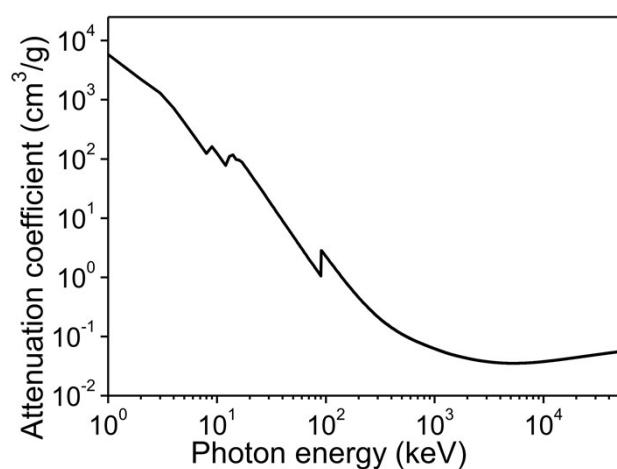
**Fig. S5** Cell viability of HeLa cells after cultured with various concentrations PVP-Cu<sub>3</sub>BiSe<sub>3</sub> NPs for 24 h.



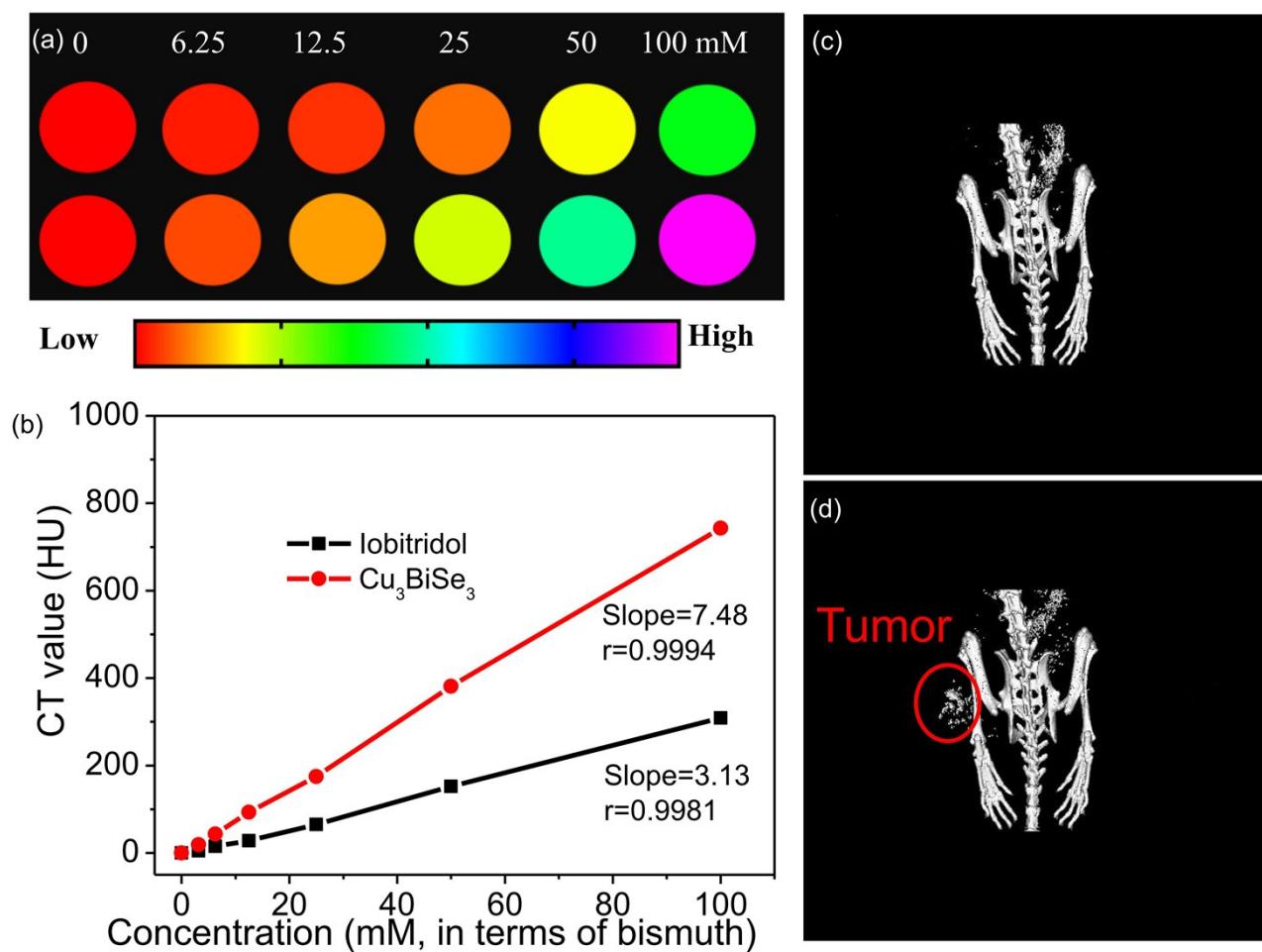
**Fig. S6** *In vitro* photothermal effect. Live-dead staining of HeLa cells. HeLa cells were incubated