Electronic supplementary information (ESI)

Hydrodechlorination of chlorophenols at low temperature on a novel Pd catalyst†

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Experimental Section

Pd/MSCN catalyst characterization: the X-ray diffraction (XRD) measurements of MSCN and Pd/MSCN were taken on a Rigaku Dmax-3C diffractometer using Cu KR radiation (40 kV, 30 mA, $\lambda = 0.15408$ nm). N_2 adsorption-desorption isotherms were measured at 77 K with a Quantachrome NOVA 4000e analyzer. A JEM 2100F microscope, equipped with a field emission gun, operated at 200 kV was used for the scanning transmission electron microscopy-high angle annular dark field (STEM-HAADF) imaging. X-ray photoelectron spectroscopy (XPS) measurements were performed on a Perkin-Elmer PHI 5000CESCA system with a base pressure of 10^{-9} Torr. The Pd loading on the carriers was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Varian VISTA-MPX). H_2 chemisorption was conducted on a Quantachrome CHEMBET-3000 system by pulsing hydrogen on the supported Pd catalyst.

A typical reaction procedure: Pd/MSCN (5 wt.%) (0.10 g, 47.3 μmol) was added to a stirred solution of 4-chlorophenol (0.648 g, 5 mmol), Et_3N (0.511 g, 5 mmol) and 50 ml methanol in a 100-ml three-nicked flask in an ice brine bath at 258 K. The resulting reaction mixture was flushed 6 times with hydrogen to remove air before being stirred vigorously under hydrogen atmosphere (balloon). At a given time point, 0.4 ml reaction mixture was sampled using a 1-ml syringe through the rubber stopper to a 0.7-ml plastic centrifuge tube and the catalyst was removed by centrifugal separation. Samples were analyzed by gas chromatography. The concentration of chlorophenols in the bulk liquid phase was determined from the total molar balance in the reaction mixture.

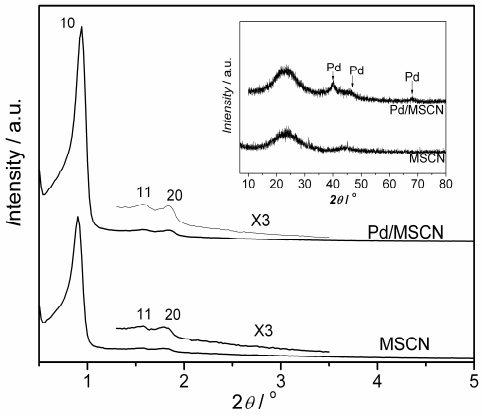

Figure S1. Small-angle and wide-angle XRD patterns of Pd/MSCN and MSCN.

