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Mesoporous Fe_3C sponges as magnetic supports and for catalysis

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Supporting information

XRD measurements were performed on a D8 Diffractometer from Bruker instruments equipped with Cu-Kα radiation, λ = 0.154 nm and a scintillation counter. Nitrogen sorption experiments were done with a Quantachrome Autosorb-1 or Quadrasorb at liquid nitrogen temperature, and data analysis were performed by Quantachrome software. The sample was degassed at 150 °C for 20 hours before measurements. Elemental analysis was done for Carbon and Nitrogen using a Vario EL Elementar and for Iron ICP-OES was done using a Vista-MPX CCD Simultaneous ICP-OES with radial plasma. TEM images were taken using a Zeiss EM 912 Ω operated at an acceleration voltage of 120 kV. A HR-TEM Philips CM 200 LaB6, operated at an acceleration voltage of 200 kV was also used. SEM images were performed on a LEO 1550-Gemini instrument. The samples were loaded on carbon coated stubs and coated by sputtering an Au/Pd alloy prior to imaging. IR spectrums were performed with a Varian 1000 FT-IR using KBr pellets. Raman measurements were made using a WiTec Confocal Raman Microscope alpha300 R, frequency doubled green 532 nm Nd/YAG laser with optical resolution diffraction limited to 200 nm laterally and 500 nm vertically, spectral resolution down to 0.02 wave numbers.

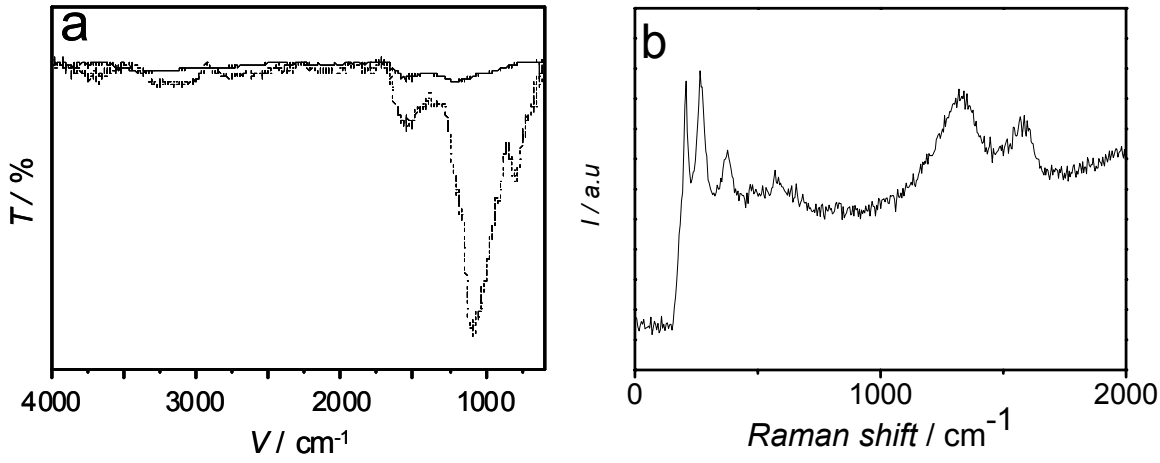

Fig. 1 Spectra of Fe_3C powder: a) IR, before (dashed line) and after (continuous line) silica removal; b) Raman after silica removal.

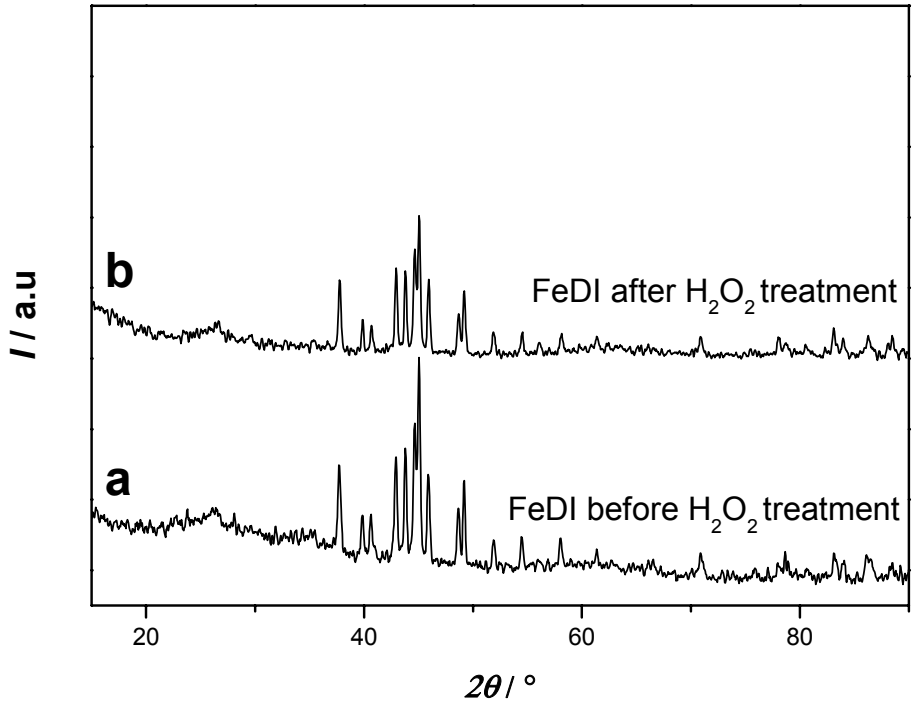

Fig. 2 XRD patterns of synthesized Fe_3C before and after treatment with H_2O_2 (see text). None significant structural modifications are shown.