with 100  $\mu\text{g}/\text{mL}$  of PVP-Cu<sub>3</sub>BiSe<sub>3</sub> NPs for 24 h and irradiated for 10 min using a 1064 nm laser with 0.75  $\text{W}/\text{cm}^2$  of power densities.



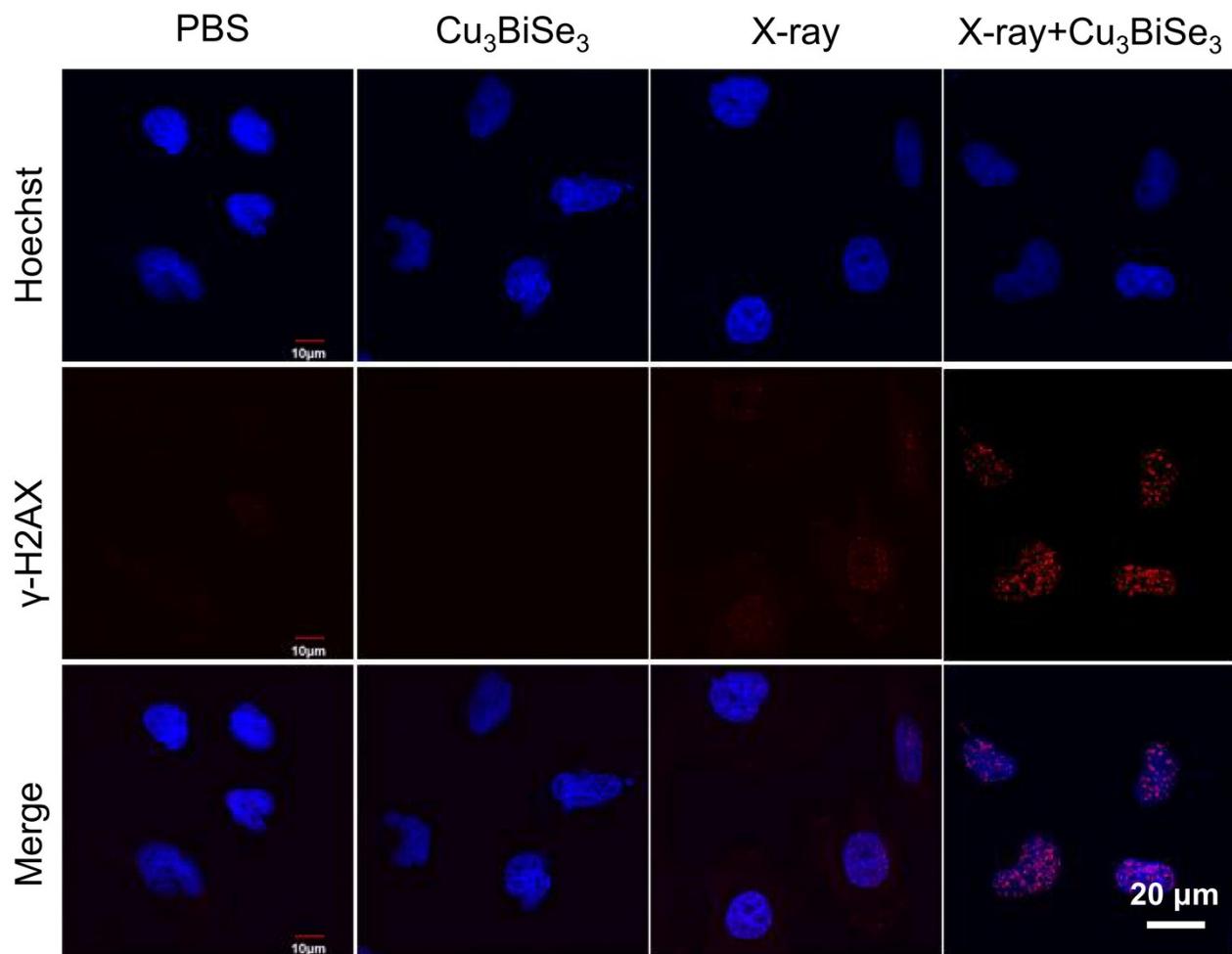
**Fig. S7** (a-b) *In vivo* different dimensions of PAT imaging of a tumor-bearing mouse before (a) and after (b) Injection with PVP-Cu<sub>3</sub>BiSe<sub>3</sub> NPs. (laser light range: 750-850 nm for *in vivo* imaging)



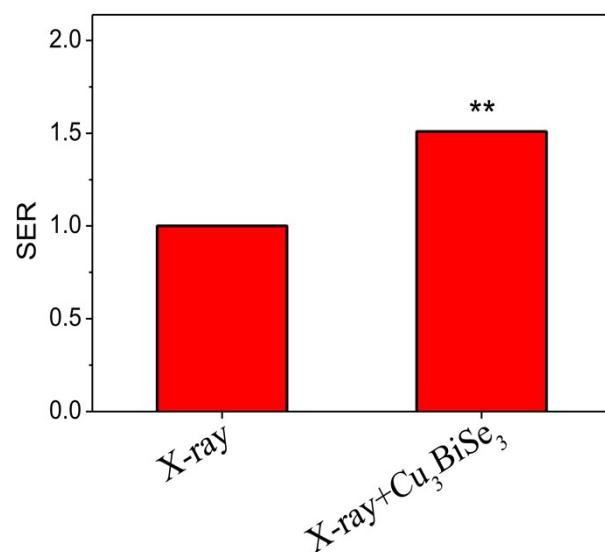
**Fig. S8** The X-ray absorption coefficient of Cu<sub>3</sub>BiSe<sub>3</sub> NPs versus the X-ray energy was calculated by XMuDat computer program.



