Supplementary Material (ESI) for Chemical Communications

Supporting information

for

Heating Rate Influence on the Synthesis of Iron Oxide Nanoparticles. The Case of Decanoic Acid

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Figure S1. Temperature diagram with the different heating rates used on the synthesis of iron oxide nanoparticles

Figure S2. (Left) Detailed TEM images of cubic-shaped nanoparticles with side length: (A) 45 nm, (B) 67 nm and (C) 100 nm. (Right) Histograms of the particle size distribution for the samples as shown in Figure 1.

Figure S33a and S3b. X-Ray diffraction pattern for different samples with average particle size indicated in the labels.

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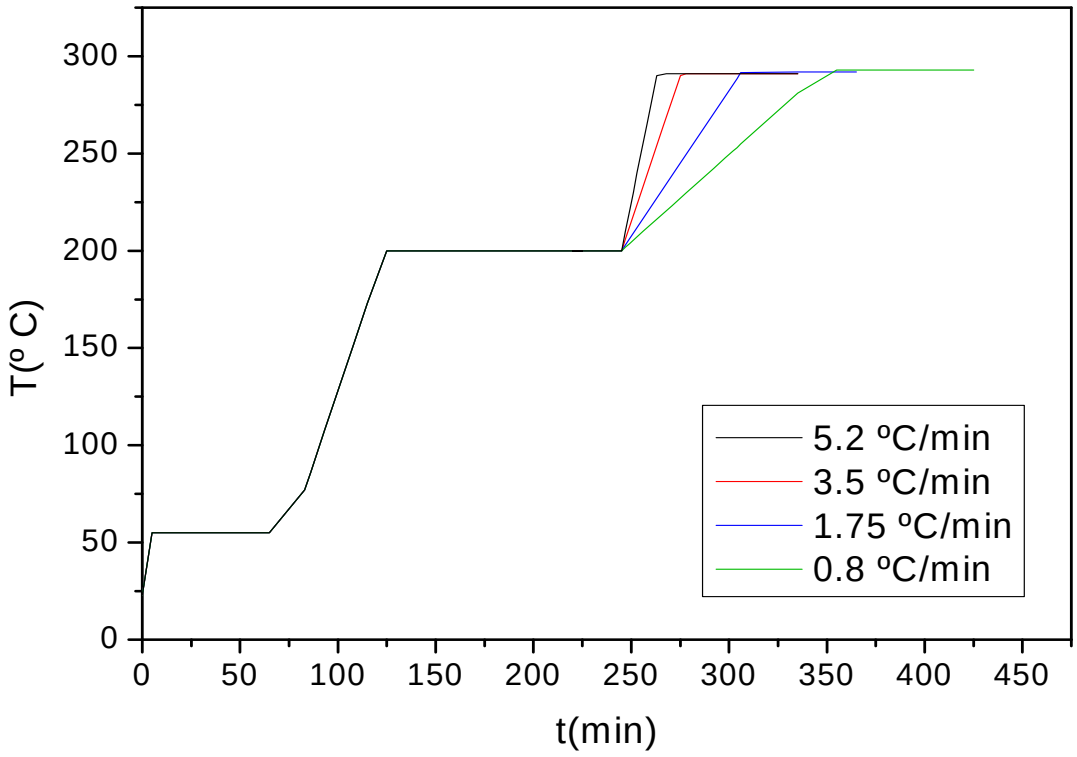
Figure S6.1. High-resolution narrow scan XPS spectra in the Fe2p region for 8 different levels during a depth-profiling experiment for 67 nm iron oxide nanoparticles. The spectra have been shifted for the sake of clarity. The numbers in the labels correspond to the sputtering time

Figure S6.2. High resolution XPS spectrum from a sample of 67 nm iron oxide nanoparticles in the Fe2p region as received a), and after sputtering during 2630 seconds b). The band was deconvoluted to identify the contributions from different oxidation states.

Figure S7. Low field detail of the magnetization hysteresis loops, showing the coercive fields and the remanent magnetization, at 5 K (empty symbols) and 300 K (full symbols) for 13 nm, 45 nm and 180 nm iron oxide nanoparticles.

Table S1. Evolution of the iron content (in the oxidized and Fe^0 states) during the etching process, for a sample of 67 nm iron oxide nanoparticles, obtained from the XPS spectra in Figures S6.1 and S6.2.

Table S2. Saturation magnetization, M_s, for the 13, 45, 67, 100, 124 and 180 nm iron oxide nanoparticles.



