Supporting Information

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Sub-10-nm Pd Nanosheets with Renal Clearance for Efficient Near-Infrared Photothermal Cancer Therapy

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Experimental Details

Materials

Pd(acac)₂ (99%) and N,N-Dimethylpropionamide (DMP) was purchased from Alfa Aesar. PVP (MW=30000, AR) and sodium bromide were purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). Male Sprague-Dawley rats (220 ± 30 g) and Balb/c mice were purchased from the Shanghai SLAC Laboratory Animal Co. Ltd. Human hepatoma cells (QGY-7703) were purchased from cell storeroom of Chinese Academy of Science. RPMI 1640 cell culture medium, bovine serum albumin (BSA) and Penicillin-Streptomycin compound were purchased from Hyclone Laboratories Inc. MTT was purchased from Sigma. The water used in all experiments was ultrapure. All reagents were used as received without further purification.

Synthesis of 20.1 nm Palladium Nanosheets

Pd(II) acetylacetonate (Pd(acac)₂, 10.0 mg), poly(vinylpyrrolidone) (PVP, MW=30000, 32.0 mg) and NaBr (20 mg) were mixed together with Dimethylacetamide (4 mL) and water (2 mL). After 10 hours on standing, the resulting homogeneous yellow solution was transferred to a glass pressure vessel. The vessel was then charged with CO to 1 bar and heated at 100 °C.
for 2 h before it was cooled to room temperature. The dark blue products were precipitated by acetone, separated via centrifugation and further purified by an ethanol-acetone mixture.

**Synthesis of 18.7 nm Palladium Nanosheets**

Pd(II) acetylacetonate (Pd(acac)2, 10.0 mg), poly(vinylpyrrolidone) (PVP, MW=30000, 32.0 mg) and NaBr (20 mg) were mixed together with Dimethylacetamide (2 mL) and water (4 mL). After 10 hours on standing, the resulting homogeneous yellow solution was transferred to a glass pressure vessel. The vessel was then charged with CO to 1 bar and heated at 100 °C for 2 h before it was cooled to room temperature. The dark blue products were precipitated by acetone, separated via centrifugation and further purified by an ethanol-acetone mixture.

**Synthesis of 11.1 nm Palladium Nanosheets**

Pd(II) acetylacetonate (Pd(acac)2, 10.0 mg), poly(vinylpyrrolidone) (PVP, MW=30000, 32.0 mg) and NaBr (30.6 mg) were mixed together with N,N-Dimethylpropionamide (4 mL) and water (2 mL). After 10 hours on standing, the resulting homogeneous yellow solution was transferred to a glass pressure vessel. The vessel was then charged with CO to 1 bar and heated at 100 °C for 2 h before it was cooled to room temperature. The dark blue products were precipitated by acetone, separated via centrifugation and further purified by an ethanol-acetone mixture.

**Excretion Studies and Urine Microscopic Analysis**

Male Sprague-Dawley rats (220 ± 30 g) were purchased from the Shanghai SLAC Laboratory Animal Co. Ltd. Rats were injected via the tail vein with 200 μL of PBS containing 300 μg of SPNS-GSH. Those rats were placed in stainless-steel metabolic cages and urine was collected. After collection of urine, samples of 1 mL digested by aqua regia and measured by ICP-MS.

**ICP-MS analysis**

Each tissue sample was completely digested by 2 mL of aqua regia at room temperature overnight. Subsequently, the solution was diluted to 10 mL using 0.5% HCl and 2% HNO₃. Samples were passed through a 0.22-mm filter to remove any undigested debris prior, and then subjected to ICP-MS measurement. The analysis of Pd content was performed on ICP-MS (PerkinElmer DRC-II). Quantification was carried out by external five-point calibration with internal standard correction and the percentage of injected Pd dose per gram of tissue (%ID/g) was calculated.
Figure S1. (a) Temperatures of the solutions containing 8 ppm LPNS and SPNS, both of which were increased rapidly during continuous irradiation and then returned to its initial value after discontinuing irradiation. (b) The linear relationship between the absorbance at 808 nm and the concentration of LPNS and SPNS.
Figure S2. Micrograph corresponding to *in vitro* photothermal therapy by LPNS-PEI at an optical power density of 0.8 w cm$^{-2}$. Dead cells were stained with Trypan blue.
In vitro stability of SPNS-GSH after incubation with feral bovine serum (FBS). PDDFs [p(r)] of SPNS and SPNS-GSH incubated in whole serum after 24 hours at 37 °C. The arrows indicate the intercepts at the abscissa.
Figure S4. The temperature evolution on tumors of mice with and without SPNS-GSH injection. The tumors were exposed to the 808 nm laser at a power density of 1 W cm$^{-2}$. 
Figure S5. Body weight curves after various treatments. Neither SPNS-GSH injection nor laser irradiation resulted in significant body weight drop of the treated mice.
**Figure S6.** Major organs H&E stained images from untreated mice and mice survived after photothermal therapy. Scale bar: 100 μm.