Supplementary Information

Prussian blue microcrystals prepared by selective etching and their conversion to mesoporous magnetic iron(III) oxides

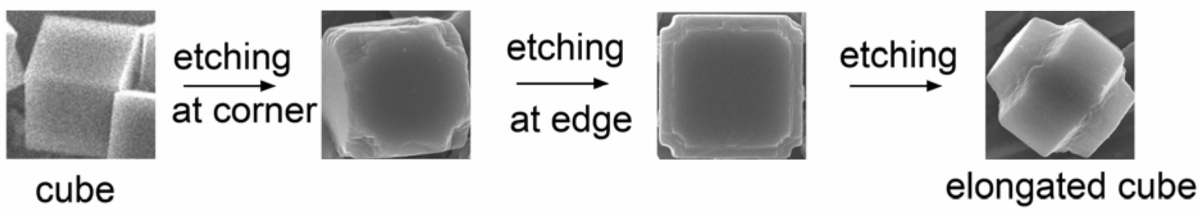
Ming Hu, Ji-Sen Jiang and Yi Zeng

Synthesis of PB: In a typical synthesis, 168.9 mg $K_4[Fe(CN)_6]$ and 291.6 mg cetyltrimethylammonium bromide (CTAB) were mixed into 13 mL distilled water under stirring, then 20 mL isopropanol and 7mL 36% hydrochloric acid were added into the present mixture in subsequence, finally the mixture became transparent. The transparent solution were transferred into a 50 mL Teflon-lined stainless-steel autoclave, sealed and maintained at 120 °C for 24 h. After the solution was cooled to room temperature, the obtained deep blue solid was collected by centrifugation, washed several times with water and ethanol, and then dried in a vacuum oven at 60 °C for 10 h.

Convert to iron oxide: Thermal treatment of PB was conducted using an electric muffle furnace in air at 450 ^{o}C for one 1h.

Characterization: The phase composition of samples was monitored by X-ray diffraction (XRD) using an X'Pert-Pro MPD diffractometer with Cu Kα radiation and conventional θ-2θ geometry. The transmission ^{57}Fe Mössbauer spectrum of 256 channels was collected using a Mössbauer spectrometer in a consultant acceleration mode with a ^{57}Co(Cr) source. Scanning electron microscopy (SEM) measurements were made on a JSM 6700F microscope and a JSM 6460 microscope. A JEOL JEM-2100F transmission electron microscope operating at 200 kV accelerating voltage was used for transmission electron microscopy (TEM) analysis. The porosity of sample was determined by N_2 adsorption using an ASAP-2000 surface area analyzer. The room-temperature magnetic hysteresis loop of the iron(III) oxides was measured by a vibrating sample magnetometer (HH-15, China).

Scheme S1. The illustration of selective etching process.



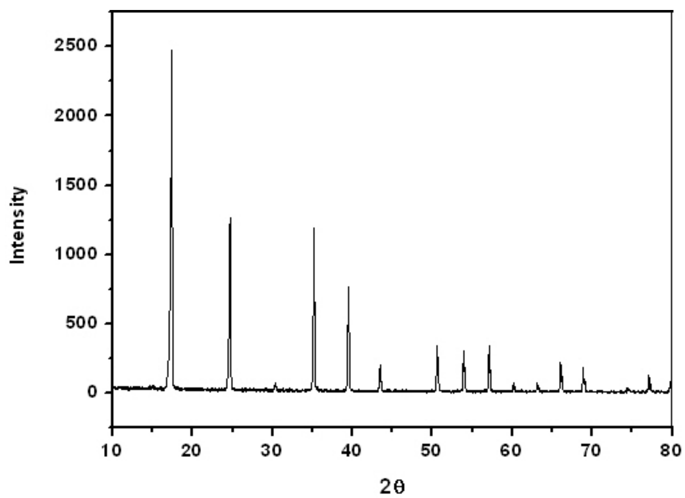


Fig. S1 XRD of sample prepared following a typical procedure.

