Supporting Information

Large-scale Preparation of 3D Self-assembled Iron Hydroxide and Oxide Hierarchical Nanostructures and Their Applications for Water Treatment

Jinbo Fei[a], Yue Cui[a], Jie Zhao[a], Liang Gao[a],Yang Yang[b] and Junbai Li[*a,b]

[a]Beijing National Laboratory for Molecular Sciences (BNLMS), Institute of Chemistry, Chinese Academy of Sciences, Beijing, 100190, China.

[b]National Center for Nanoscicence and Technology, No. 11, Bei Yi Tiao, Zhong guan cun, Beijing, 100190, China.

*Address correspondence to jbli@iccas.ac.cn

Experimental Section

In a typical procedure, 2.78 g ferric sulfate ($FeSO_4 \cdot 6H_2O$) was dissolved in distilled water of 50 mL with the electronic resistance of 18.2 M$\Omega \cdot$cm. Ethanol saturated solution of deposited S of 50 mL was added into the solution above quickly. The mixture was under sonication at 70 °C for 60 min. After cooling, the as-synthesized iron oxide precursor was collected as a yellow precipitant by six centrifugation and ethanol washing cycles.

A Hitachi S-4300F scanning electron microscope (SEM) was used to investigate the morphologies of the precursors and final products. For transmission electron microscope (TEM) observation, the samples were re-dispersed in ethanol by ultrasonic treatment and dropped on carbon–copper grids. TEM images were collected by using a JEOL JEM 2010F microscope working at 200 kV. X-ray powder diffraction (XRD) measurements were carried out with a Rigaku D/max-2500 instrument using filtered CuKα radiation. X-ray photoelectron spectroscopy (XPS, VG ESCA-LAB 220i-XL) was performed to investigate the chemical bonding configurations of the sample. A Hitachi U-3010 spectrophotometer was used to record the UV/Vis

spectra of various samples. BET analysis (Micrometrics ASAP 2020) was employed to get the

Brunauer–Emmett–Teller (BET) specific surface areas of the samples through Nitrogen adsorption

isotherms. They were used directly without further disposal. A taper flask (capacity ca. 100 mL)

was used as the reactor vessel. 30 mg of the adsorbent sample was added to the system containing

Congo red ($C_{32}H_{22}N_6O_6S_2Na_2$, Amresco Inc.) solution (100 mg/L or 200 mg/L) with different

volumes under stirring. The adsorption isotherm was obtained by varying the initial Congo red

concentrations with the volume of 40 mL and stirring for 12 h at room temperature (20 °C).

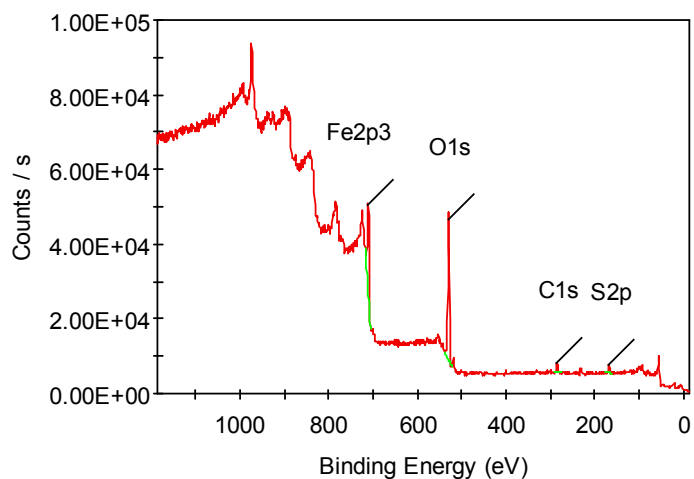UV-Vis absorption spectra were recorded at different intervals to monitor the process.
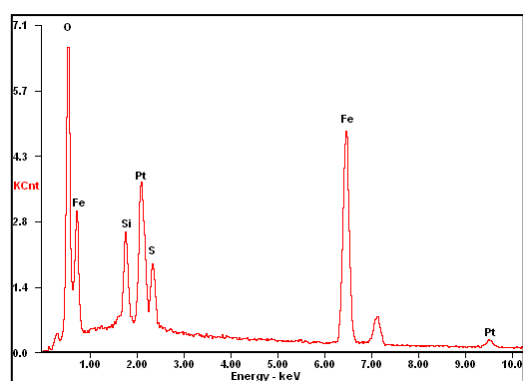
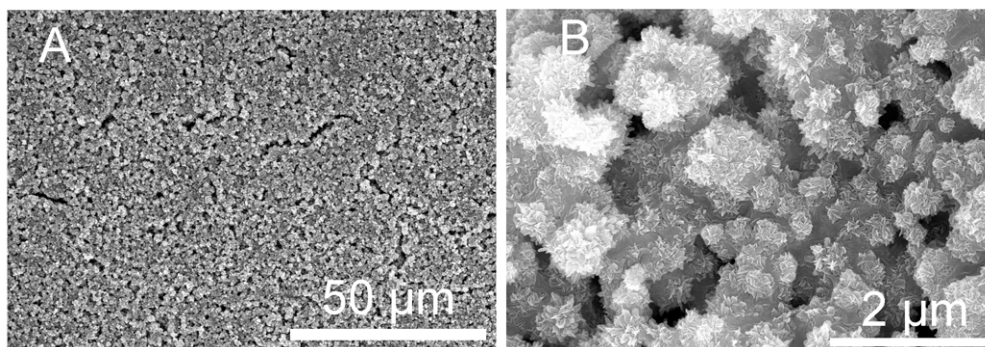| Element	Wt%	At%
OK	24.07	52.99
SiK	06.47	08.11
PtM	16.96	03.06
SK	05.82	06.40
FeK	46.68	29.44
Matrix	Correction	ZAF

Fig. S1 The EDX pattern and relative element analysis of the precursor.
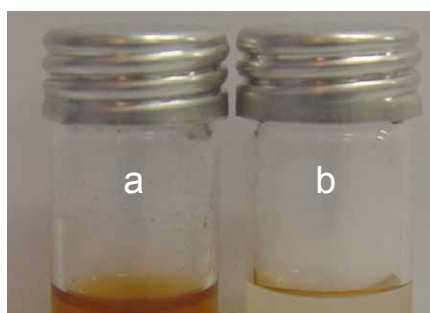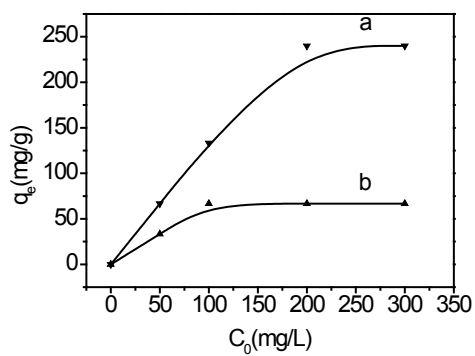

Fig. S2 The photo image of different precursors by using a) $FeCl_3$ and b) $FeSO_4$ as iron source.

Fig. S3 XPS pattern of the precursor.

Fig. S4 SEM images of iron hydroxide nanostructures with A) low magnification and B) higher magnification, using $(NH_4)_2Fe(SO_4)_2$ as iron source under the similar condition.

Fig.S5 Absorption isothermals of Congo red on the as-prepared (a) α-FeOOH and (b) α-Fe_2O_3.