

Supplementary Material

**Au/3DOM Co₃O₄: Highly active nanocatalysts for the oxidation
of carbon monoxide and toluene**

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Catalyst Characterization Procedures

The real Au contents in the $x\text{Au}/3\text{DOM Co}_3\text{O}_4$ and $x\text{Au}/\text{bulk Co}_3\text{O}_4$ samples were measured using the ICP-AES technique on a Thermo Electron IRIS Intrepid ER/S spectrometer. The samples were dissolved in a mixture of concentrated HCl and HNO_3 with volumetric ratio of 3/1 prior to the analysis. X-ray diffraction (XRD) patterns of the samples were recorded on a Bruker D8 Advance diffractometer with Cu $K\alpha$ radiation and nickel filter ($\lambda = 0.15406 \text{ nm}$). BET (Brunauer-Emmett-Teller) surface areas of the samples were determined via N_2 adsorption at -196°C on a Micromeritics ASAP 2020 analyzer with the samples being outgassed at 300°C for 2.5 h under vacuum before measurement. Scanning electron microscopic (SEM) images of the samples were recorded on a Gemini Zeiss Supra 55 apparatus (operating at 10 kV). Transmission electron microscopic (TEM) images and selected-area electron diffraction (SAED) patterns of the samples were obtained using the JEOL-2010 equipment (operating at 200 kV). X-ray photoelectron spectroscopy (XPS, VG CLAM 4 MCD analyzer) was used to determine the Co 2p, O 1s, Au 4f, and C 1s binding energies (BEs) of surface species using Mg $K\alpha$ ($h\nu = 1253.6 \text{ eV}$) as excitation source. In order to remove the adsorbed water and carbonate species on the surface, the samples were pre-treated in O_2 (flow rate = 20 mL/min) at 450°C for 1 h and then cooled to RT, followed by transferring the pre-treated samples into the spectrometer in a transparent Glove Bag (Instruments for Research and Industry, USA) filled with helium. The pre-treated samples were outgassed in the preparation chamber (10^{-5} Torr) for 0.5 h and then introduced into the analysis chamber ($3 \times 10^{-9} \text{ Torr}$) for XPS spectrum recording. The C 1s signal at 284.6 eV was taken as a reference for BE calibration.

Hydrogen temperature-programmed reduction (H_2 -TPR) experiments were carried

out on a chemical adsorption analyzer (Autochem II 2920, Micromeritics). Before TPR measurement, *ca.* 0.02 g of catalyst (40–60 mesh) was loaded to a quartz fixed-bed U-shaped microreactor (i.d. = 4 mm) and pretreated in an O₂ flow of 30 mL/min at 300 °C for 1 h. After being cooled at the same atmosphere to RT, the pretreated sample was exposed to a flow (50 mL/min) of 5% H₂–95% Ar (v/v) mixture and heated from RT to 700 °C at a ramp of 10 °C/min. The alteration in H₂ concentration of the effluent was monitored on-line by the chemical adsorption analyzer. The reduction peak was calibrated against that of the complete reduction of a known standard of powdered CuO (Aldrich, 99.995%).