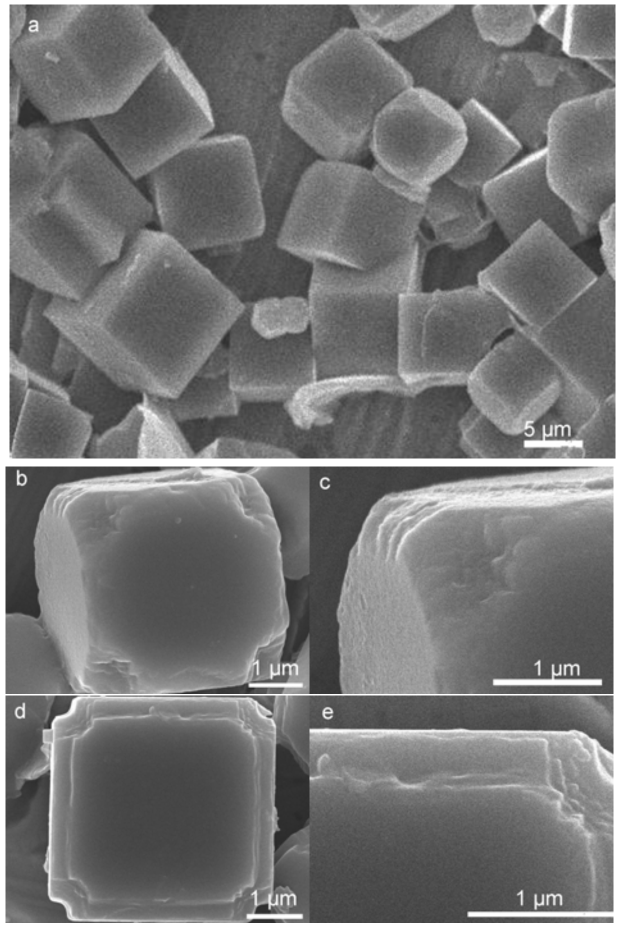


Fig. S2 SEM images of PB crystal colleted at early reaction stage of 2h. (a) An overview of all particles. (b) An individual crystal with slightly truncated corners. (c) An enlarged photo of one corner of the crystal shown in (b). (d) An individual crystal with truncated edges. (e) An enlarged photo of one edge of the crystal shown in (d).