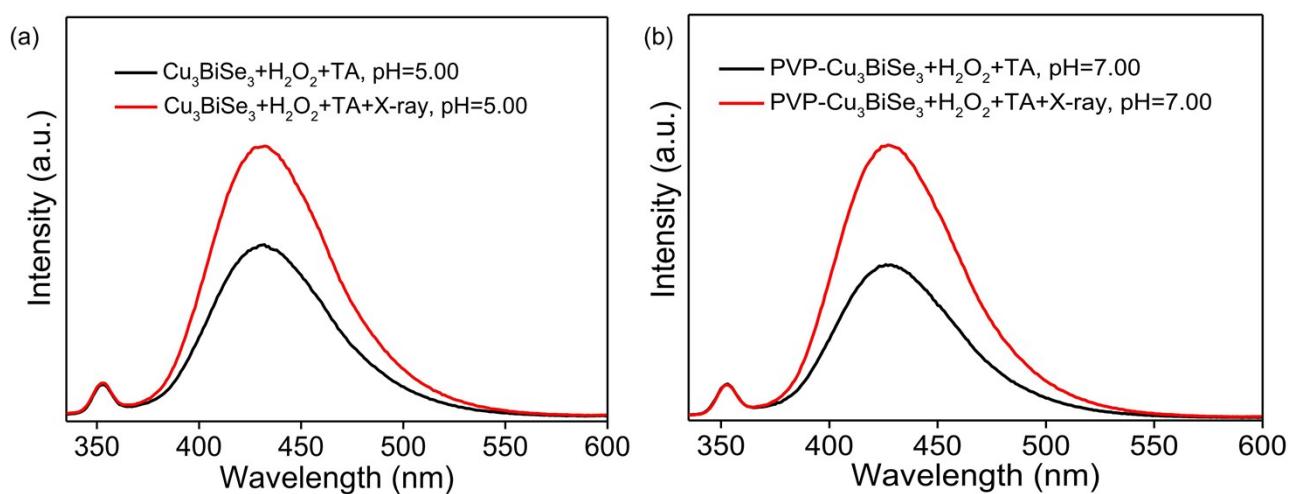
**Fig. S9** (a) CT phantom images of PVP- $\text{Cu}_3\text{BiSe}_3$  NPs (upper) and iopromide (bottom) with different concentrations. (b) Plot of HU values of PVP- $\text{Cu}_3\text{BiSe}_3$  NPs and iopromide versus the sample concentrations. (c-d) CT image of mice before injection of PVP- $\text{Cu}_3\text{BiSe}_3$  NPs (c) and after intratumoral injection of PVP- $\text{Cu}_3\text{BiSe}_3$  NPs for 3 h (d).



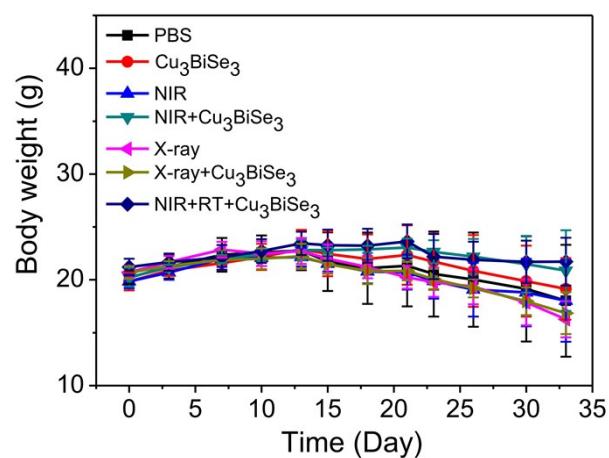
**Fig. S10** Representative fluorescence images of DNA fragmentation and nuclear condensation induced by PVP- $\text{Cu}_3\text{BiSe}_3$  NPs (100 ppm, 2 mL) or/and X-ray radiation (6 Gy), stained with DAPI and  $\gamma$ -H2AX for nuclear visualization and DNA fragmentation, respectively.



