

Mesoporous silica coating on hierarchical flowerlike Fe₂O₃ for catalytic activity enhancement to Fenton-like reaction

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1. Experimental Details

1.1 Materials and Reagents:

Ethylene glycol (EG), NH₃·H₂O (25 wt%), Tetrabutylammonium bromide (TBAB), Tetraethyl orthosilicate (TEOS), Ethanol, Methanol, Cetyltrimethylammonium bromide (CTAB), FeCl₃·6H₂O, H₂O₂, urea, and Methylene Blue were purchased from Beijing Chemical Reagent Co. All chemicals were used as received without further purification.

1.2 Preparation of flowerlike Fe₂O₃

Flowerlike Fe₂O₃ were synthesized according to the method developed in our group. In a typical process, 1.2 g FeCl₃·6H₂O, 2.7 g urea, and 7.2 g tetrabutylammonium bromide were added to 180 mL ethylene glycol in a 250 mL round flask and then the mixture was heated at 195 °C for 0.5 h. The green Fe₂O₃ precursor precipitate was

collected by centrifugation and washed with ethanol four times. The Fe_2O_3 precursor was calcined in air at 500 °C for 2 h to obtain crystalline Fe_2O_3 .

1.3 Preparation of Fe_2O_3 @meso- SiO_2 composite

Typically, 0.2 g Fe_2O_3 precursor was dispersed in 70 mL mixture solution of H_2O and EtOH (4:3, V/V). Then 0.14 g CTAB, 0.56 g $\text{NH}_3\cdot\text{H}_2\text{O}$ was added to the solution. Finally, 0.2 mL TEOS was added to the solution under stirring and reacted for 6 h. The precipitate was collected by centrifugation and washed with ethanol four times. The solid was calcined in air at 500 °C for 2 h to obtain Fe_2O_3 @meso- SiO_2 composite.

1.4 Characterization

The powder XRD pattern was recorded on Rigaku D/MAX-2500V (Cu $\text{K}\alpha$ radiation). Scanning electron microscopy (SEM) images were obtained on JEOL-6701F scanning electron microscope at 10.0 kV. Transmission electron microscopy (TEM) was carried out on a JEOL 1011F electron microscope running at 100 kV while high resolution (HR) TEM image and energy dispersive absorption X-ray (EDAX) spectroscopy were acquired from JEOL 2010F electron microscope with an energy dispersive X-ray system operated at 200 kV. The UV-Vis Spectra was conducted on Shimadzu UV-2700 and the photoluminescence spectra was obtained on Shimadzu RF-6000 at room temperature.

1.5 Measurement of catalytic activity

In a typical run, the reaction suspension was prepared by adding a given amount of catalyst with equivalent Fe_2O_3 (10.0 mg Fe_2O_3 or 14.0 mg Fe_2O_3 @meso- SiO_2) to a 50 mL beaker containing 20ml MB solution (50ppm). Prior to reaction, suspension was

sonicated for 5 min and magnetically stirred in the dark for 60 min to establish the adsorption/desorption equilibrium. Fenton-like reaction was initiated by adding a known concentration of H₂O₂ (0.6 mL, 30 wt %) to the solution. Samplings were taken at a given time intervals during the reaction. The sample was separated quickly by centrifugation and 150 μL of them was diluted to 3 mL for the further Uv-Vis detection. The catalyst was recovered from the solution by centrifugation and washed with water for 3 times for the next run of Fenton-like reaction. ·OH radicals concentration was determined by the terephthalic acid (TA) fluorescence method. The experimental procedure was similar to the measurement of Fenton catalytic activity except that the MB aqueous solution was replaced by terephthalic acid (5×10^{-4} mol L⁻¹) aqueous NaOH solution (2×10^{-3} mol L⁻¹). The photoluminescence spectra (PL) of TA samples were obtained using a fluorescence spectrophotometer at room temperature, and a wavelength excitation of 325 nm.

