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Engineering near-infrared fluorescent styene-terminated porous silicon nanocomposites with bovine serum albumin encapsulation for *in vivo* imaging

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Experimental

1. Preparation of PSiNPs. The single side polished, (100) oriented, and p-type silicon wafers (boron doped, 8~10 Ω cm resistivity, purchased from Hefei Kejing Materials Technology Co. Ltd., China) were boiled in 3:1 (v/v) concentrated H_2SO_4/30% H_2O_2 for 30 min and then rinsed copiously with Milli-Q water (≥18 MΩ cm resistivity). The porous silicon (PSi) samples (1.54 cm^2) were prepared by electrochemically etching in an ethanolic HF solution (40% HF/ethanol (1:1 v/v)) at 20 mA/cm^2 for 45 min. After the sonication in water to detach the porous layer and filtration with 0.45 μm filtration membrane, PSiNPs samples were prepared for next experiments.

2. Synthesis of BSA/S- PSiNPs. The single-mode heating microwave system NOVA made by Preekem of Shanghai in China was used for the microwave-assisted synthesis of styrene-terminated PSiNPs (S-PSiNPs) and dodecene-terminated PSiNPs (D-PSiNPs). The fresh prepared porous silicon samples were immersed in pure divinylbenzene, followed by sonication and 20-min microwave heating at 100 °C.

Using 30-min centrifugation at 1.2×10^5 rpm, the samples were washed by ethanol to obtain S-PSiNPs. After 30-min sonication in 200 μg/mL BSA solution, 24-h dialysis in water (100,000 Da molecular weight cut-off), and filtration with 0.45 μm filtration membrane, BSA/S-PSiNPs were prepared for next experiments. And the fresh prepared porous silicon samples were immersed in pure 1-dodecene, followed by sonication and 40-min microwave heating at 120 °C. Using 30-min centrifugation at 1.2×10^5 rpm, the samples were washed by ethanol to obtain D-PSiNPs. After 30-min sonication in 200 μg/mL BSA solution, 24-h dialysis in water (100,000 Da molecular weight cut-off), and filtration with 0.45 μm filtration membrane, BSA/D-PSiNPs were prepared for next experiments.

3. *In vivo* fluorescence imaging. BSA/S-PSiNPs were injected subcutaneously into a nude mouse, and imaged immediately with 430 nm excitation and 700 nm emission filter using the Caliper IVIS spectrum imaging system. The sections of kidney, liver, and spleen tissues harvested from the mice 1 day and 22 days after injection were stained with haematoxylin and eosin and then by a pathologist.

4. Instruments and methods. UV-Vis adsorption spectra were recorded by a Shimadzu UV-2450 spectrophotometer. PL measurements were performed using a Perkin-Elmer LS55 fluorescence spectrometer. X-ray photoelectron spectra (XPS) were recorded using Kratos AXIS Ultra DLD system with a monochromatic Al Kα X-ray beam (1486.6 eV) at 150 W in a residual vacuum of $<4\times10^{-9}$ Pa. Analysis of nanoparticles size and surface charge was performed using Malvern Zetasizer Nano ZS dynamic-light-scattering (DLS) measurements. SEM images were taken by JEOL JSM-7600F scanning electron microscope with the accelerating voltage of 15 kV. TEM images were taken by JEOL JEM-2100 UHR transmission electron microscope with the accelerating voltage of 200 kV.

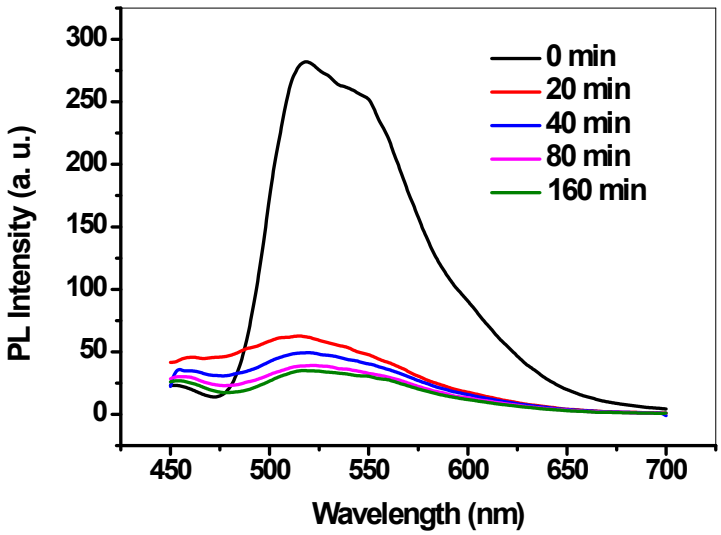

Figure S1. PL spectra of FITC in aqueous solution under UV irradiation, with an excitation of 420 nm.

