

Supporting Information for

In situ encapsulation of Au nanoparticles in mesoporous core-shell TiO₂ microspheres with enhanced activity and durability

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Experimental

1. Catalyst Preparation

The Au/TiO₂ (Au/Ti molar ratio = 0.53%) was prepared by consecutive solvothermal and hydrothermal treatments. In a typical run of synthesis, a clear solution containing 20 ml ethanol, 1.0 ml TiOSO₄, 10 ml glycerol, 10 ml ethyl ether and desired amount of AuCl₃ was added into a 50 ml autoclave and allowed solvothermal treatment at 110°C for 1 h. After being filtered and washed thoroughly with absolute ethanol, the solid product was dispersed in 40 ml H₂O via ultrasonification, followed by hydrothermal treatment at 110°C for 4 h. The pure TiO₂ was prepared in the similar way in the absence of AuCl₃. Meanwhile, the Pd/TiO₂, Pt/TiO₂, and Ag/TiO₂ samples with M/Ti molar ratios of 6.6%, 0.62% and 1.1% were also prepared by using the corresponding metallic salts instead of Au/TiO₂.

2. Activity test

The catalytic performances of the as-prepared samples were evaluated by using liquid phase photocatalytic degradation of *p*-chlorophenol at 30°C in an 80 ml

self-designed quartz photochemical reactor containing 50 ml of 1.0×10^{-4} M *p*-chlorophenol aqueous solution and 0.050 g catalyst. After reaching adsorption equilibrium, the photocatalytic reaction was initiated by irradiating the system with a 500 W xenon lamp located at 30 cm away from the reaction solution. All the UV lights with wavelength shorter than 360 nm were removed by a glass filter (JB-360). After reaction for 4 h, the unreacted *p*-chlorophenol was analyzed by a UV spectrophotometer (UV 7504/PC) at its characteristic wavelength ($\lambda = 224$ nm). The reproducibility of all results was checked by repeating the experiments at least three times and was found to be within acceptable limits ($\pm 5\%$).

In order to determine the catalyst durability, the photocatalyst was separated from aqueous solution after each run of reactions, followed by washing with H₂O for 3 times and drying at 100 °C for 12 h under vacuum. Then, the catalyst was used in the subsequent reaction under the identical conditions.

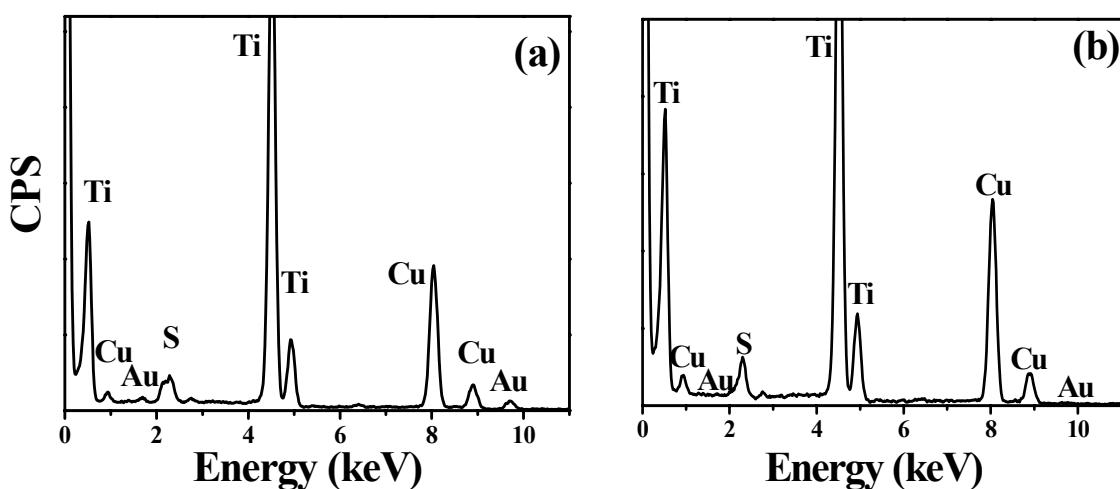


Figure S1. EDX patterns the Au/TiO₂ detected inside the core-shell chamber (a) and on the outer surface (b).