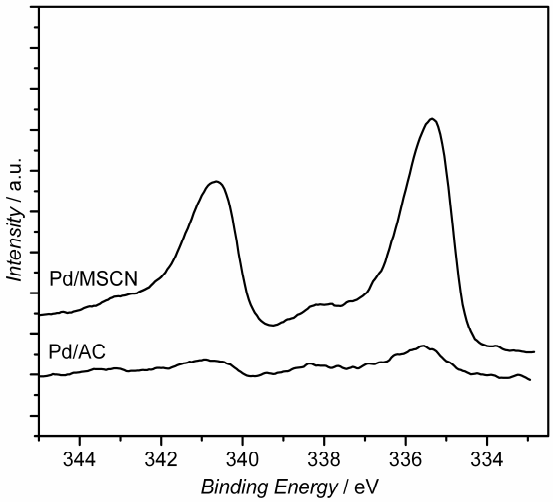

Figure S2. XPS results of Pd/MSCN and Pd/AC

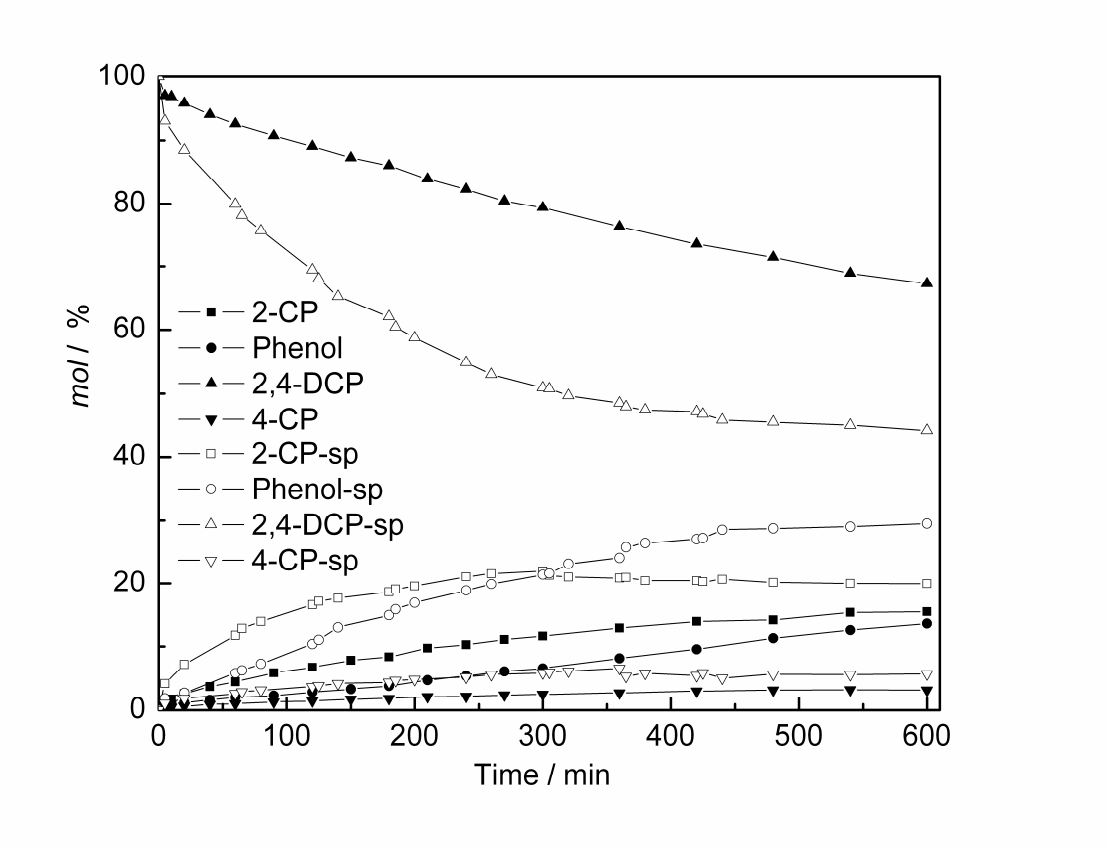

Figure S3. HDC of 2,4-DCP over 5 wt.% Pd/AC (*Pd:Cl*=1:100 mol) at 258 K under ordinary hydrogen pressure (balloon) using Et_3N (1.0 equiv Et_3N *vs* the number of chlorine atoms); 2, 4-DCP concentration: 5 mmol/50 ml methanol solution; sp: the addition of 0.1 equiv *vs* the number of chlorine atoms per 30 min.

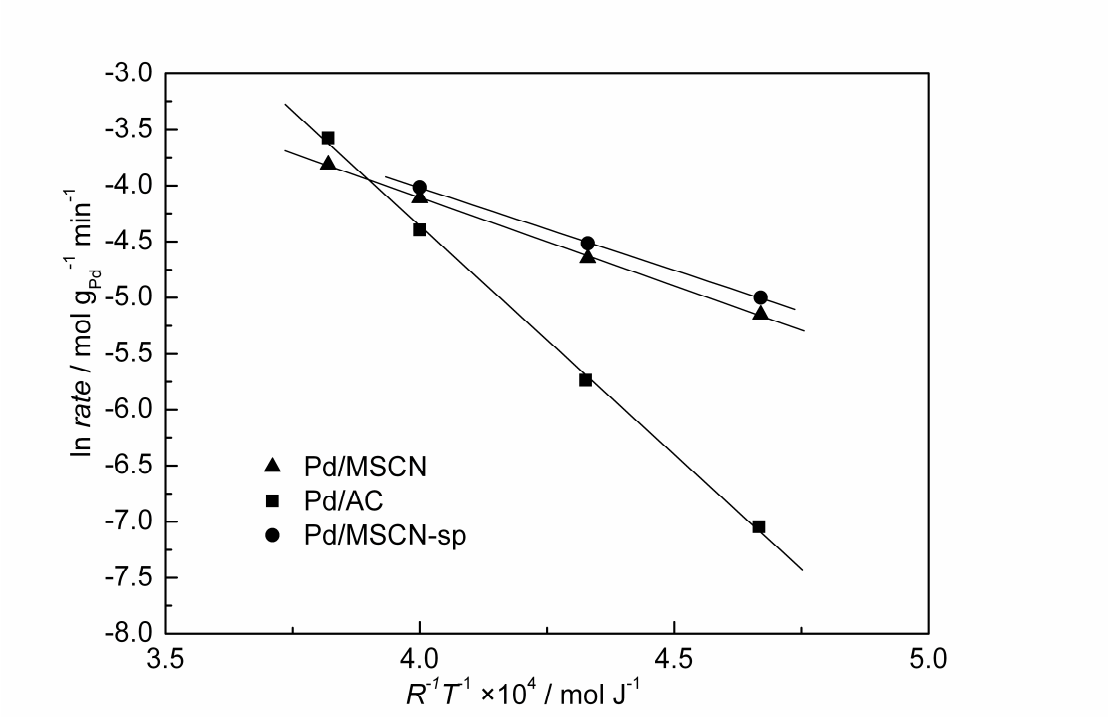

Figure S4. Initial HDC rates for 4-CP on 5 wt.% Pd/MSCN and 5 wt.% Pd/AC as a function of temperature (258-313 K) under ordinary hydrogen pressure (balloon) using Et_3N (1.0 equiv *vs* the number of chlorine atoms); 4-CP concentration: 5 mmol/50 ml methanol solution; sp: the addition of 0.25 equiv *vs* the number of chlorine atoms per 60 min.