Electronic Supplementary Information

Molecular and Textural Engineering of Conjugated Carbon Nitride Catalysts for Selective Oxidation of Alcohols with Visible Light

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Synthesis of photocatalysts

ATCN was prepared according to the reported.[1] For catalysts synthesis, 3 g DCDA was mixed with different amounts of ATCN an 12 nm SiO_2 in 15 mL water with stirring at 60 °C to remove water. The resultant solids were calcined at 550 °C for 4 h in air to obtain the final samples. The template was subsequently removed using NH_4HF_2 (4M), followed by filtered, washed with water and ethanol several times, and finally dried at 60 °C overnight. The samples thus obtained were denoted as MCN-$ATCN_x$, where x (0.01, 0.05 and 0.1) is the weight-in amount of ATCN. The pristine sample MCN was obtained following the same procedure just without ATCN.

Characterization

X-ray diffraction measurements were collected on a Bruker D8 Advance diffractometer with Cu Kα1 radiation (λ =1.5406 Å). Fourier transformed infrared (FTIR) spectra were recorded using a Nicolet Magna 670 FTIR spectrometer. Nitrogen adsorption-desorption isotherms were performed at 77 K using Micromeritics ASAP 2010 equipment. The UV/Vis diffuse reflectance spectra (DRS) were measured on a Varian Cary 500 Scan UV/Vis system. Photoluminescence (PL) spectra were recorded on an Edinburgh FI/FSTCSPC 920 spectrophotometer. Electron paramagnetic resonance (EPR) measurements were recorded using a Bruker model A300 spectrometer with a 300W Xe lamp equipped with an IR-cutoff filter (λ< 800 nm) and an UV-cutoff (λ > 420 nm) as visible light source. Transmission electron microscopy (TEM) was obtained using a JEOL model JEM 2010 EX instrument.

Photocatalytic test

The photocatalytic selective oxidation of various alcohols was performed as follows. The reaction was carried out at 1 bar pressure of oxygen, in a 10 mL Pyrex glass reactor fitted with a reflux condenser, oil bath, thermocouple, and magnetic stirrer. A typical reaction condition was 1.5 mL of benzotrifluoride (BTF), 0.1 mmol alcohol, 5 mg of catalysts; reaction temperature is 60 °C and reaction time is 3 h. To carry out the photochemical reaction, a 300 W Xenon lamp together with a 420 nm cut−off filter was used as a visible light source for the irradiation of reaction system. After the reaction, the mixture was centrifuged at 12000 rmp for 10 min to completely remove the catalyst particles. The remaining solution was analyzed with an Aglient Gas Chromatograph (GC-7820). The assignment of products was confirmed by a Hewlett-Packard Gas Chromatograph/Mass Spectrometer (HP-5973GC/MS).

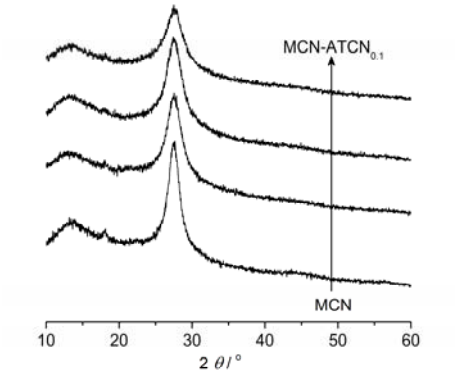

Fig. S1. XRD patterns for MCN and MACN

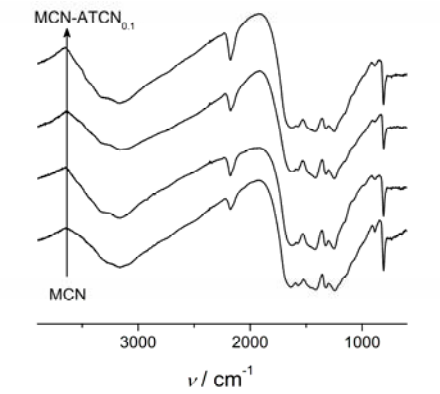

Fig. S2. FTIR patterns for MCN and MACN

Reference

[1] Y.H. Song, B.S. Jo, J. Heterocyclic Chem. 46 (2009) 1132.