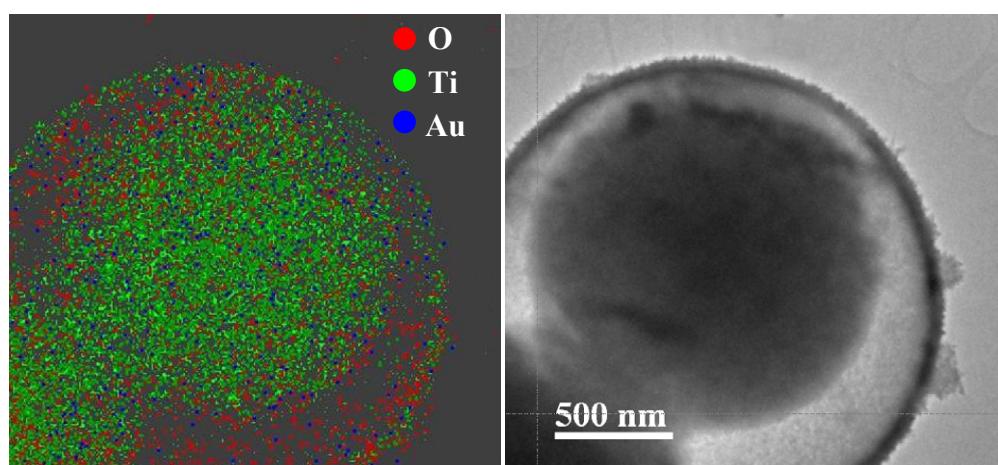


Figure S2. Mapping and TEM images of the Au/TiO₂ sample.

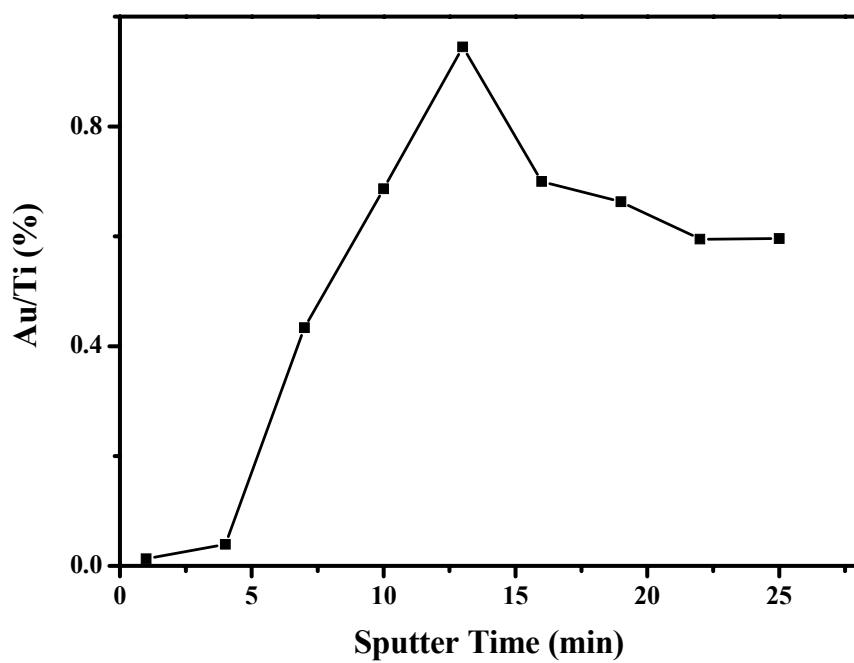


Figure S3. The Au content distribution in the Au/TiO₂ sample determined by XPS spectra with increasing Ar⁺ sputtering time.

