Supplementary Information

Preparation of TiH$_{1.924}$ Nanodots by Liquid-phase Exfoliation for Enhanced Sonodynamic Cancer Therapy

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Supplementary Figure 1. (A-D) TEM images (A-C) and XRD spectra (D) of TiH$_{1.924}$ nanodots produced by liquid-phase exfoliation in NMP after sonication for various periods of time (5, 10, and 20 min). A representative image of three biological replicates from each group is shown in (A-C).
**Supplementary Figure 2.** EDS spectrum of as-made TiH$_{1.924}$ nanodots.

**Supplementary Figure 3.** The full XPS spectrum of TiH$_{1.924}$ nanodots.
Supplementary Figure 4. TEM images and XRD spectra of the commercial ZrH₂ (A), CaH₂ (B), and HfH₁.₉₈₃ (C) powders. A representative image of three biological replicates from each group is shown in (A-C).

Supplementary Figure 5. (A&B) Time-dependent oxidation of DPBF by TiH₁.₉₂₄ along (A) and the US along (B). (C) Comparison of DPBF oxidation by TiH₁.₉₂₄ only, US only, and TiH₁.₉₂₄ plus US treatment.
Supplementary Figure 6. Time-dependent oxidation of DPBF by US-activated commercial TiH$_{1.924}$ (A) and TiO$_2$ (B).

Supplementary Figure 7. (A-E) UV-VIS spectra to time-dependent oxidation of DPBF by US-activated TiH$_{1.924}$ with the different exfoliated degrees (exfoliated time: 0, 5, 10, 15, and 20 min). (F) Comparison of DPBF oxidation by TiH$_{1.924}$ produced by various exfoliated time periods.
**Supplementary Figure 8.** Quantitative analysis of $^1\text{O}_2$ generation for the two groups based on data in Figure 3D.

**Supplementary Figure 9.** (A) ESR spectra demonstrating ROS ($\bullet$OH) generation for TiH$_{1.924}$ and TiO$_2$ under US irradiation for 1 min. (B) Quantitative analysis of $\bullet$OH generation for these two groups as indicated.
Supplementary Figure 10. Mass extinction coefficient of TiH$_{1.924}$ nanodots at 1064 nm (NIR-II).

Supplementary Figure 11. Laser power-dependent photothermal heating curves of TiH$_{1.924}$ nanodots (0.5, 0.75, 1.0, 1.25, and 1.5 W·cm$^{-2}$).

Supplementary Figure 12. Time constant ($\tau_s$) for the heat transfer from the system determined by applying the linear time data from the cooling period.
**Supplementary Figure 13.** (A) The photographs of TiH$_{1.924}$ and TiH$_{1.924}$-PVP dispersed in the H$_2$O and PBS. (B) Fourier transforms infrared spectrometry (FTIR) spectra of TiH$_{1.924}$ and TiH$_{1.924}$-PVP samples. (C) Thermogravimetric analysis (TGA) of the obtained TiH$_{1.924}$ before and after surface modification. (D) The photographs of TiH$_{1.924}$-PVP in different buffers including H$_2$O, PBS, and RPMI 1640 cell culture medium for 1, 3, and 7 days.

**Supplementary Figure 14.** UV-vis-NIR absorbance spectra of TiH$_{1.924}$ nanodots before and after PVP modification. Insert is the photograph of TiH$_{1.924}$-PVP. The insert is the photograph of TiH$_{1.924}$-PVP solution.
Supplementary Figure 15. (A&B) Time-dependent oxidation of DPBF by US-activated TiH$_{1.924}$ (A) and TiH$_{1.924}$-PVP (B). (C) Comparison of DPBF oxidation by TiH$_{1.924}$ and TiH$_{1.924}$-PVP under US irradiation for 5 min.

Supplementary Figure 16. (A&B) The photographs (A) and XRD spectra (B) of TiH$_{1.924}$ nanodots after treated with H$_2$O$_2$ (1 mM) for 7 days. (C&D) Time-dependent oxidation of DPBF by US-activated TiH$_{1.924}$ nanodots (C) and H$_2$O$_2$-treated TiH$_{1.924}$ (D). (E) Comparison of DPBF oxidation by untreated TiH$_{1.924}$ and H$_2$O$_2$-treated TiH$_{1.924}$ under US irradiation.
Supplementary Figure 17. (A) The relative viabilities of 4T1 cells after incubation with different concentrations of TiH$_{1.924}$-PVP in the presence or absence of laser irradiation (n=6 biologically independent samples). (B) The relative viabilities of 4T1 cells after PTT with TiH$_{1.924}$-PVP for varied laser irradiation durations (n=6 biologically independent samples). (C) Confocal images of 4T1 cells stained with Calcein AM (green, live cells) and propidium iodide (red, dead cells) after different treatments (TiH$_{1.924}$-PVP: 50 µg•mL$^{-1}$, NIR laser: 1064 nm, 0.8 W•cm$^{-2}$, 10 min). Error bars= standard deviation (n=6). Data are presented as mean values ±SD. A representative image of three biological replicates from each group is shown.

Supplementary Figure 18. Quantitative analysis of intracellular ROS generation for cells in different groups as indicated based on confocal fluorescence images in Figure 4E (n=20 cells examined over independent micrographs). Data are presented as mean values ±SD.
Supplementary Figure 19. (A) Confocal images of 4T1 cells stained with DHE probe after various treatments. A representative image of three biological replicates from each group is shown. (B) Quantitative analysis of intracellular red fluorescent signals in different groups as indicated (n=20 cells examined over independent micrographs). Data are presented as mean values ±SD.

Supplementary Figure 20. (A) Tumor inhibition rates of different treated groups. (B) The body weight variation of mice after various treatments (n=5 biologically independent mice). Data are presented as mean values ±SD.
Supplementary Figure 21. H&E staining of major organs (liver, spleen, kidney, heart, lung, and brain) to examine their histological changes after TiH1.926-PVP treatment at 1, 7, and 14 days p.i. A representative image of three biological replicates from each group is shown.