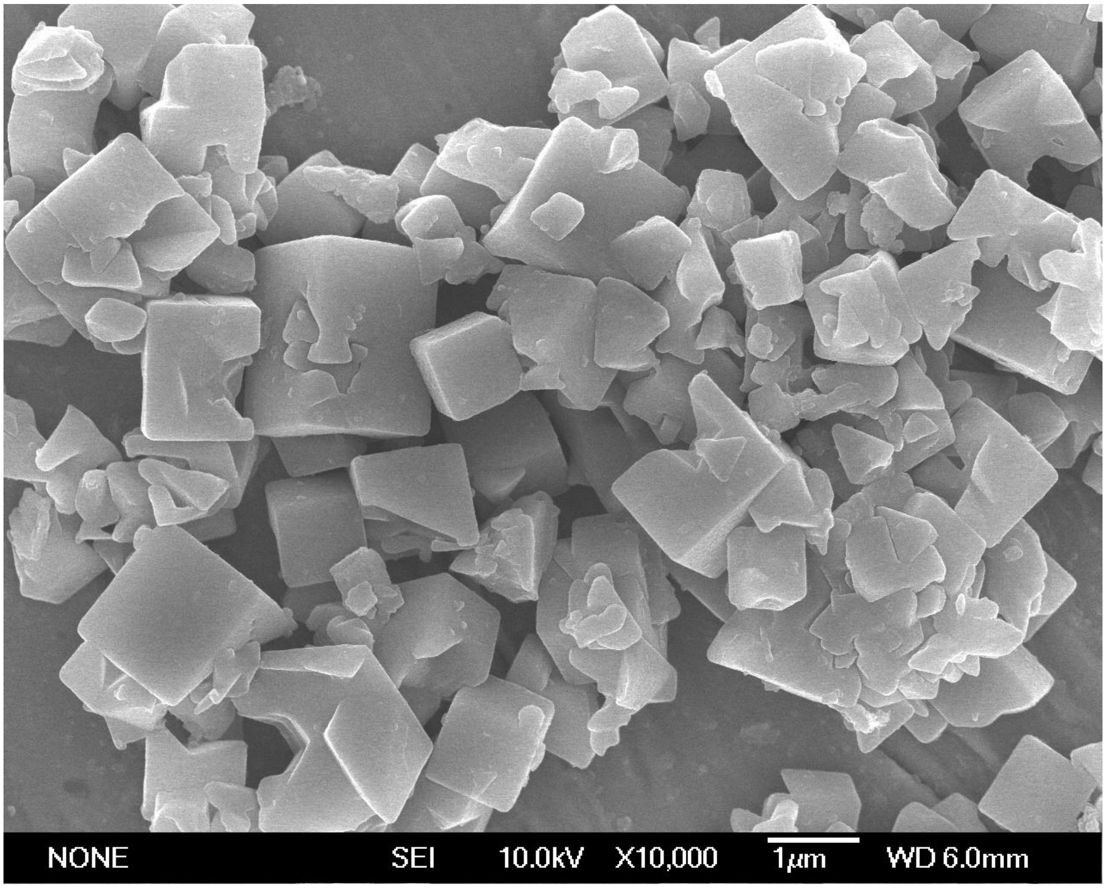


Fig. S3 SEM image of PB crystals obtained in the absence of CTAB.

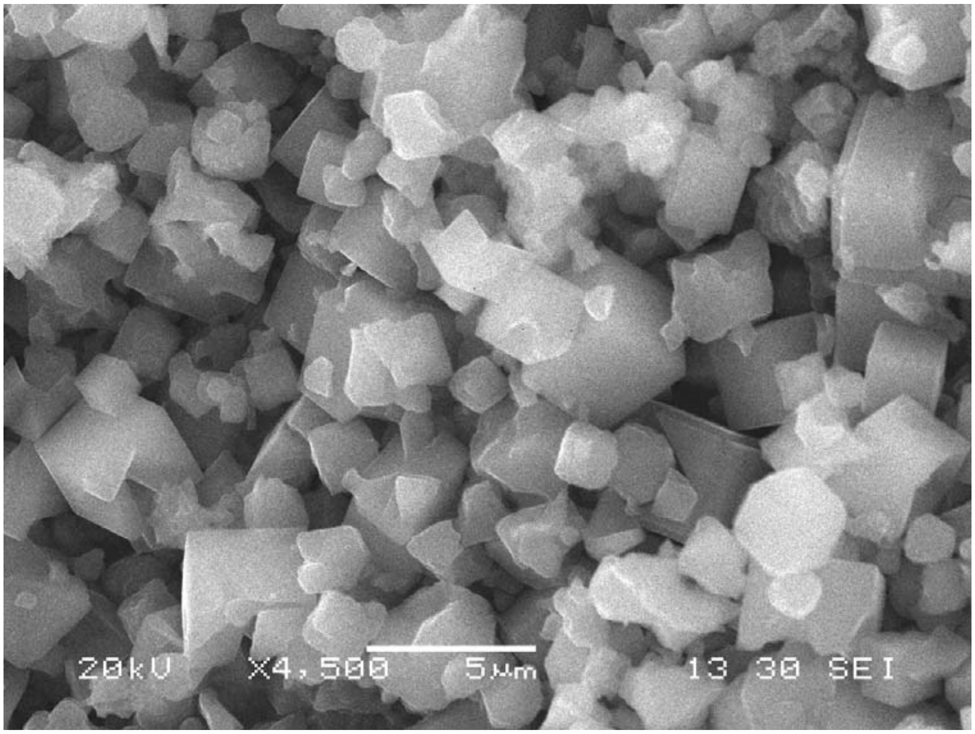


Fig. S4 SEM image of PB crystal when CTAB was replaced by TBAB.