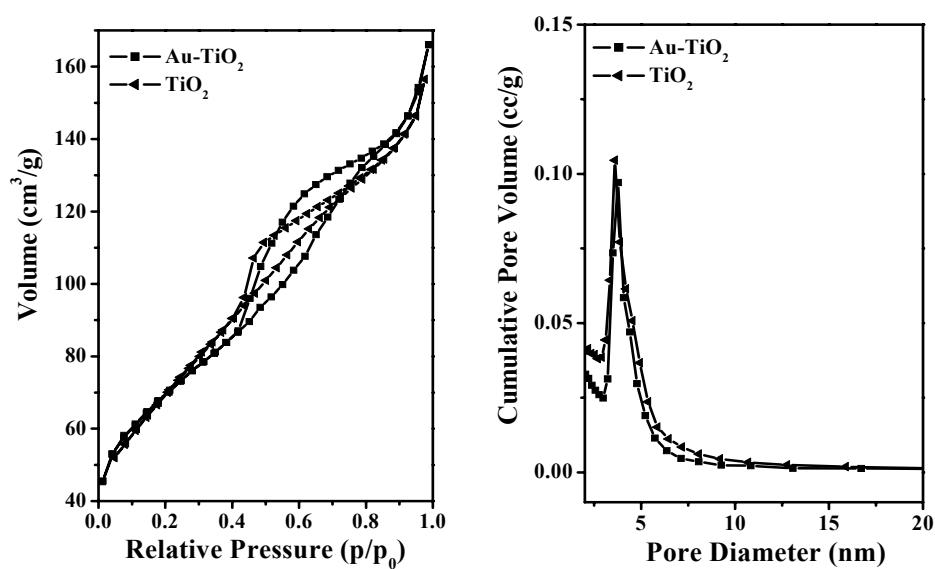


Figure S4. N₂ adsorption-desorption isotherm (Left) and pore size distribution curve (Right) of the Au/TiO₂ sample.

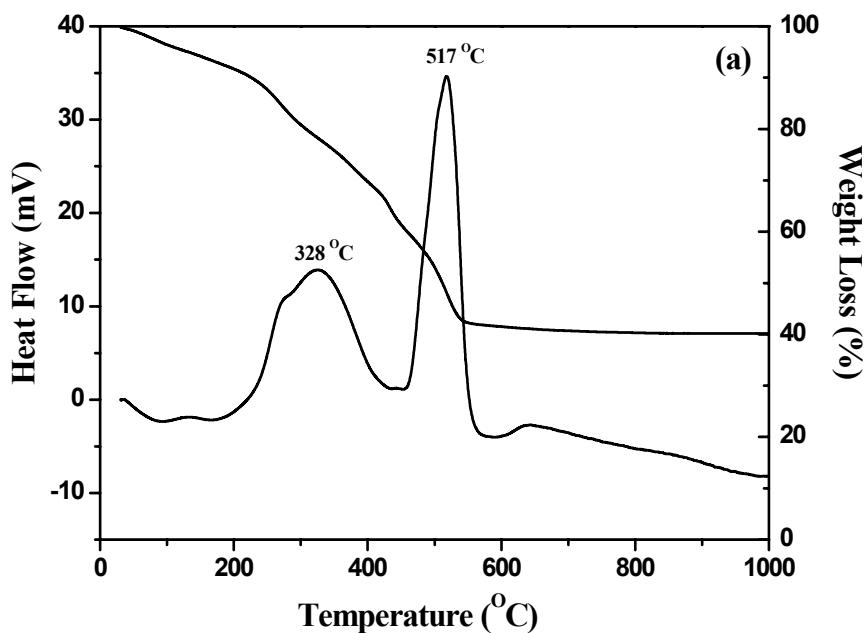


Figure S5. TG/DTA curves of the Au/TiO₂ precursor obtained *via* solvothermal treatment at 110°C for 1 h.