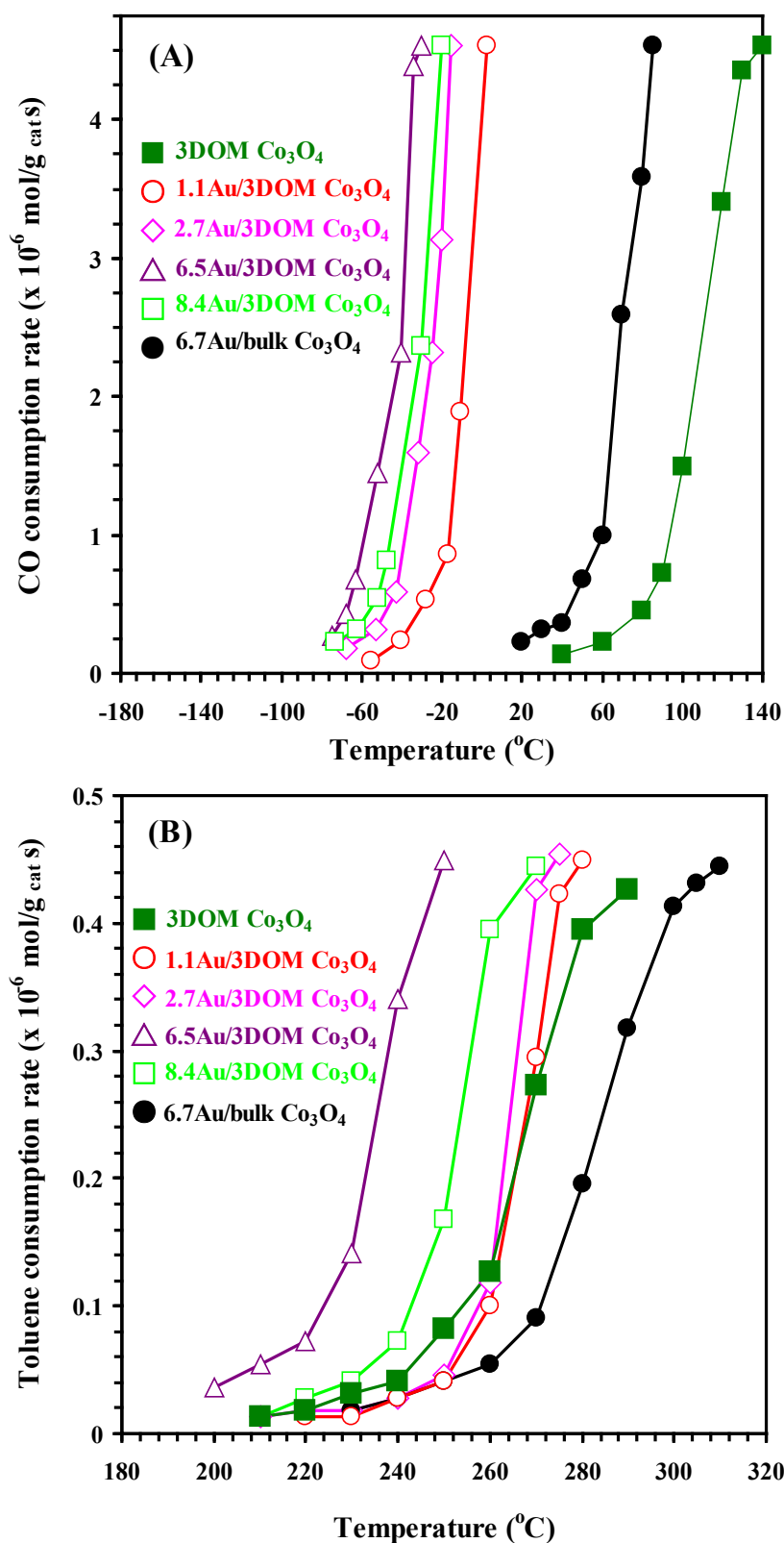


Fig. S1. (A) CO consumption rate and (B) toluene consumption rate as a function of temperature over the 3DOM Co_3O_4 and $x\text{Au}/\text{Co}_3\text{O}_4$ samples under the conditions of CO concentration = 1 vol%, CO/O_2 molar ratio = 1/20, and $\text{SV} = 10,000 \text{ mL}/(\text{g h})$ or toluene concentration = 1000 ppm, toluene/ O_2 molar ratio = 1/400, and $\text{SV} = 20,000 \text{ mL}/(\text{g h})$.