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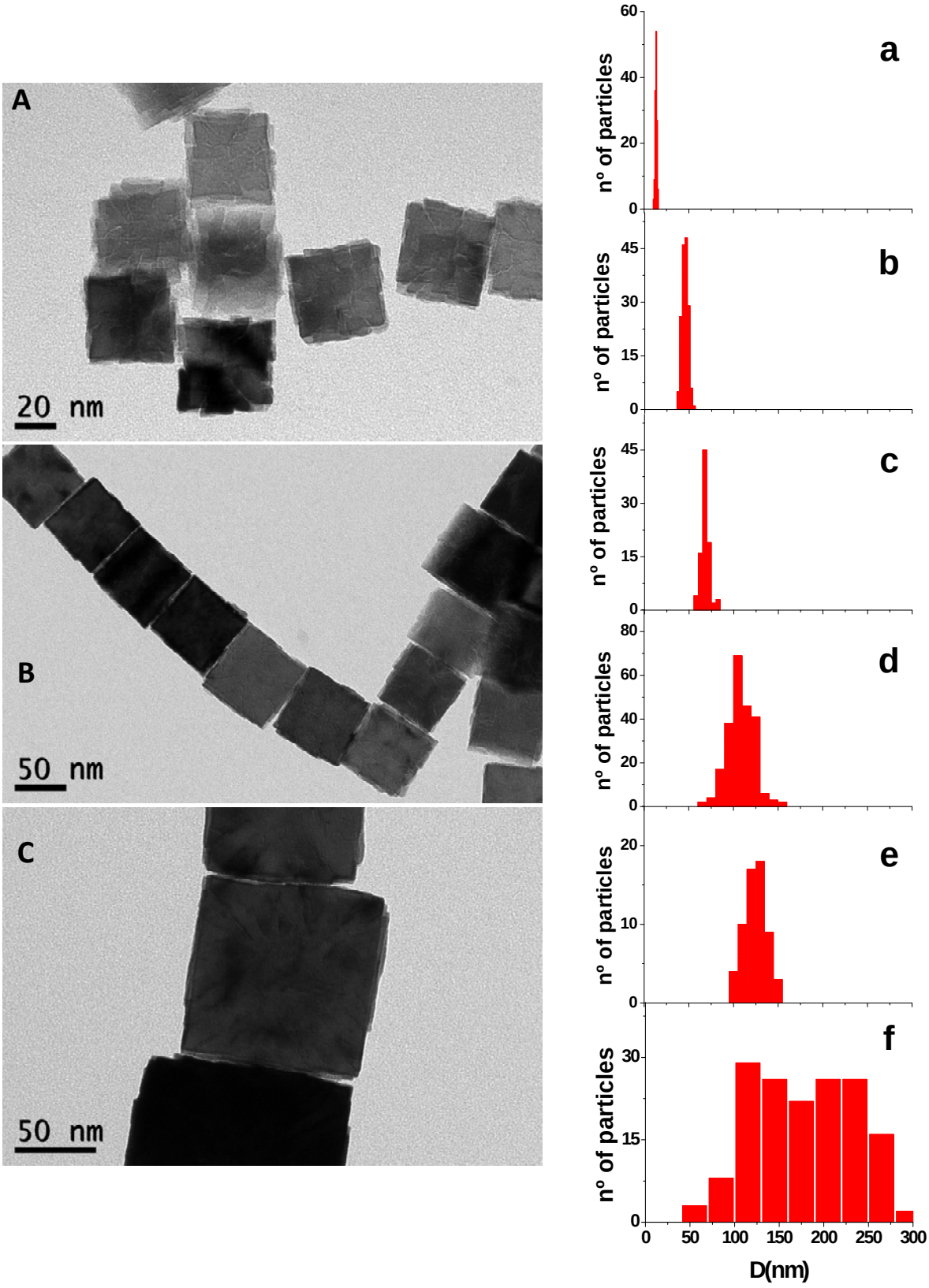

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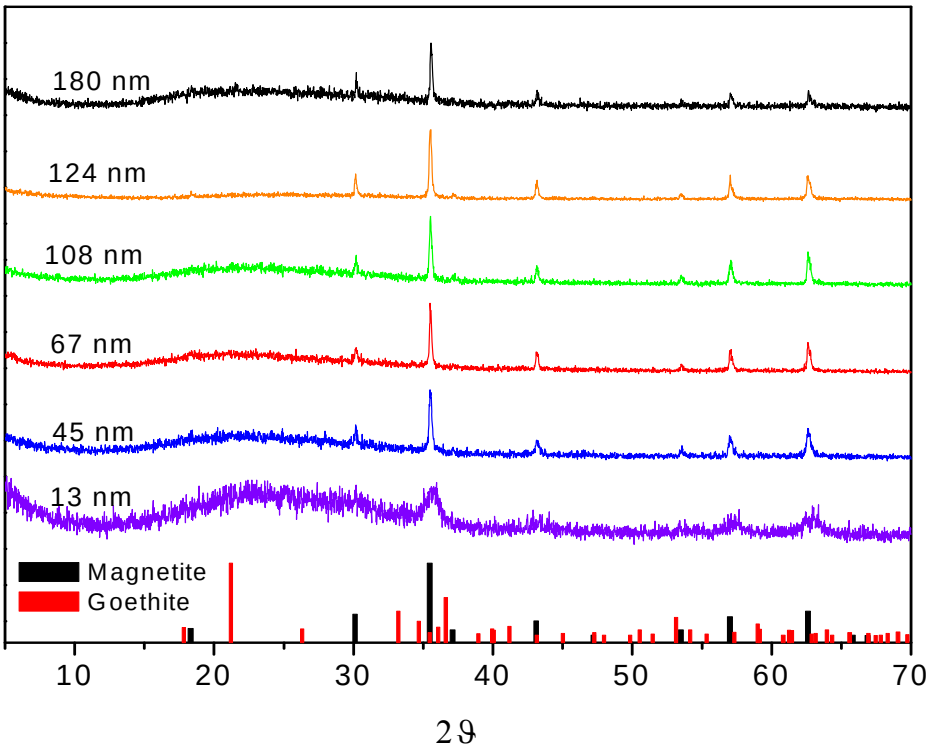


Figure S3a. X-Ray diffraction pattern for different samples (with average particle size indicated in the labels) compared to magnetite (PDF#00-019-0629) - black bars - and goethite - red bars - (PDF#01-081-0462).