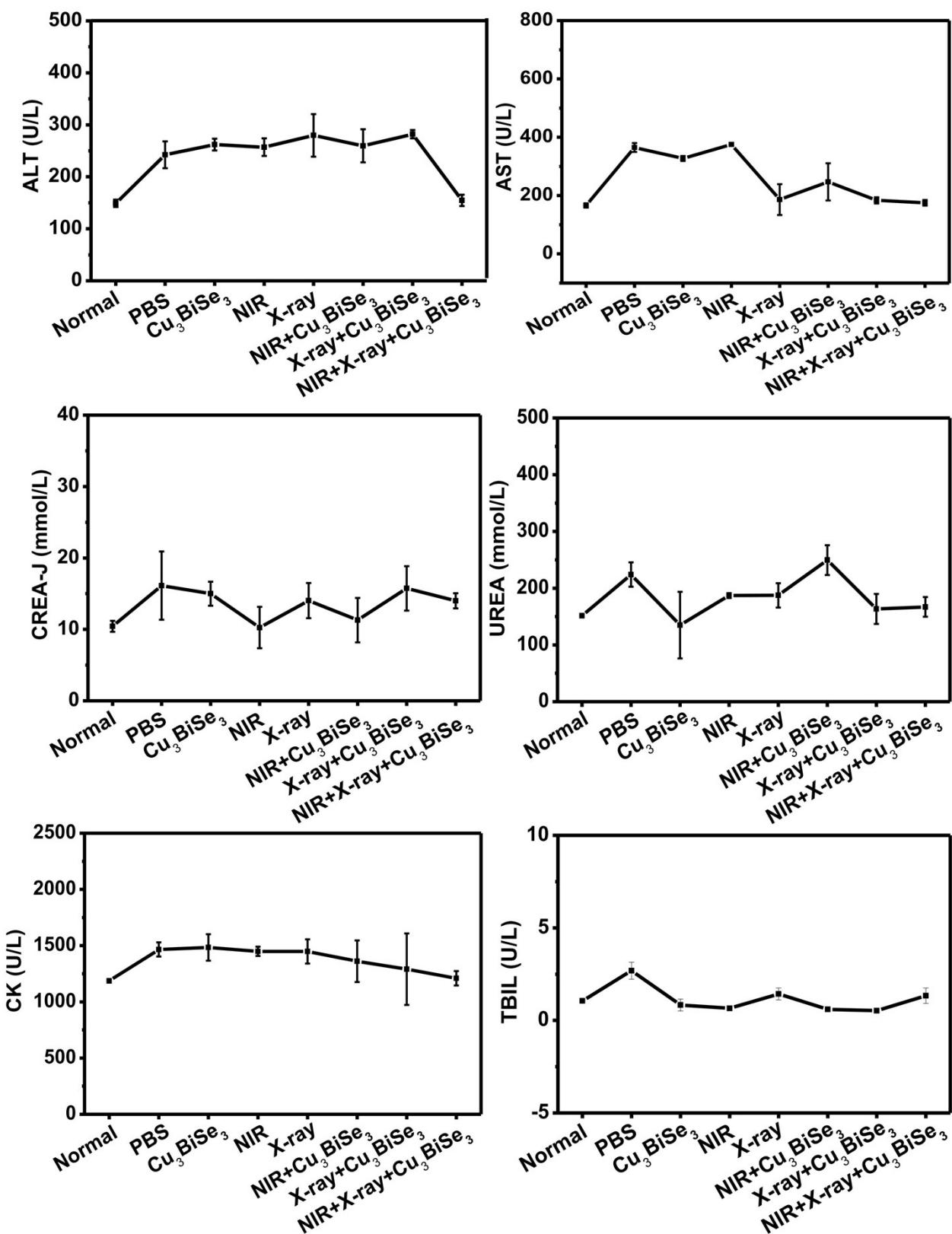
**Fig. S11** Produce 63% biological effects of the sensitizer enhancement ratio (SER) of PVP-Cu<sub>3</sub>BiSe<sub>3</sub> NPs. P values were calculated by *t* test: \*\* P<0.01.



