Supporting information

Au@Carbon Yolk-Shell Nanostructures via One-Step Core-Shell-Shell Template

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Au@SiO₂@RF polymer: 15 nm gold colloid solution was prepared by a previously reported method. [1] Typically, 212 mL of water was vigorously stirred under reflux and 25 mL of 2.54 mM HAuCl₄ solution was added. The resultant solution was stirred until boiling point achieved. Then, 12.5 mL of 10 mg/mL⁻¹ sodium citrate solution was added and the system was refluxed for 30 min. Finally, the resultant colloid was cooled to room temperature. Next, 0.325 mL of 12.8 mg/mL⁻¹ polyvinylpyrrolidone aqueous solution, which was freshly prepared by dissolution in water with ultrasonication, was added to a previously prepared, cold, colloidal gold solution. The resultant mixture was stirred for 24 h to allow complete adsorption of the polymer on the gold surface. After this time, the solution was centrifuged and the supernatant was removed. The volume of the concentrated colloid was then adjusted to 6 mL by dilution with water (~ Au concentration 0.03 µM). 2 mL of Au colloid solution was then well dispersed in 10 mL ethanol, then 1 mL ammonia aqueous solution (32 wt%) and 0.5 mL TEOS were added. After 15 min stirring, 70 mg resorcinol and 0.1 mL formaldehyde were added. The
reaction mixture was stirred for 24 h at 30 °C and subsequently heated for 24 h at 100 °C under a static condition in a Teflon-lined autoclave. The brown solid product was collected by centrifugation, washed with water and ethanol, and air-dried at 100 °C overnight. No surface plasmon resonance peak of gold nanoparticles was observed from supernatant, indicating 6×10⁻⁴ µmol Au trapped into the final product with 100% loading efficiency. Au@silica was obtained by calcination of Au@SiO₂@RF polymer at 600 °C in air for 1 hour. The Au@polymer yolk-shell nanostructure was obtained by dissolving the silica core with HF (5 wt %).

*Au@Carbon yolk-shell:* Au@SiO₂@RF polymer was carbonized under N₂ atmosphere at 600 °C for 1 h with a heating rate of 1 °C/min using a MTI OTF-1200X tube furnace. After washing in 5 wt % HF aqueous solution for 24 h, Au@Carbon yolk-shell was obtained.

*Catalytic study:* 1 mL of 0.025 mM o-nitrophenol was mixed with a freshly prepared aqueous solution of NaBH₄ (2 mL, 0.1M). Au@C yolk-shell (5 mg) was then added. UV/Vis absorption spectra were recorded to monitor the change in the reaction mixture. After the reduction process was completed, the catalyst was separated from the mixture and dried in a vacuum oven overnight for reuse in the next reaction cycle – this process was repeated 5 times.

**References**

Figure S1 TEM images of Au-SiO$_2$-RF CSS obtained at different reaction times (a) 1 h, (b) 6 h, (c) 12 h, (d) 18 h and (e) 24 h.

Figure S2 EDX of Au@C yolk-shell nanostructure.
Figure S3 Relationship of $\ln(C_t/C_0)$ and reaction time for the catalyzed reduction of $p$-nitrophenol by Au@C yolk-shell.

Figure S4 TEM images of Au@C yolk-shell nanocatalysts after 5 reaction cycles.