SUPPORTING INFORMATION

Nanoscale Pd Supported on 3D Porous Carbon for Enhanced Selective Oxidation of Benzyl Alcohol

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1.Experimental Details

1.1 Catalyst Synthesis

The PCF was applied in synthesizing Pd/PCF catalysts with impregnation method. Typically, PCF was mixture with ethyl alcohol and ultrasound by ultrasonic cleaning machine for half a minute. Simultaneously, the palladium nitrate solution (10wt% $Pd(NO_3)_2$ in 10 wt% nitric acid, Sigma-Aldrich) was mix and ultrasound in absolute ethyl alcohol for half a minute.

Then put the solution which contains Pd dropwisely to the alcoholic solution of PCF, and the mixture was stirred on magnetic stirrer apparatus until the alcohol was volatilized completely and dried in vacuum desiccator at 60°C for about 2 h. Whereafter, the finished product was treated in horizontal tubular furnace under flowing combination gas, which consisted of 75% helium and 25% hydrogen at 250°C for 2 h, and samples were cooled under helium to room temperature and obtained the low-loading Pd/PCF, ultimately.

1.2 Catalyst Test of Benzyl Alcohol Oxidation Reaction

The two catalysts were tested in a commercial available continuous flow reactor Phoenix Flow Reactor™ from ThalesNano. The oxidation reaction systems include a JASCO PU-2085 plus HPLC pump, a Gas Module and a JASCO BP-2080 Plus back pressure regulator with the Phoenix Flow Reactor™, which can achieve reactions under high temperature at high pressure. The catalysts were put into a CatCart@, which has a specification of approximately 30 mm length×3.5 mm internal diameter. Both catalysts were tested on the same conditions of atmospheric pressure at 80°C, with flow rates of atomic oxygen and liquid reactant were 5ml/min and 0.05ml/min, respectively. Ultimately, the products obtained were detected by the High Performance Liquid Chromatography (HPLC), the results of which were used to calculate the condition of benzyl alcohol and the selectivity of aim product benzaldehyde.

1.3 Characterization

All samples were treated with aqua regia, and the solution after filtered was measured with an inductively coupled plasma optical emission spectrometer (ICP-OES, PerkinElmer Optima 8000) to analysised the metal content of each sample. Field-emission scanning electron microscopy (FESEM; JSM 6701F) was employed to investigate the surface and cross-sectional morphologies

of the as-prepared carbon before and after reaction. To investigate the structure and composition of the samples, powder X-ray diffraction (XRD; RigakuD/Max-2400) was performed using Cu Kα radiation, which performed at 40 kV and the diffraction angles(2θ) ranges from 5° to 90°. X-ray photoelectron spectroscopy(XPS) was performed with a Thermo ESCALAB 250 instrument equipped with monochromatic AL Kα radiation, 150W and UHV conditions. Raman spectra were carried on a Horiba Scientific LabRAM HR Evolution system with an Ar-ion laser at an excitation wavelength of 633 nm. TEM images were obtained in a JEOL JEM-2100F operating at 200 kV. The temperature programmed desorption (TPD) were performed on Chemisorption Analyzer AutoChem II. The samples were purged and further heat-treated in purified Ar, when the reactor was cooled to 50°C in Ar, the samples were exposed to pure CO_2 or 20% NH_3/Ar for 30 min, and purged with Ar for 2 hours at the same temperatures in order to eliminate the physically adsorbed CO_2/NH_3.

2. Supplementary Figures

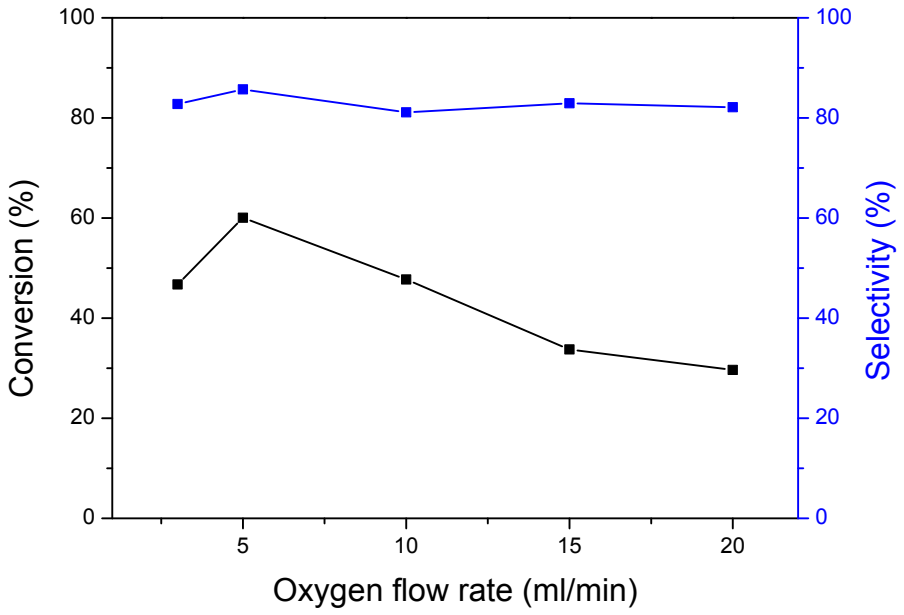

Figure S1. Catalytic performances of Pd/PCF catalyst for benzyl alcohol oxidation of different oxygen flow rates.

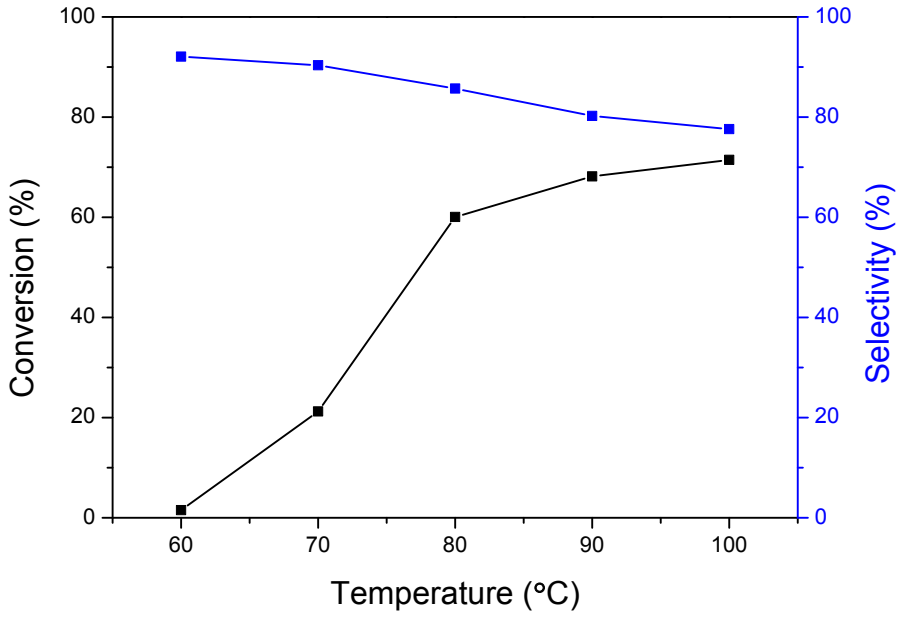

Figure S2. Catalytic performances of Pd/PCF catalyst for benzyl alcohol oxidation of different temperatures.