Supporting Information

Sonogenerated Metal-Hydrogen Sponges for Reactive Hard Templating

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Table S1 – Mössbauer spectra approximation of iron based magnetic particles (Isomer shifts IS refer to α-Fe)

Sample	Fraction	IS, ±0.01 mm/s	QS, ±0.02 mm/s	H_{eff}, ±0.2 T	Input A, ±2 %
Rods [1]	SPM γ-Fe_2O_3 $(Fe^{3+})_{internal}$	0.37	0.50	—	49
	SPM γ-Fe_2O_3 $(Fe^{3+})_{external}$	0.38	0.91	—	51
Dendritic structures [2]	SPM γ-Fe_2O_3 $(Fe^{3+})_{internal}$	0.35	0.51	—	34
	SPM γ-Fe_2O_3 $(Fe^{3+})_{external}$	0.36	0.83	—	22
	FM Fe_xO_y	0.44	0.01	37.1	27
	FM Fe_xO_y	0.40	-0.01	45.4	17

[1] EM characterization is presented in Fig. 2 (right part)
[2] EM characterization is presented in Fig. 2 (left part)

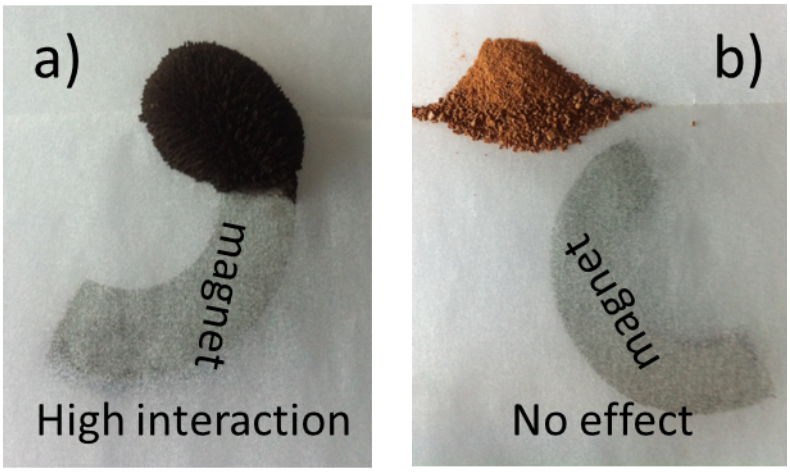

Fig. S1. Characterisation of magnetic behaviour and optical images of the samples of iron based materials formed by a) sono-RHT and d) Mg RHT without sonochemical irradiation.

Experimental Section

$FeCl_3$ $6H_2O$ (97% purity, Sigma Aldrich) $FeCl_2$ (98% purity, Sigma Aldrich) $Fe(NO_3)_3$ $9H_2O$ (99% purity, Sigma Aldrich), magnesium powder (325 mesh, 99,8%, Alfa Aesar), pyrrole (98%, reagent grade, Sigma Aldrich) were used as received. The water was purified prior to use in a three-stage Millipore Milli-Q Plus 185 purification system, and it exhibited a resistivity greater than 18.2 MΩm cm.

Ultrasonication. Ultrasonic treatment was carried out in a thermostated reactor with the VIP1000hd (Hielscher, Germany) operated at 20 kHz with a maximal electric output power of 1000 W.

Temperature programmed desorption (TPD) data were obtained by CATLAB microreactor (Hiden Analytical, United Kingdom). Amount of desorbed hydrogen and water were monitored with integrated into microreactor quadrupole mass spectrometer. The sample mass was typically 0.05g. Argon was used as a carrier gas with a flow rate of 40ml/min, while heating the sample in the range of 50°C to 500°C. To calculate desorption energy values, each sample were subject to set of different heating rates – 20,15,10, 5K / min.

Microscopy. Scanning electron microscopy (SEM) measurements were conducted with a Gemini Leo 1550 instrument at an operating voltage of 3 keV. Transmission electron microscopy (TEM) measurements were performed on a Zeiss EM 912 Omega (Carl Zeiss AG, Germany) transmission electron microscope operated at 300 kV and equipped with an electron-diffraction (ED) unit. TEM measurements were used to obtain information about the crystal structures and local orientations of the samples, which allowed different crystallographic phases to be distinguished. The unit cell and symmetry of an unknown phase that has been determined from the geometry of the diffraction pattern with the proper interpretation of the intensities of spots yields the positions of atoms in the crystal. High-resolution transmission electron microscopy (HRTEM) was performed by TEM in a Philips CM30 operated at 300 kV. The samples were ultramicrotomed (Leica EM FC6) and placed onto carbon-coated copper grids.

Mössbauer spectra were collected on ^{57}Fe with ^{57}Co/Rh (40 mCi). Magnetic susceptibility measurements were performed from 300–10 K with a Quantum Design MPMS-XL-5 SQUID magnetometer in the settle mode and between 5T and -5T in the hysteresis mode at different temperatures.