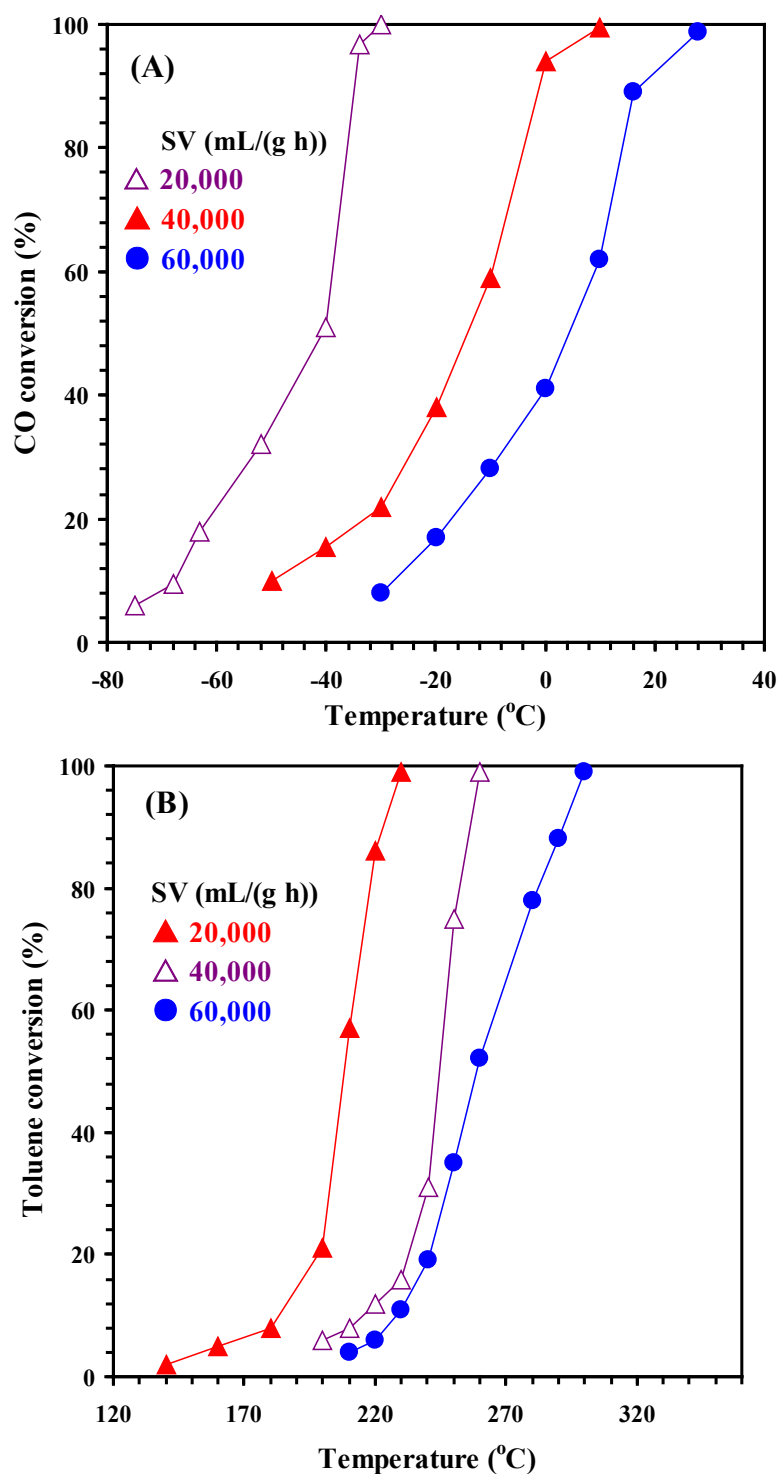


Fig. S2. Effect of SV on (A) CO and (B) toluene conversions over 6.5Au/3DOM Co_3O_4 under the conditions of CO concentration = 1 vol% and CO/O₂ molar ratio = 1/20 or toluene concentration = 1000 ppm and toluene/O₂ molar ratio = 1/400.

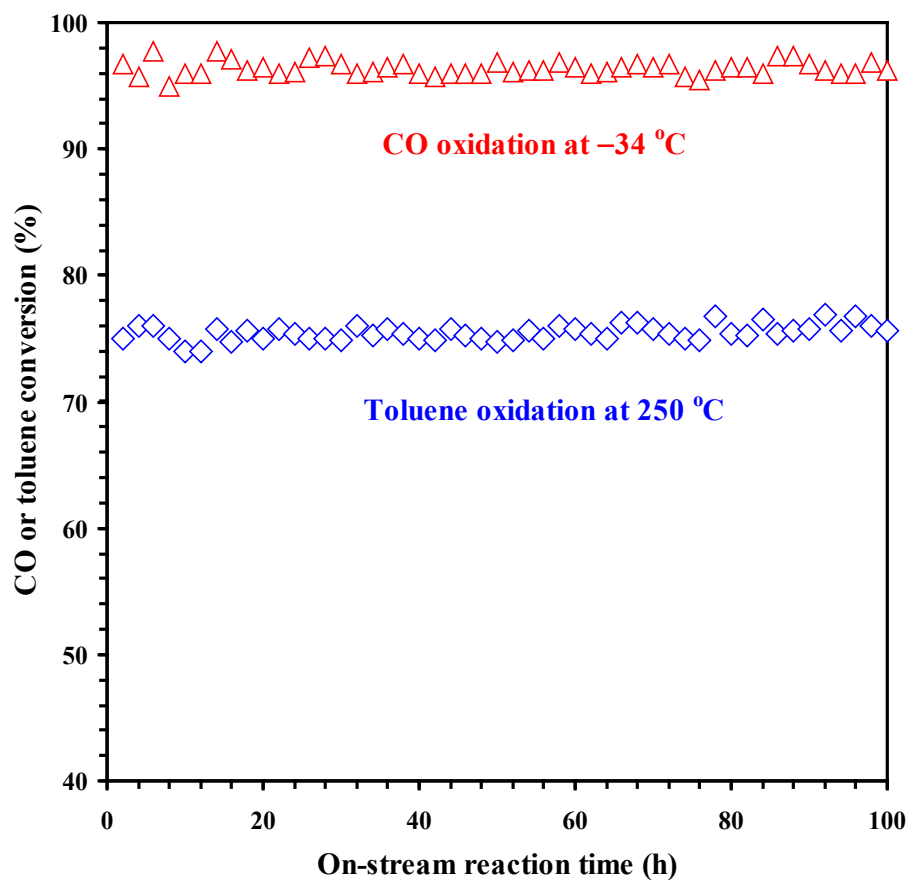


Fig. S3. CO conversion (Δ) and toluene conversion ($\diamond$) as a function of on-stream reaction time over 6.5Au/3DOM Co_3O_4 at $-34\text{ }^\circ\text{C}$ for CO oxidation at $\text{SV} = 20,000\text{ mL}/(\text{g h})$ and $250\text{ }^\circ\text{C}$ for toluene oxidation at $\text{SV} = 40,000\text{ mL}/(\text{g h})$.