Electronic Supplementary Information (ESI)

Hydrogenation of TiO₂ nanosheets with exposed {001} facets for enhanced photocatalytc activity

Wang Wei, Ni Yaru, Lu Chunhua*, Xu Zhongzi

State Key Laboratory of Materials-Orient Chemical Engineering, College of Materials

Science and Engineering, Nanjing University of Technology, Nanjing 210009, PR

China

Corresponding author: lcming.com (Lu Chunhua)

Experimental details

Synthesis of {001) facets dominated TiO₂ nanosheets and hydrogenation treatment: All chemicals were used without further purification during the experiment process. The F doped $\{001\}$ facets dominated TiO₂ were prepared by a simple hydrothermal method in the light of previously described procedures.¹ Typically, 25 ml of $Ti(OBu)_4$ and 6 ml of HF were added into 9 ml deionized water by sonication and magnetic stirring to produce a homogeneous solution. Then the solution was transferred into a dried Teflon autoclave (60 ml) and kept at 180 °C for 24 h. The as-obtained white product was washed with deionized water and ethanol. Hydrogenation of the TiO₂ was performed using a professional hydrogenation furnace. The furnace was first evacuated and filled with pure H₂ to remove the air several times at room temperature, and then the furnace was filled with pure H₂ to a 10-bar pressure. TiO₂ was treated in the H₂ atmosphere at 400 $\,^{\circ}$ C for 2 h with a temperature ramp rate of 3 °C/min. Finally, the hydrogen treated TiO₂ (TiO₂-H) was allowed to cool to room temperature naturally.

(b) Characterization: The resulting products were measured on an ARL X'TRA powder X-ray diffraction (XRD) pattern employing Cu K_{α} radiation. The morphology and structure of as-prepared samples were conducted on a field emission scanning electron microscopy (FESEM) and a JEM-2010 high resolution transmission electron microscopy (HRTEM) with an accelerating voltage of 200 kV. UV-visible diffuse reflectance spectra were obtained using a 3101 spectrophotometer (Shimadzu) with

BaSO₄ as the reflectance sample. Electron paramagnetic resonance (EPR) spectra were recorded at 77 K on an EMX 10/12 spectrometer working in the X-band with 100 mg of the sample introduced into an EPR quartz probe cell. Surface composition and chemical environment of the products were analyzed by X-ray photoelectron spectroscopy (XPS) on a PHI5000 VersaProbe system with monochromatic Al K_{α} X-rays. Raman spectra were conducted on a Labram HR800 spectrometer with 514 nm laser as the excitation source under ambient conditions.

(C) Photocatalytic activity measurement: In a typical process, 20 mg photocatalyst was suspended in the aqueous solution of the MB or phenol $(10^{-5} \text{ M}, 25 \text{ ml})$, which was then placed in a 40 ml cylindrical quartz culture dish under the irradiation of a 500 W xenon lamp (fixed with an UV filter to obtain visible light). The xenon lamp was surrounded by the cooling water and was placed 15 cm above the reactor. Prior to the photocatalytic reaction, the suspension was kept in the dark with magnetic stirring for 60 min to establish the adsorption-desorption equilibrium. During the measurement process, 3 ml of the solution was collected at certain time intervals (10 min) to remove the suspended photocatalyst, and the resulting solution was analyzed with the UV-visible spectrophotometer by recording the absorption band maximum of MB or phenol.



Fig. S1 Enlarged (101) peak XRD pattern of TiO_2 and TiO_2 -H



Fig. S2 XPS valence band spectra of TiO_2 and $\text{TiO}_2\text{-}\text{H}$



Fig. S3 O 1s XPS spectra of TiO_2 and $TiO_2\text{-}H$



Fig. S4 F 1s XPS spectra of TiO_2 and TiO_2 -H



Fig. S5 Photocatalytic activity of P25, TiO₂, and TiO₂-H in decomposing MB under visible light irradiation



Fig. S6 Photocatalytic activity of P25, TiO_2 , and TiO_2 -H in decomposing phenol under UV-visible light irradiation



Fig. S7 Photocatalytic activity of P25, TiO_2 , and TiO_2 -H in decomposing phenol under visible light irradiation

References

1. X. Han, Q. Kuang, M. Jin, Z. Xie and L. Zheng, J. Am. Chem. Soc., 2009, 131,

3152.