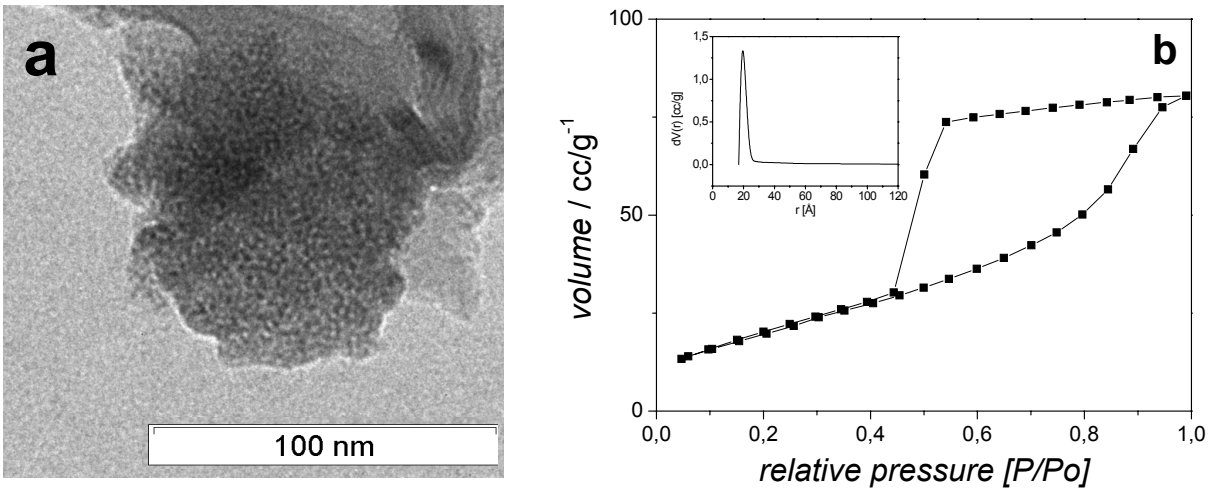

Fig. 3 a) TEM image and b) Nitrogen sorption and pore size distribution (small graph) of synthesized Fe_3C without the addition of silica particles. None mesoporous structure can be observed, rather a typical texture for very small, strongly interacting particles. The nitrogen sorption shows a surface area of 76 m^2g^{-1} and only pores around d = 4 nm (also observed in the templated structure).

Tab. 1 Elemental analyses and BET measurements

Sample name	**Expected product by XRD**	**Elemental analysis**			**surface area [m^2/g]**
		N [%]	*C [%]*	*Fe [%]*	
FeDI after template removal and before H_2O_2 treatment	Fe_3C	6.7	39.0	37.2	415
Fe-DI after H_2O_2 treatment	Fe_3C	5.2	39.5	35.4	--

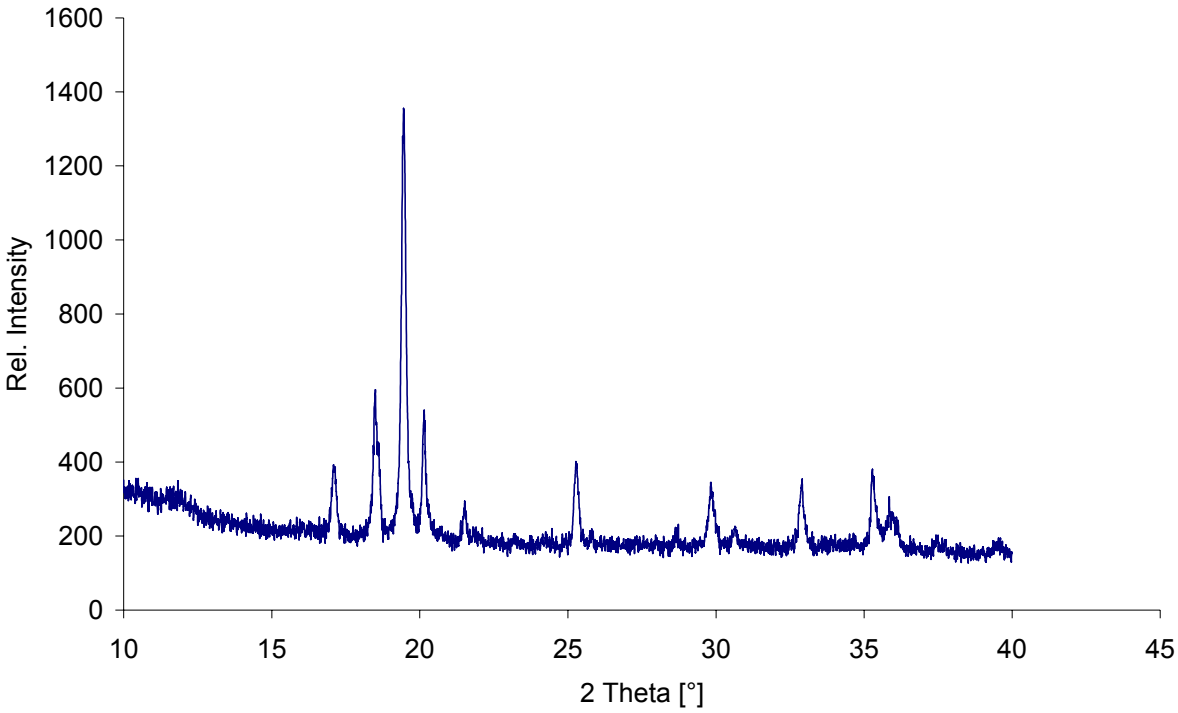

Fig. 4 XRD Diffraction Pattern of Fe_3C after catalysis (Measured with Mo Kα Radiation)