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 \(^{29}\)Si 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\(_{\alpha 1}\): 1.54060 Å. The data were collected in the range between 0 to 10° 2\(\theta\) with a step width of 0.01° 2\(\theta\). For the measurements, the sample was prepared between two polyacetate foils. IR spectra were performed with Nicolet Magna-IR 750 instrument. The \(^{29}\)Si 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 \(\mu\)s \(\pi/4\) 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 $^{29}$Si MAS NMR spectra of sample 3a. From the spectra, the resonances near -100 and -109 ppm represent the $Q^3$ HOSi(OSi)$_3$ and $Q^4$ Si(SiO)$_4$ environments of the SiO$_4$, whereas signals about 65 ppm and 56 ppm arise from $T^3$ and $T^2$ 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.