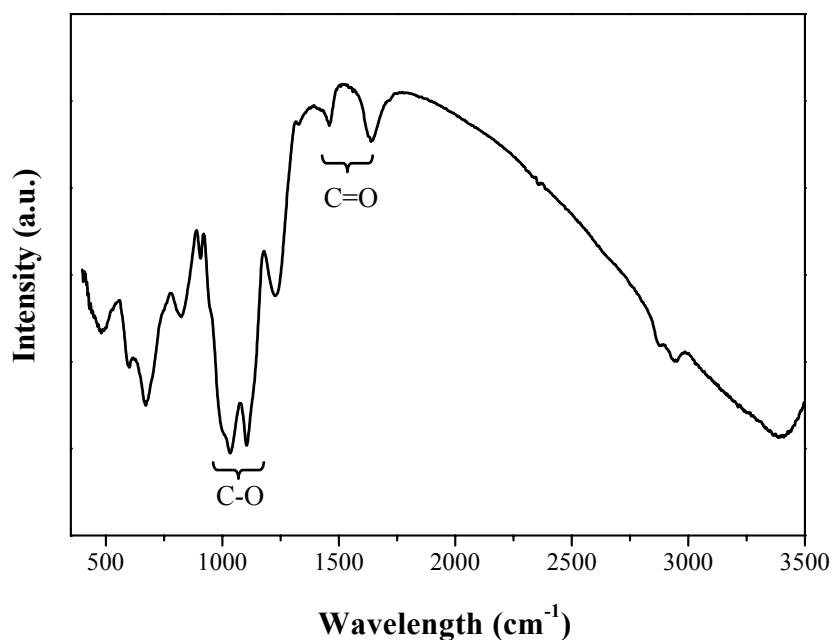


Figure S6. FTIR spectrum of the Au/TiO_2 precursor obtained *via* solvothermal treatment at 110°C for 1 h.

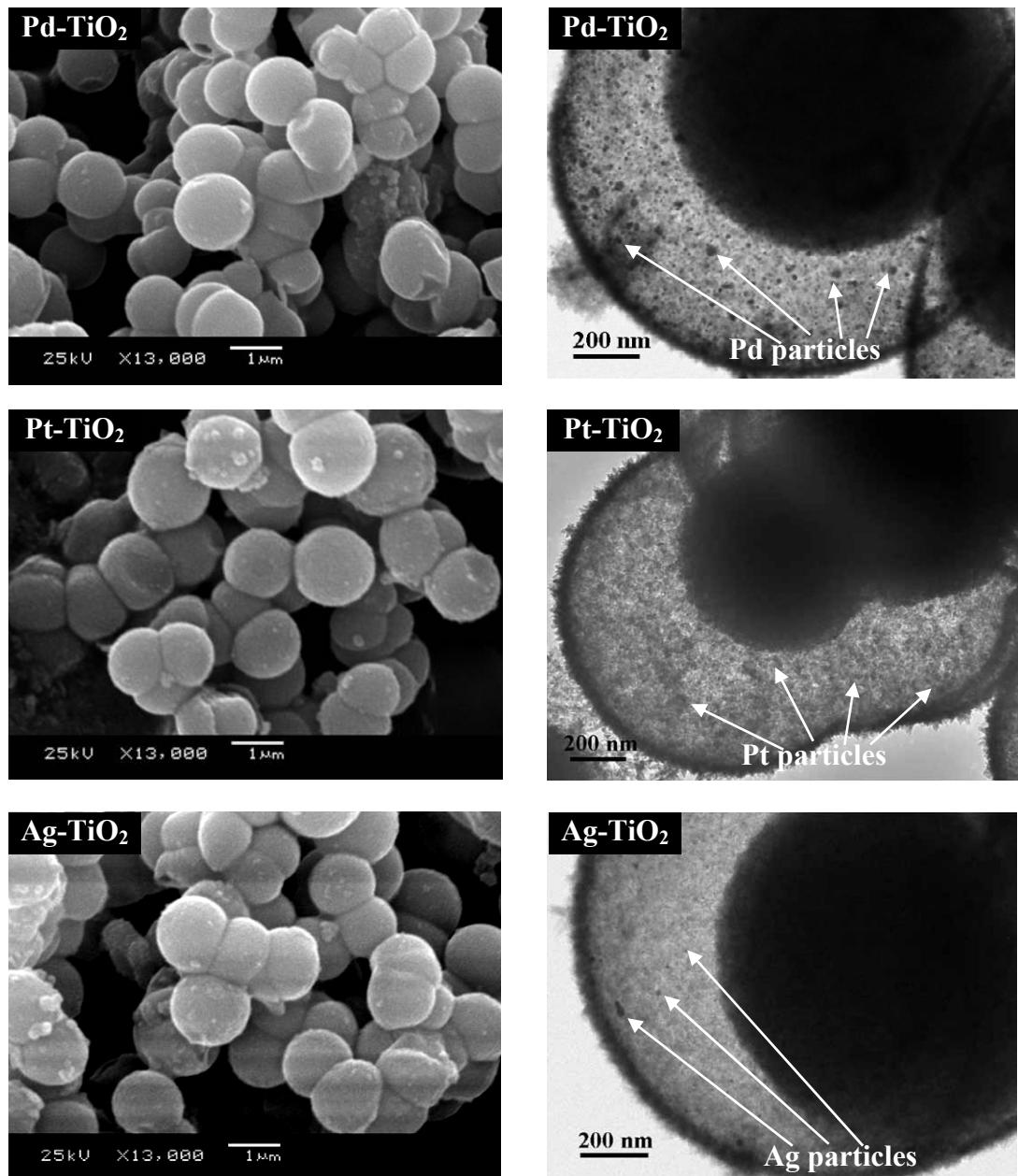


Figure S7. SEM (Left) and HRTEM (Right) images of Pd/TiO₂, Pt/TiO₂, and Ag/TiO₂ samples.

