Supplementary Information

Supported PEG-Phase Nanoparticles and their Application in Palladium-Catalyzed Aerobic Oxidation in Supercritical Carbon Dioxide

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Characterization of the resulting solid catalysts by XRD, IR, TEM and ²⁹Si MAS NMR

The X-ray powder patterns for qualitative phase analysis were collected on a Stoe STADI P transmission diffractometer in Debye-Scherrer geometry with a primary monochromator curved germanium (111) and a linear position sensitive detector. The used radiation was Cu $k_{\alpha l}$: 1.54060Å. The data were collected in the range between 0 to 10° 20 with a step width of 0.01° 20. For the measurements, the sample was prepared between two polyacetate foils. IR spectra were performed with Nicolet Magna-IR 750 instrument. The ²⁹Si MAS NMR spectroscopy were recorded on a Bruker Avance 500WB instrument using a 4-mm MAS probe at a spinning rate of 10 kHz, 30 s recycle delay, 2,800 scans, and 2.2 µs $\pi/4$ pulse. Nitrogen sorption isotherms were measured at 77 K with a Quantachrome instrument Nova 3000e sorption analyzer. Prior to the measurements, the samples were evacuated at 393 K for 8 h. Transmission electron microscopy (TEM) were chosen to investigate structural features of the catalysts with a H-7500 instrument. All results on characterization which are not included in the main text are shown in Figures 1S-5S.



Fig. 1S XRD pattern of sample **4a**. Samples **4b** and **7** display the similar characteristics like **4a**. The broad peak around $2\theta = 20-30^{\circ}$ are assigned from the formation of amorphous silica particles.



Fig. 2S IR spectra of samples **4a** and **8** showing no fundamental differences. The peak around 2900 cm⁻¹ and 1000-1500 cm⁻¹ (some peaks overlapped) are assigned to the adsorption of PEG and the peak around 1000 cm⁻¹ is assigned to the adsorption of silica.



Fig. 3S ²⁹Si MAS NMR spectra of sample **3a**. From the spectra, the resonances near –100 and -109 ppm represent the Q^3 HO*Si*(OSi)₃ and Q^4 *Si*(SiO)₄ environments of the SiO₄, whereas signals about 65 ppm and 56 ppm arise from T³ and T² connectivities of the organic-functionalized silicon centers, respectively.



Fig. 4S TEM micrographs of sample 5 before (A) and after reaction (B). The scale bar is 50 nm.



Fig. 5S TEM micrographs of sample 8 before (left) and after reaction (right). The scale bar is 50 nm.