Electronic Supporting Information

Synthesis of small Fe$_2$O$_3$ nanocubes and their enhanced water vapour adsorption-desorption properties

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Experimental Section:

All chemicals were of analytical reagent grade and used without further purification. In the typical procedure, 0.5 mmol of Fe(NO$_3$)$_3$·4H$_2$O was added to the mixed solvent of 18 ml of diethylene glycol (DEG) and 2 ml of H$_2$O with continuous stirring. 4 mmol of urea was added after the Fe(NO$_3$)$_3$·4H$_2$O was completely dissolved. After 10 min of stirring, the resulting reaction mixture was transferred into 25 mL of Teflon-lined autoclave. Finally, the autoclave was sealed and heated at 180 °C for 12h, and then cooled down to room temperature. The precipitate was separated by centrifugation, washed with distilled water and absolute ethanol several times. The product was dried in air at 60 °C for 2 h.

For comparison, we also prepared porous Fe$_2$O$_3$ nanofibers by an electrospinning method in combination with a calcination step. In a typical process, appropriate amounts of Fe(NO$_3$)$_3$ and PVP were dissolved in DMF solvent. The solution was also stirred for 10 h to obtain a homogeneous hybrid sol for electrospinning. The distance between the spinneret and collector was tuned to 15 cm, and the high-voltage supply was changed to 17 kV. The spinning rate was controlled at 0.3 mL·h$^{-1}$ by a syringe pump. The as-spun composite fibers obtained were calcined at 600 °C for 2 h in air at a heating rate of 1 °C min$^{-1}$ to yield dark red porous α-Fe$_2$O$_3$ nanofibers. The bulk counterpart Fe$_2$O$_3$ powder used in this work was purchased from chemicals without further treatment.
The raw diatomite used in this work was obtained from Changbai, China. In order to prepare a kind of diatomite having similar BET surface area of Fe$_2$O$_3$ nanocubes, the raw diatomite was heated to 600 °C with a ramping rate of 2 °C/min and kept at 600 °C for 2 h under air atmosphere. This sample was designated as calcined diatomite.

The X-ray diffraction analysis of the materials was performed on the Japan Rigaku X-ray diffractometer using Cu Kα radiation, with the operation voltage and electric current setting at 40 kV and 40 mA. Field-emission scanning electron microscopy (FESEM) images were obtained with a JEOL JEM-7100F microscope. Transmission electron microscopic (TEM) analyses and selected area electron diffraction (SAED) patterns were obtained by the JEOL JEM-2010 microscope. Specific surface areas were calculated using the Brunauer-Emmett-Teller (BET) model, and pore size distributions were evaluated from the desorption branches of the nitrogen isotherms using the Barrett-Joyner-Halenda (BJH) model.

The water vapour adsorption/desorption kinetics and isotherms of Fe$_2$O$_3$ and diatomite materials were performed on the Dynamic Water Vapor Sorption analyzer (Aquadyne DVS, Quantachrome Instruments). Before the experiment, approximately 50 mg of each sample was dried at 120°C overnight to completely remove the water on the surface of the materials. Adsorption/desorption isotherms were obtained on the basis of equilibrium adsorption capacity at different RHs from 0% to 90%. The adsorption/desorption kinetics was observed via surveying the product weight change with time at room temperature.
Fig. S1 SEM images and XRD patterns (inset) of (A) the bulk Fe$_2$O$_3$ powder and (B) porous Fe$_2$O$_3$ nanofibers.

Fig. S2 SEM images of the Fe$_2$O$_3$ samples with different amounts of urea: (A) 0, (B) 0.5, (C) 1 and (D) 4 mmol. In all cases, samples were prepared at 180 °C, and a reaction time of 12 h.
Fig. S3 SEM images of products prepared under similar conditions but with DEG replaced by (A) EG, (B) PEG 400.

Fig. S4 $N_2$ adsorption-desorption isotherm for the conventional raw diatomite and calcined diatomite samples. The inset shows BJH pore size distributions of the samples.
Fig. S5 Water sorption kinetics of the diatomite samples equilibrated for fixed times at room temperature: (A) raw diatomite and (B) calcined diatomite.