Supporting Information

Facile Synthesis of Monodisperse Meso-microporous Ta_3N_5 Hollow Spheres and Their Visible Light-Driven Photocatalytic Activity

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Experimental Section

Synthesis of poly(styrene-co-acrylamide) (PSAM) colloidal spheres. Monodisperse PSAM colloidal spheres were synthesized by soap-free emulsion polymerization as follows: Styrene (10.0 g), acrylamide (1.5 g), H_2O (140 g) were charged into a 250 mL three-neck flask and then deoxygenated by bubbling nitrogen gas at room temperature for 60 min. The above mixture was heated to 70 °C under a strirring rate of 200 rpm and ammonium persulfate solution (0.1 g in 5 mL of H_2O) was then injected rapidly. The polymerization was continued for about 10 h. The obtained PSAM spheres were centrifuged and redispersed into H_2O for further use.

Synthesis of Ta_3N_5 hollow spheres. 0.1 g of PSAM was centrifuged and redispersed into 20 mL of ethanol containing 0.1 g of H_2O. Meanwhile, tanatalum ethoxide was dissolved in 20 mL of ethanol containing 60 mg of acetic acid (19.4-39 mM), and then added into the PSAM solution and stirred at room temperature for 2 h to obtain PSAM/Ta_2O_5 hybrid hollow spheres. The as-obtained PSAM/Ta_2O_5 hybrid hollow spheres were separated from the reaction solution by centrifugation, washed with ethanol and deionized water, and dried in a vacuum oven at 40 °C, and then heated at 700 °C under a ammonia flow (1 L/min) for 5 h to obtain Ta_3N_5 hollow spheres.

Characterization. The mophology and structure of as-prepared products were observed by transmission electron microscopy (TEM, Hitachi H-600) and scanning electron microscopy (SEM, Philips XL30). The phase and composition of as-prepared products were measured using X-ray diffraction (XRD) patterns recorded in a Rigaku D/max-kA diffractometer with Cu Kα radiation. FTIR analysis carried out with a Nicolet Nexus 470 FTIR spectrometer with powder pressed KBr pellets. Thermogravimetry analysis was performed using a TGA instrument (Perkin-Elmer TGA-7) in air from room temperature to 900 °C at a rate of 20 °C min^{-1}. The zeta potential was measured by ZetaPlus ζ potential analyzer (Malvern Zetasizer Nano ZS90). The UV-vis adsorption spectra were scanned with Hitachi U-4100 spectrophotometer. Nitrogen adsorption-desorption isotherms were determined at 77 K using an ASAP 2010 analyzer. The surface area was calculated according to the BET method.

Photoreactivity measurement. The photocatalytic activities of Ta_3N_5 hollow spheres powders were evaluated by the degradation of Methylene blue (MB) dye in an aqueous solution under visible light irradiation using 100 W high pressure Hg lamp with a cutoff filter ($\lambda > 400$ nm). 10 mg of Ta_3N_5 hollow spheres powder were suspended in 50 mL of MB aqueous solution (10 mg/L) containing 10 mg of H_2O_2. Prior to irradiation, the suspension was stirred in the dark for 1 h to ensure adsorption/desorption equilibrium. Then, 2 mL of suspension was taken out every 10 min and centrifuged. The MB concentration was analyzed by an UV-vis spectrophotometer. The photodegradation efficiency was monitored at 665 nm. For the sake of comparison, the photocatalytic activities of a commercial Ta_3N_5 (C-Ta_3N_5) and TiO_2 (P25) were also tested in the same condition.

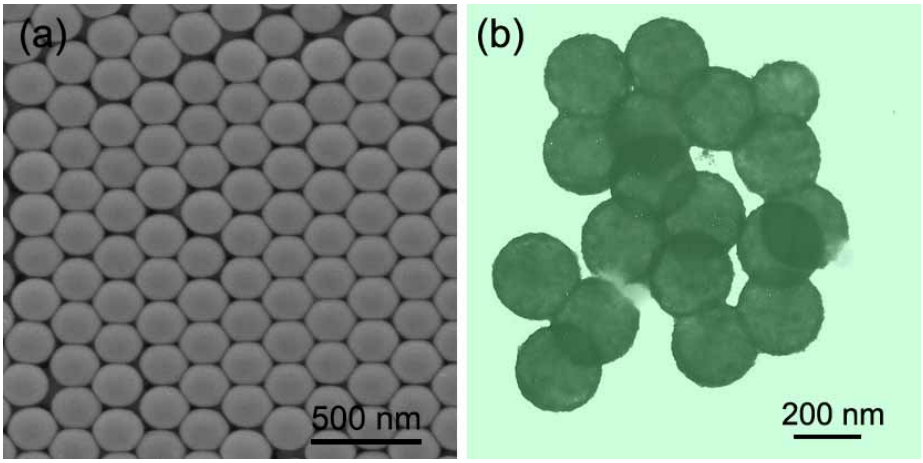

Figure S1. (a) SEM image of monodisperse PSAM spheres. (b) TEM image of PSAM/Ta_2O_5 core/shell spheres obtained from the initial synthesis stage.

