Supporting information of

## Marriage of PEG-decorated black phosphorus nanosheets and immunoadjuvant for photoimmunotherapy of melanoma

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Figure S1. TEM image of BP NS.



Figure S2. AFM image of BP NSs.



Figure S3. XPS spectrum of BP NPs (a) and the detailed spectra of the P 2p peak (B).

The X-ray photoelectron spectroscopy (XPS) of BP displayed that the characteristic 2p3/2 and 2p1/2 doublets appeared at 129.5 and 130.3 eV, respectively. The subband at 132.9 eV was proabably ascribed to the oxidized phosphorus (i.e., POx).<sup>1</sup>

Three prominent peaks appeared at 361 cm<sup>-1</sup> ( ${}^{A_{g}^{1}}$ ), 438 cm<sup>-1</sup> ( ${}^{B_{2g}}$ ) and 465 cm<sup>-1</sup> ( ${}^{A_{g}^{2}}$ ). Compared to bulk BP, the typical peaks of BP nanosheets are slightly redshifted, suggesting the formation of ultrasmall BP nanosheets<sup>4</sup>.



Figure S4. DLS results of BP NSs and BP-PEG in FBS.



Figure S5. Zeta potential of BP NSs and BP-PEG.



Figure S6. FTIR spectra of BP, BP-PEG, and PEG-NH<sub>2</sub>.



Figure S7. Infrared thermal images of BP-PEG suspensions upon treatment of 808 nm laser for 10 min. The concentrations of BP-PEG were 2.5  $\mu$ g/mL (a), 5  $\mu$ g/mL (b), 10  $\mu$ g/mL (c), 20  $\mu$ g/mL (d), and 40  $\mu$ g/mL (e).



Figure S8. Photothermal curves of BP NSs at varying concentrations.



Figure S9. Infrared thermal images of tumor-bearing mice after irradiation of 808 nm laser for 3

min at 4 h post injection of saline (a) and BP-PEG (b).



Figure S10. H&E stained organ slices of mice in different groups on day 11.

## References

1. Zhang, L.; Ding, L.-X.; Chen, G.-F.; Yang, X.; Wang, H., Ammonia Synthesis Under Ambient Conditions: Selective Electroreduction of Dinitrogen to Ammonia on Black Phosphorus Nanosheets. *Angewandte Chemie-International Edition* **2019**, *58* (9), 2612-2616.