Electronic Supplementary Material (ESI) for RSC Advances

Selective Oxidation of Benzene to Phenol by $FeCl_3$/mpg-C_3N_4 Hybrids

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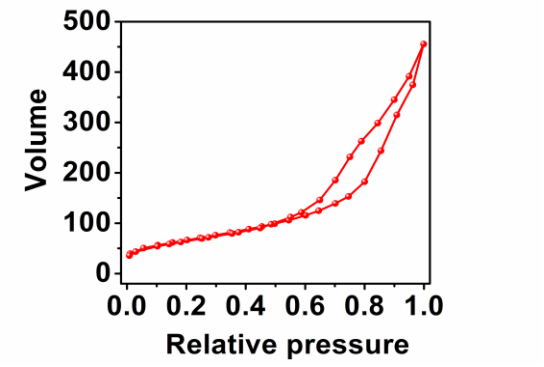

Figure S1. Nitrogen adsorption-desorption isotherms for as-synthesized mpg-C_3N_4.

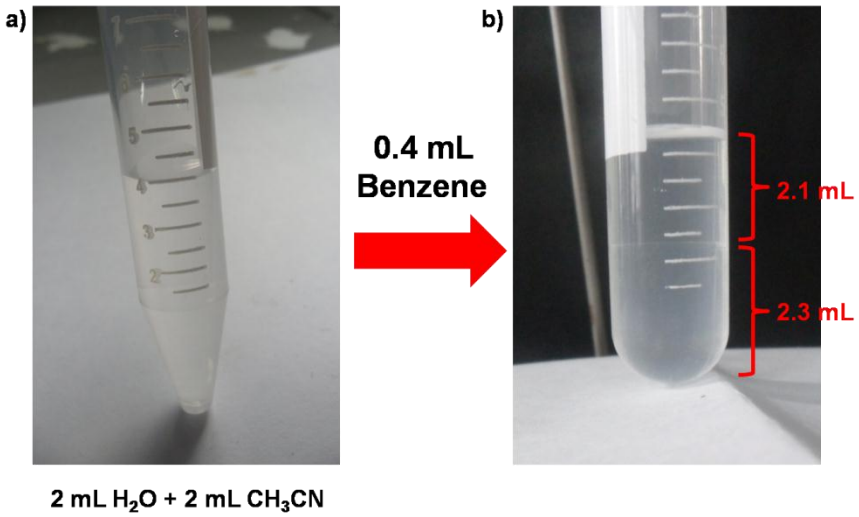

Figure S2. a) A mixture of water (2 mL) and acetonitrile (2 mL); b) After adding 0.4 mL benzene to the mixture above, a biphasic reaction system formed (Organic phase: ~2.1 mL; aqueous phase: ~2.3 mL). The oxidation of benzene was carried out in a biphase system.

Materials and Methods for Catalytic Tests

Unless otherwise stated, all solvents and chemicals were of analytical grade. They were commercially purchased and used without further treatment. A mercury lamp (100W) together with a 420 nm cut-off filter was used as a visible light source for the irradiation of reaction system. All GC experiments were carried out and recorded using a SHIMADZU GC-2010 with FID detector. The structure of products and by-products was identified using HP6890 GC/MS spectrometer by comparing retention times and fragmentation patterns with authentic samples. Electron spin resonance (ESR) signals were recorded at room temperature (298 K) with a Bruker ESR A300 spectrometer. 30 mL

Nitric acid (70 wt%, semiconductor grade) was used to dissolve the 5%$FeCl_3$/mpg-C_3N_4 (15 mg) at 80 ^oC for 12 h. The Fe content was then measured by Atomic Absorption Spectroscopy Analysis.

Catalyst Characterization

The X-ray photoelectron spectra were obtained with an ESCALAB MARK II spherical analyzer using a magnesium anode (Mg 1253.6 eV) X-ray source. The powdered samples were pressed to pellets and fixed to a stainless steel sample holder without further treatment. The XPS spectrum was shifted according to C1s peak being at 288.2 eV, so as to correct the charging effect. X-Ray powder diffraction (XRD) patterns were measured on a Bruker D8 diffractometer equipped with scintillation counter. BET surface area was obtained from 77 K N_2 adsorption-desorption isotherms using an ASAP 2010. Samples were outgassed at 150 ^oC for 20 h. Morphological observations of the catalysts were carried out with scanning electron microscope (LEO 1550). Optical absorption at the range of 300-800 nm was recorded with the T70 UV/VIS spectrometer (PG Instruments LTD).

Methods for the photocurrent tests

Photoelectrochemical experiments were performed in a conventional three-electrode cell (50 mL pyrex vial) with a platinum wire as the auxiliary electrode and an saturated calomel electrode as the reference electrode. The working electrode was IFO/$FeCl_3$@mpg-C_3N_4. All the tests were performed in 0.1M KOH solution. A 100 W mercury lamp (OSRAM, Germany) was used as light source with optical filter to get visible light (λ>420 nm) and positioned ~ 5 cm away from the photoelectrochemical cell.