Electronic Supplementary Information

Shape-controllable synthesis of CeO₂ particles in CO₂-expanded ethanol towards CO oxidation application

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Experiments

All the chemicals used in this work were of analytical grade and used as received. In a typical procedure, 10 mL of $Ce(NO_3)_3 \cdot 6H_2O$ (10 mg·mL⁻¹) dispersed in ethanol were first transferred to the vessel. Subsequently, the pressure was increased to a desired initial CO_2 pressure at ambient temperature. Then, the temperature was increased to 110 °C. After reaction for 2 h, the vessel was cooled down and the precipitates was collected, followed by being washed repeatedly with deionized water and absolute ethanol several times. Before characterizations and catalysis application, the as-obtained products were calcined at 600 °C for 1 h.

Characterations

The crystalline structure was characterized by X-ray diffraction (XRD) using a Rigaku D/Max 2500 powder diffractometer with Cu K α radiation ($\lambda = 1.5406$ Å). Raman spectra were collected using a BrukerRFS-100/S Raman spectrometer with Fourier transform. A 514 nm YAG laser was used as the excitation source, and its power was kept at 150 mW. The morphology of the catalyst was evaluated by field emission scanning electron microscopy (FE-SEM, Leo 1530 FEG SEM), while transmission electron microscopy (TEM) was used to examine the microstructure (Philips Tecnai 20 G2 S-TWIN).

Catalyst evaluation

The CO oxidation reactions were carried out in a fixed-bed reactor using 20 mg catalyst for each sample. The reactant gas (1 vol. % CO and 1.98 vol. % O₂ balanced with helium) was

admitted at a flow rate of 20 ml/min, corresponding to a space velocity of 24 $L^{\bullet}h^{-1}g_{cat}^{-1}$. The flow rate of reactant gas was monitored by mass flow controllers. The temperature was controlled by a thermocouple placed inside the catalyst bed. The effluent gas was analyzed with an HP-6890 gas chromatograph equipped with a thermal conductivity detector (TCD) and a molecular 13 \times column using helium as the carrier gas.