Supporting Information

**In-situ** assembled titanium carbide-based heterojunctions for synergistic enhancement of NIR-II photothermal/photodynamic therapy against breast cancer

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Fig. S1. XPS spectrum Ti$_3$C$_2$/TiO$_2$ in Ti 2p region.

Fig. S2. The morphology of TiO$_2$ nanoparticles.

Fig. S3. FITR spectra PVP, Ti$_3$C$_2$/TiO$_2$ and Ti$_3$C$_2$/TiO$_2$-PVP HJs.
Fig. S4. Mass extinction coefficient of Ti$_3$C$_2$/TiO$_2$-PVP HJs at 1064 nm.

Fig. S5. Fluorescence spectra of TiO$_2$-PVP and Ti$_3$C$_2$/TiO$_2$-PVP HJs with the same titanium concentration.

Fig. S6. Calculation of the photothermal-conversion efficiency at 1064 nm (NIR-II). Orange and blue line: photothermal effect of an aqueous dispersion of TiO$_2$/Ti$_3$C$_2$-PVP
under the irradiation with NIR-II laser for certain periods, and then the laser was switched off. Purple line: time constant ($\tau_s$) for the heat transfer from the system determined by applying the linear time data from the cooling period.

**Fig. S7.** Time-dependent degradation of DPBF caused by $^1$O$_2$ generated by TiO$_2$-PVP (A) and Ti$_3$C$_2$-PVP (B), respectively, under laser irradiation (660 nm, 1 W/cm$^2$, 10 min).

**Fig. S8.** Cell apoptosis of 4T1 cells after incubated with 0 and 200 ppm concentrations of Ti$_3$C$_2$/TiO$_2$-PVP HJs with 1064 nm irradiation. Note: 1: Control; 2: Only 660 nm; 3: Only 1064 nm; 4: Ti$_3$C$_2$/TiO$_2$-PVP+660; 5: Ti$_3$C$_2$/TiO$_2$-PVP+1064 nm; 6: Ti$_3$C$_2$/TiO$_2$-PVP+660 nm+1064 nm.
Fig. S9. In vivo Ti bio-distributions after injecting Ti$_3$C$_2$/TiO$_2$-PVP into female tumor-bearing nude mice for 4 h, 12 h. Data are expressed mean ± SD (n=3).

Fig. S10. H&E staining of heart, liver, spleen, lung and kidney from different groups (scale bar = 50 μm). Note: G0: control group (treated only with PBS), G1:1064 nm laser group (only exposed to 1064 nm laser irradiation), G2: 660 nm laser group (only exposed to 660 nm laser irradiation), G3: Ti$_3$C$_2$/TiO$_2$-PVP HJs group (only intravenously injected with Ti$_3$C$_2$/TiO$_2$-PVP HJs), G4: Ti$_3$C$_2$/TiO$_2$-PVP HJs + 660 nm laser group, G5: Ti$_3$C$_2$/TiO$_2$-PVP HJs + 1064 nm laser group, and G6: Ti$_3$C$_2$/TiO$_2$-PVP HJs + 660 and 1064 nm laser group.