Supporting Information

Enhanced Catalytic Performance of Pd/SiC for Hydrogenation of Furan Derivatives at Ambient Temperature under Visible Light Irradiation

Zhi-Feng Jiao,^{*ab*} Xiao-Ning Guo,^{*a*,*} Zhao-Yang Zhai,^{*a*} Guo-Qiang Jin,^{*a*} Xiao-Min Wang,^{*b*} and Xiang-Yun Guo^{*a*,*}

^{*a*} State Key Laboratory of Coal Conversion, Institute of Coal Chemistry, Chinese Academy of Sciences, Taiyuan 030001, PR China.

E-mail: guoxiaoning@sxicc.ac.cn (XNG); xyguo@sxicc.ac.cn (XYG)

^b College of materials science and engineering, Taiyuan University of Technology, Taiyuan 030024, PR China.

Preparation of high surface area SiC: The synthesis process included the xerogel preparation and subsequent carbothermal reduction. The xerogel was prepared as follows. Firstly, 6 g of phenolic resin and 0.5 g of nickel nitrate were dissolved in 18 ml of ethanol (AR) and then mixed with 25 ml of tetraethoxysilane (TEOS, AR) under stirring. Secondly, 0.5 ml of hydrochloric acid was added into the mixture, and the mixture was then stirred for 24 h to enhance the hydrolysis of TEOS. Finally, 5 ml of hexamethylenetetramine (HMTA, 35.8%) aqueous solution was dropped into the above mixture for rapid gelation. The xerogel was obtained by drying the gel at 110 °C for 12 h. The carbothermal reduction was carried out in a horizontal alumina tubular furnace. The xerogel was placed in a small alumina boat and then put into the heating zone of the furnace. The xerogel was heated in Ar flow (40 cm³/min) to 1000 °C at a rate of 10 °C/min, then to 1300 °C at a rate of 2 °C /min and maintained at this temperature for 5 h. After the furnace was cooled down to room temperature, the as-prepared product containing SiC was collected. The raw product was heated in air at 700 °C for 3 h to remove the residual carbon, and subsequently treated by nitric acid (HNO₃) and then hydrofluoric acid (HF) to eliminate the unreacted silica and other impurities. A light-green powder was obtained after washing with distilled water.

Preparation of Pd/SiC catalysts: The Pd/SiC catalyst was prepared via a two-step route. First, 291 mg of SiC and 22.5 mg of palladium nitrate were dispersed into absolute ethanol under sonication, then the suspension was magnetically stirred to get a mixture of palladium nitrate and SiC. Second, the mixture was reduced using diethylene glycol for 2 h at 180 °C to obtain a 3wt.% Pd/SiC catalyst. For comparison, Pd/SiO₂, Pd/Al₂O₃ and Pd/TiO₂ with the same Pd loading of 3wt% were also prepared in a similar method but using different supports.

Photocatalytic hydrogenation of furan derivatives: The photocatalytic reactions were conducted in a 25 mL stainless steel reactor with a quartz window and a high-precision pressure gauge (0.4 %) (Figure S1). The reactant mixture consisted of 10 mL n-amyl alcohol, 4 mmol furan (or furan derivatives), and 80 mg 3wt% Pd/SiC catalyst. After purging the reactor by N₂ three times, 1.0 MPa of H₂ was refilled. Then the reactor was immersed in a temperature-controlled oil bath, and the temperature was kept at 25°C. The irradiation intensity of the Xe lamp was 0.15 W/cm², and the reaction time was 2.5 h. The dependence of the catalytic performance on the wavelength range of light was investigated by employing various low pass optical filters to block light below specific cut-off wavelengths while maintaining the light intensity to the reaction system unchanged. For instance, a filter with the cut-off wavelength of 450 nm can block the light with wavelengths shorter than 450 nm (the system is irradiated by the light with wavelengths between 450 and 800 nm). Similarly the light with wavelengths in the ranges of 530-800 and 600-800 nm were applied to the reaction system when using filters with cut-off wavelengths of 530 nm and 600 nm, respectively.



Scheme S1 Schematic diagram of the photocatalytic reactor for hydrogenation of furan and its derivatives.

Supplement results



Fig. S1 Catalytic performance of Pd/SiC for furan hydrogenation at different reaction times.

The furan conversion increases with the reaction time increasing. It can reach nearly 100% at 2.5 h and achieve equilibrium. Therefore, we employ 2.5 h as the reaction time in our case.



Fig. S2 TEM images (A, B) of the used 3wt% Pd/SiC catalyst after 5 rounds at 25 °C.

No obvious changes in morphology and aggregation of the Pd nanoparticles can be found, indicating that the Pd/SiC catalyst has excellent photocatalytic stability for hydrogenation of furan and its derivatives.