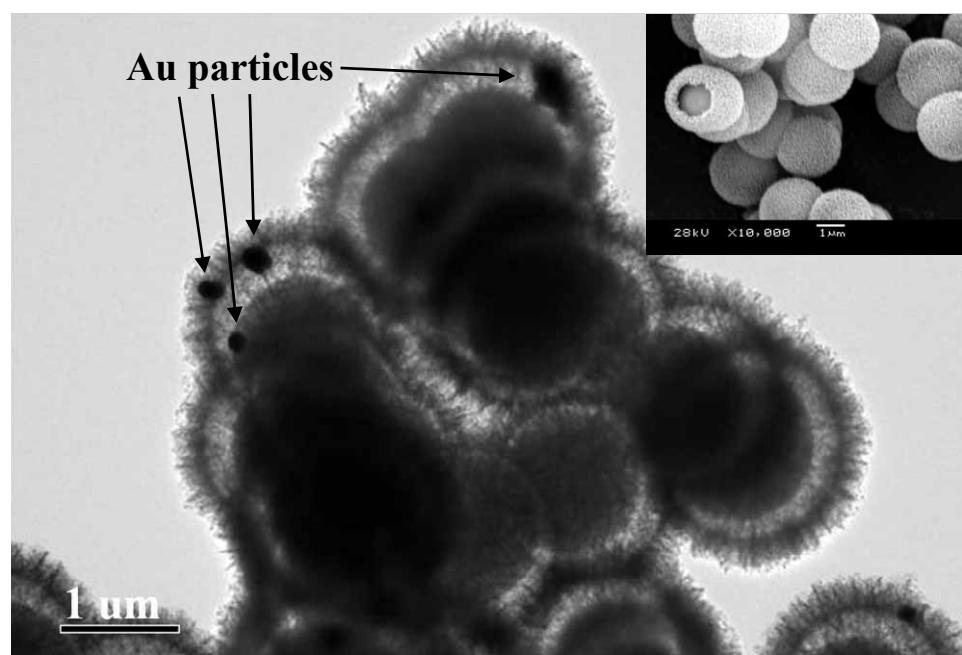


Figure S8. TEM image of the Au/TiO₂ sample obtained via solvothermal treatment for 48 h. The attached is the SEM image.

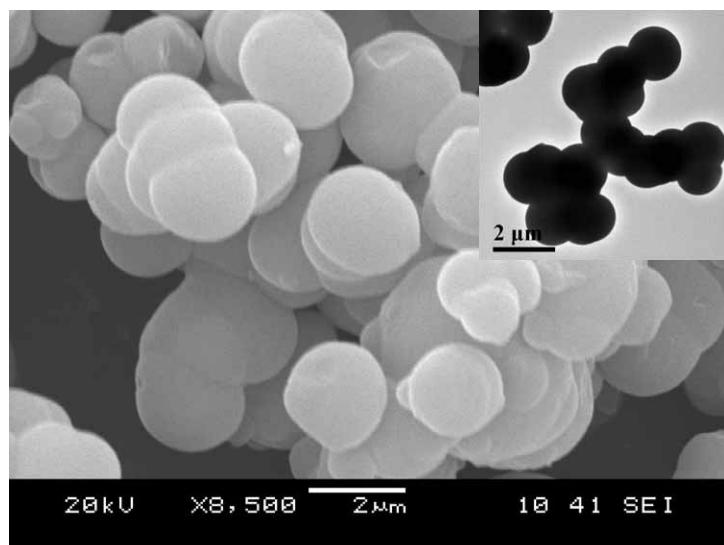


Figure S9. SEM image of the Au/TiO₂ sample obtained via hydrothermal treatment at 110°C for 48 h in the presence of 5.0 ml water. The attached is the SEM image.