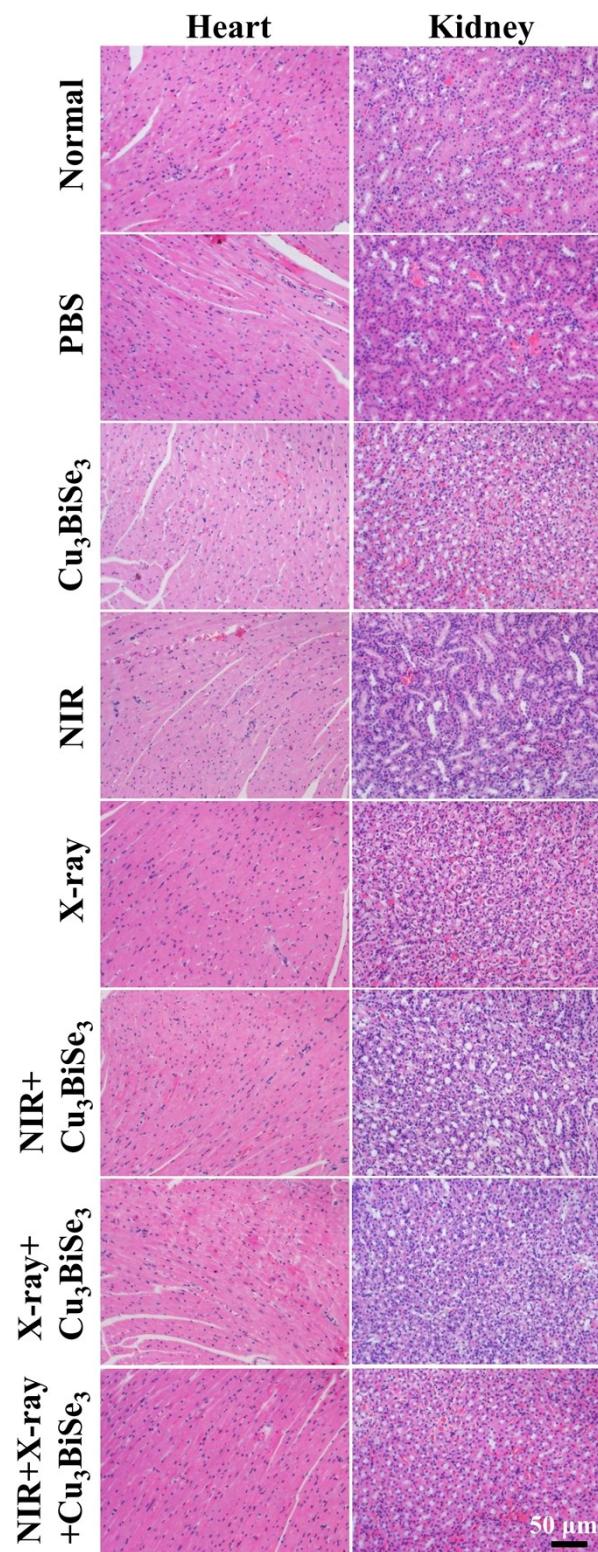
**Fig. S12** Determination of the formation of HO<sup>•</sup> using TA as a fluorescent probe. Fluorescence intensity with or without X-ray irradiation in the presence of PVP- $\text{Cu}_3\text{BiSe}_3$  NPs under pH=5.00 (a) and pH=7.00 (b).



**Fig. S13** The weight of HeLa tumor-bearing mice in our observation period. Error bars correspond to mean  $\pm$  standard deviations



**Fig S14.** Blood biochemistry analysis of the mice treated with PVP- $\text{Cu}_3\text{BiSe}_3$  NPs. The result exhibit mean and standard deviation of alanine aminotransferase (ALT), aspartate aminotransferase (AST), creatinine (CREA-J), UREA, creatine kinase (CK). All of the results indicated the contain PVP- $\text{Cu}_3\text{BiSe}_3$  NPs groups could decrease to different degree dysfunction of hepatic and renal dysfunction.



**Fig S15.** Hematoxylin and Eosin stained tissue sections from the mice to monitor the histological changes in heart and kidney of the mice 33 days after with different administration.