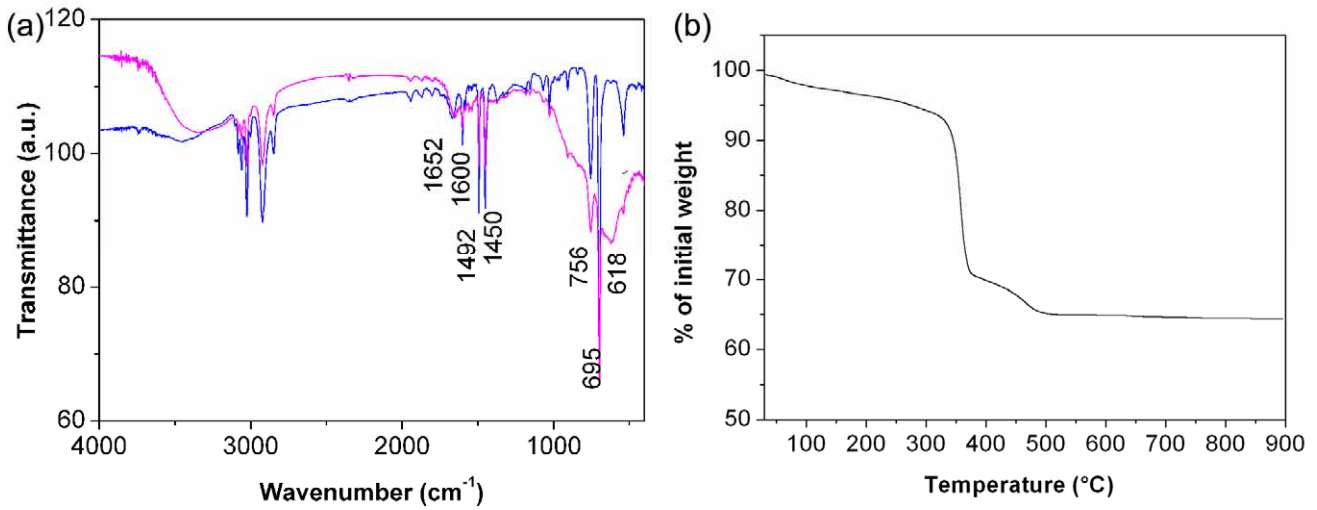

Figure S2. (a) FTIR spectra of PSAM (blue) and PSAM/Ta_2O_5 hybrid hollow spheres (magenta), (b) TGA curve of PSAM/Ta_2O_5 hybrid hollow spheres.

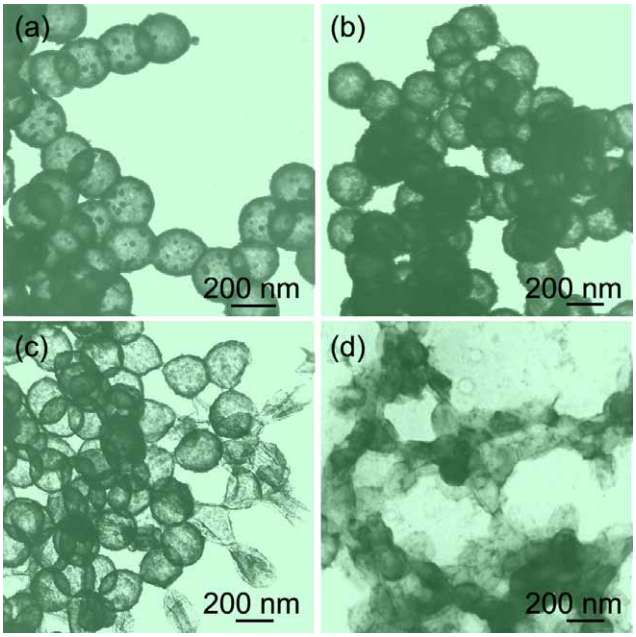

Figure S3. The TEM images of the PSAM/Ta_2O_5 hybrid hollow spheres prepared using different amounts of acetic acid: (a) 0, (b) 30, (c) 90, (d) 120 mg.

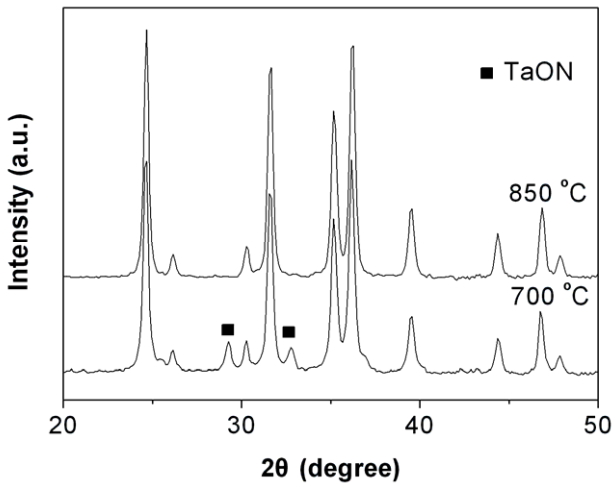

Figure S4. XRD patterns of C-Ta_3N_5 prepared at different temperatures for 5 h in ammonia flow.

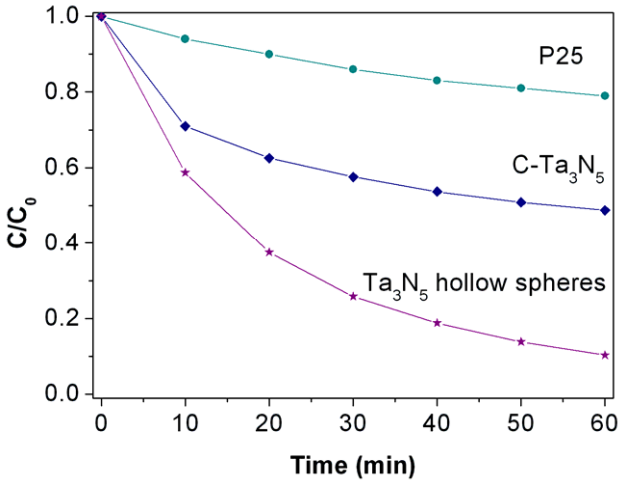

Figure S5. MB degradation efficiency curves for all samples without addition of H_2O_2.