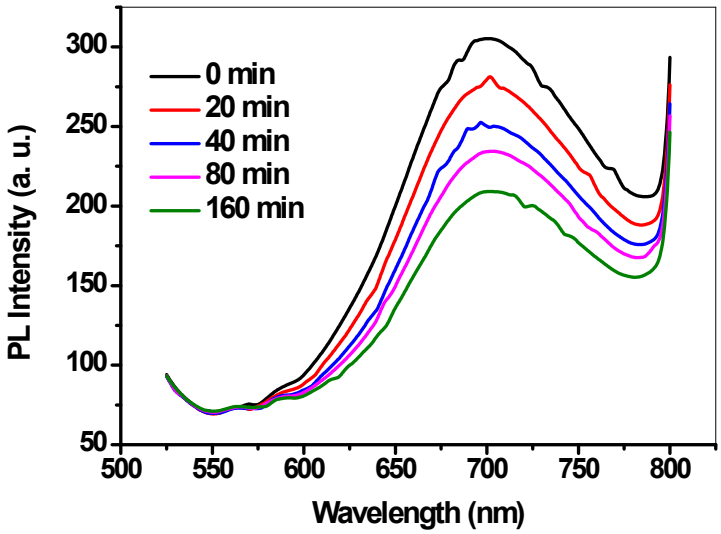

Figure S2. PL spectra of BSA/S-PSiNPs in aqueous solution under UV irradiation with an excitation of 410 nm.

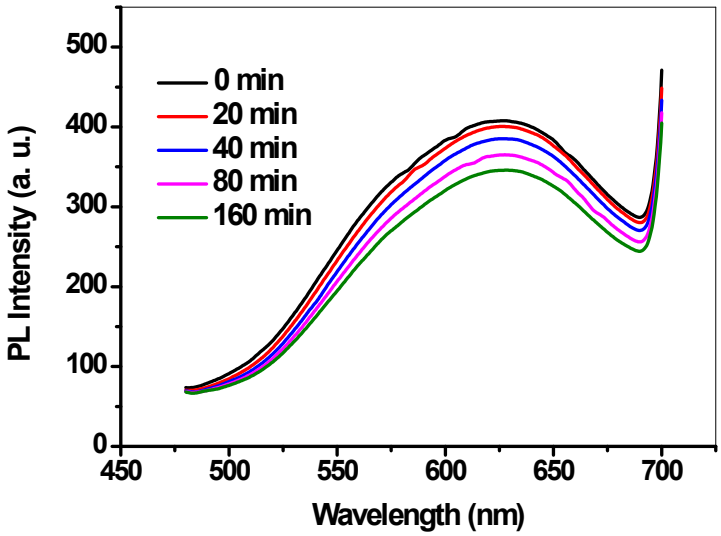

Figure S3. PL spectra of BSA/D-PSiNPs in aqueous solution under UV irradiation, with an excitation of 410 nm.

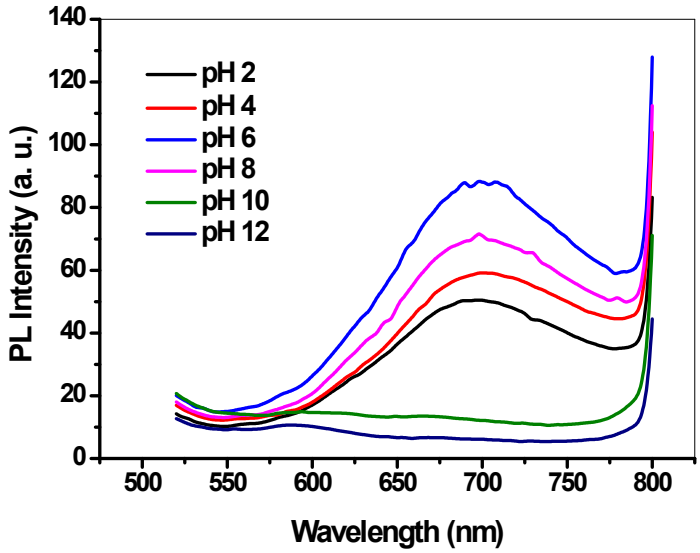

Figure S4. PL spectra of BSA/S-PSiNPs incubated in aqueous solution with different pH values, with an excitation of 410 nm.

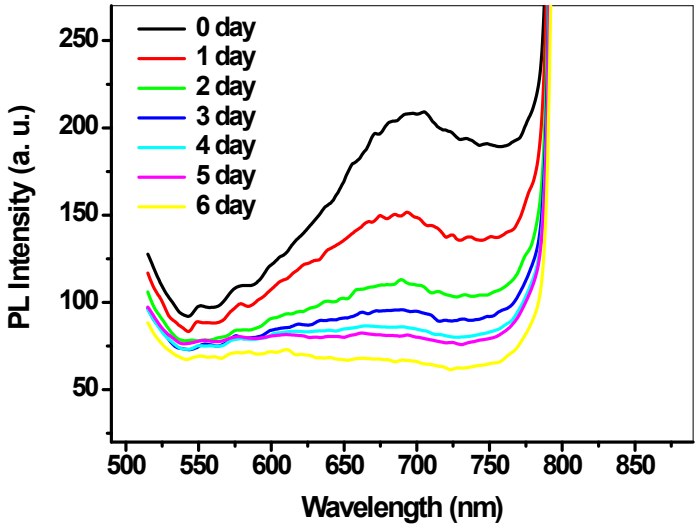

Figure S5. PL spectra of BSA/S-PSiNPs incubated in PBS solution (pH 7.4) at 37 °C for different days, with an excitation of 410 nm.