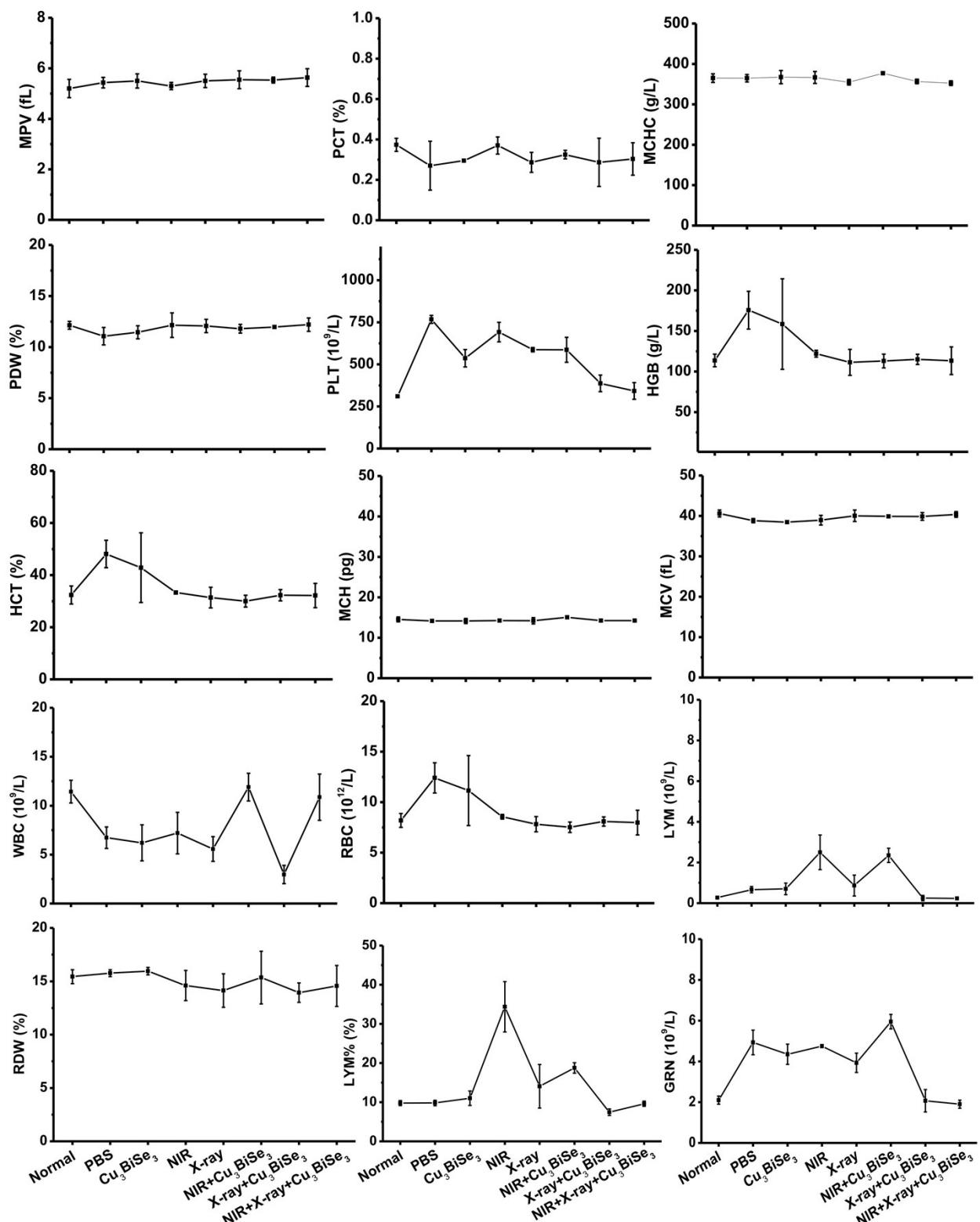


Fig S16. Blood routine analysis of the mice treated with PVP- $\text{Cu}_3\text{BiSe}_3$  NPs.