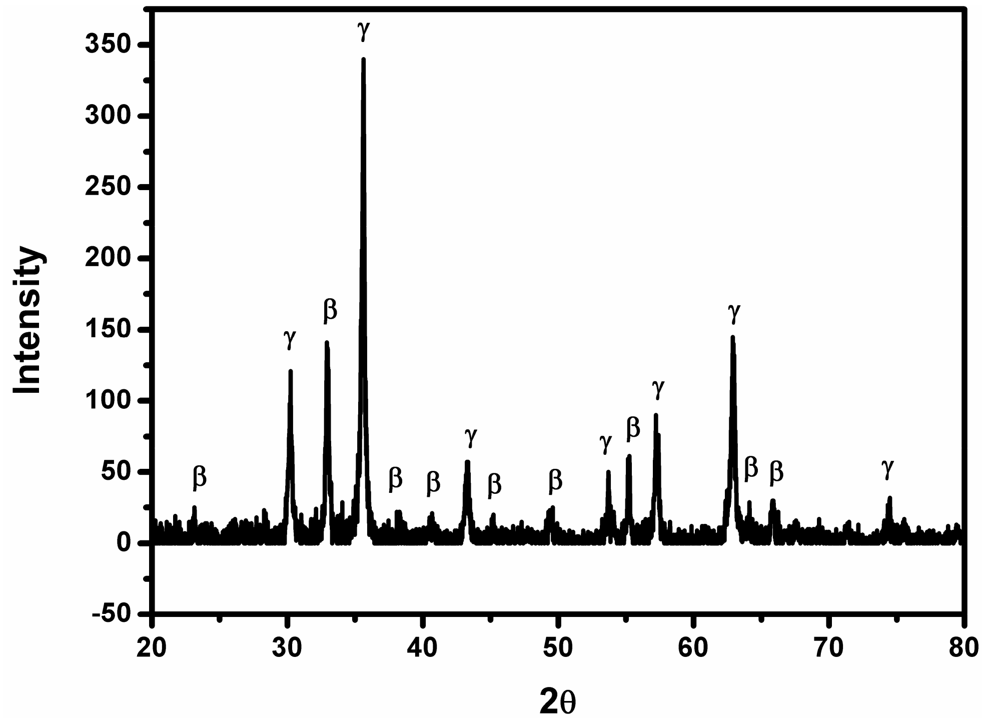


Fig. S5 XRD pattern of the thermal decomposition product of Prussian blue.

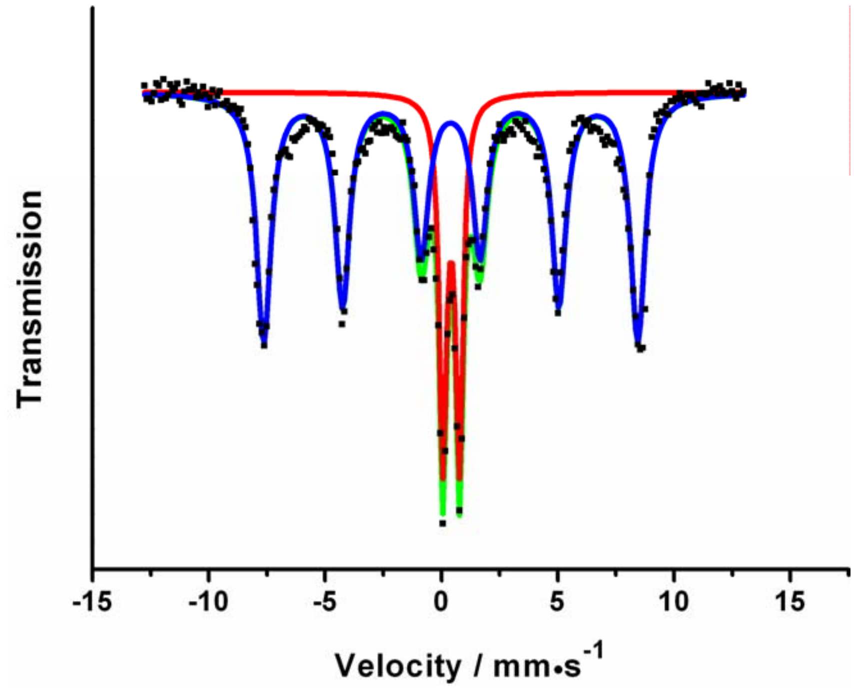


Fig. S6 Mössbauer spectrum of the thermal decomposition product of PB.

Table 1 Mössbauer parameters of iron(III) oxides obtained by thermal decomposition of PB crystals.

Fe component	Isomer shift (mm/s)	Quadrupole shift(mm/s)	Hyperfine field (T)	Area ratio (%)
$\beta-Fe_2O_3$ doublet	0.42 ± 0.01	0.71 ± 0.01		21 ± 0
$\gamma-Fe_2O_3$ sextet	0.40 ± 0.01	-0.01 ± 0.01	49.8 ± 0.1	79 ± 0