Supporting Figures

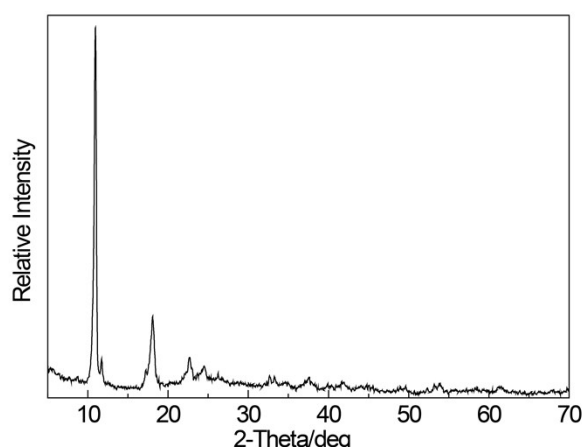


Figure S1 XRD pattern of the as-synthesized Fe₂O₃ precursor

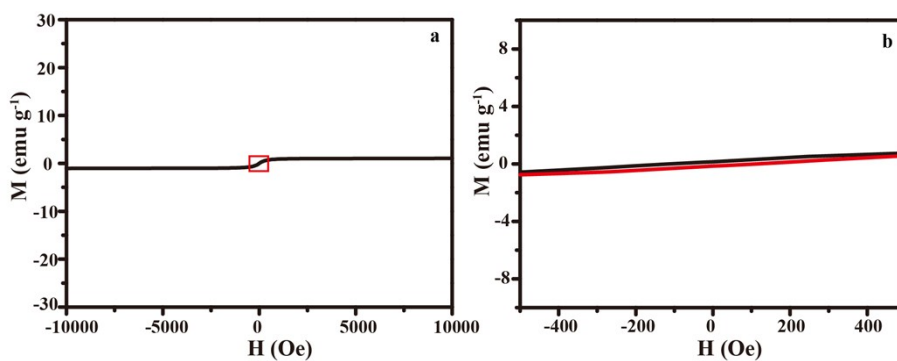


Figure S2. a) the room-temperature hysteresis curves of the flowerlike α -Fe₂O₃, b) is the enlarged view around H = 0 region.

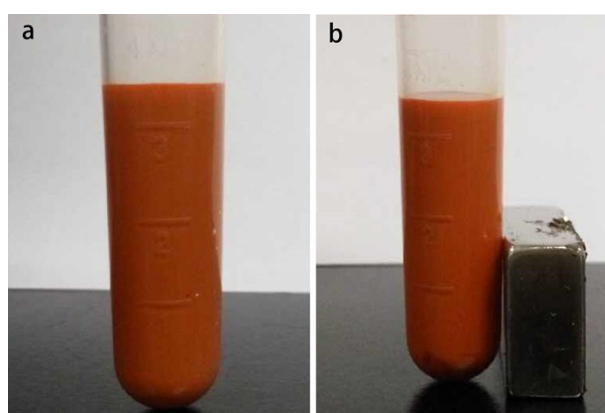


Figure S3. The digital photo of the Fe₂O₃@mesoporous silica composite catalyst after one cycle Fenton-like reaction (a) and then was put in the magnetic field (b).

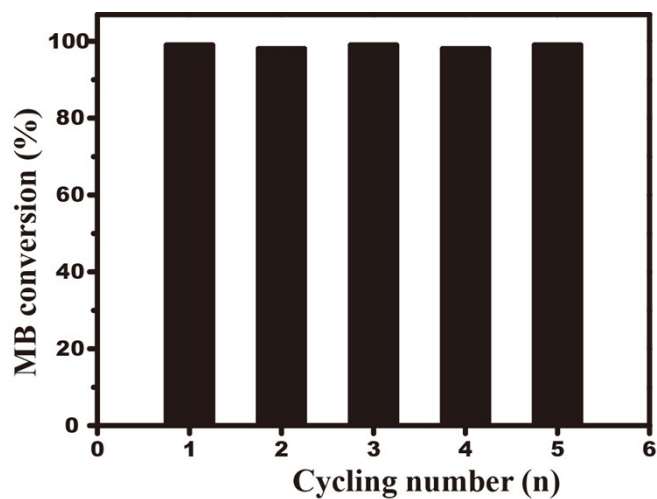


Figure S4. The cycling performance of flowerlike Fe₂O₃@mesoporous silica composite as catalyst for Fenton-like reaction to the degradation of MB

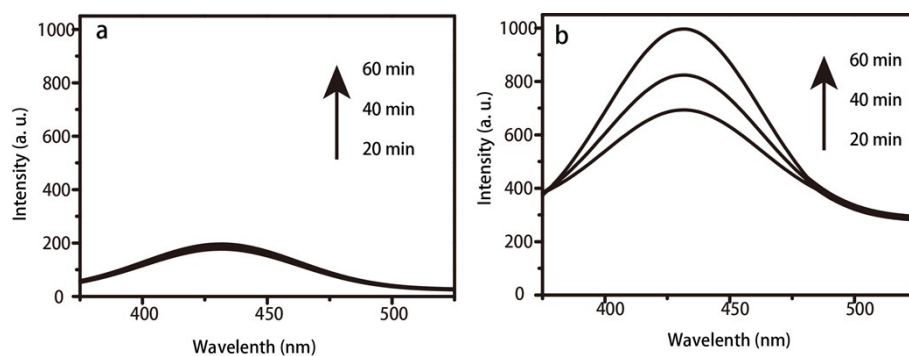


Figure S5. Total concentrations of $\cdot\text{OH}$ radicals formed as a function of time on flowerlike Fe_2O_3 (a) and $\text{Fe}_2\text{O}_3@\text{meso-SiO}_2$ composite (b), the reaction condition is similar with the degradation of MB except that the MB aqueous solution was replaced by $5 \times 10^{-4} \text{ mol L}^{-1}$ terephthalic acid aqueous solution with a concentration of $2 \times 10^{-3} \text{ mol L}^{-1}$ NaOH