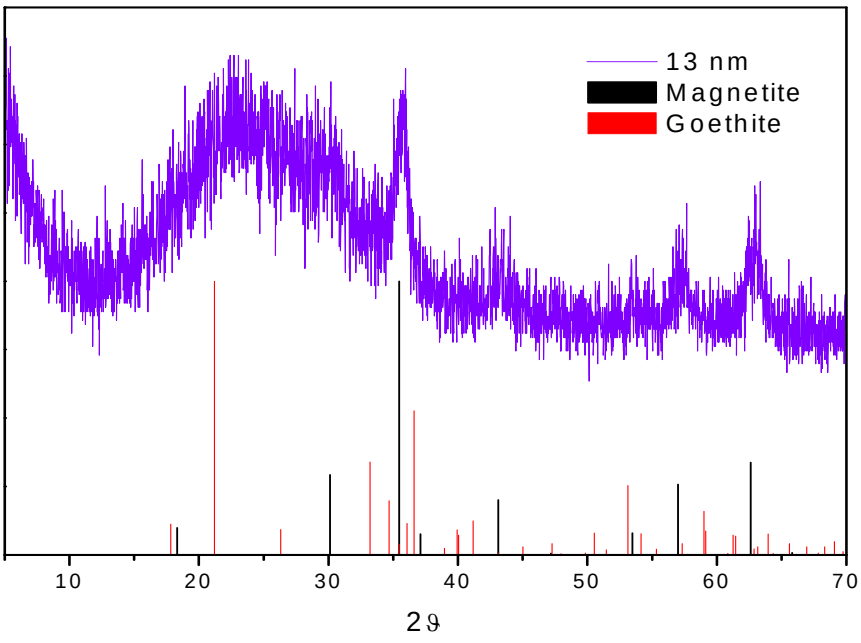


Figure S3b. XRD pattern for the 13 nm nanoparticles. A glass peak at low angles can be observed.

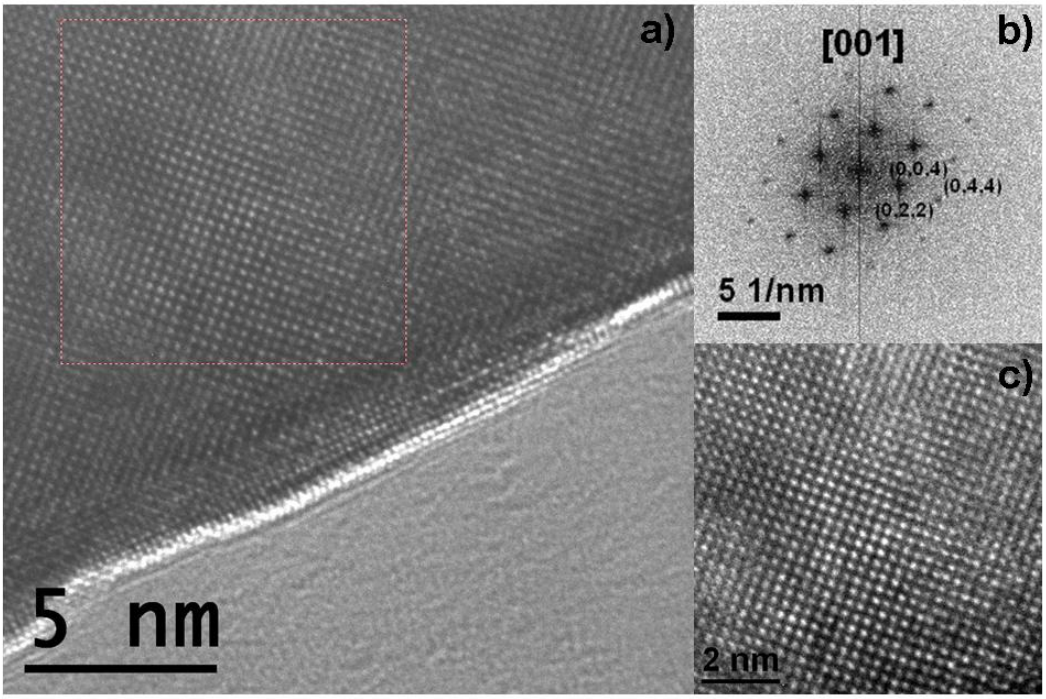


Figure S4. a) HRTEM image of a 67 nm iron oxide nanoparticle, b) magnification of the selected area framed in a), and c) Fourier transform of the latter, where the zone axis is the [001].

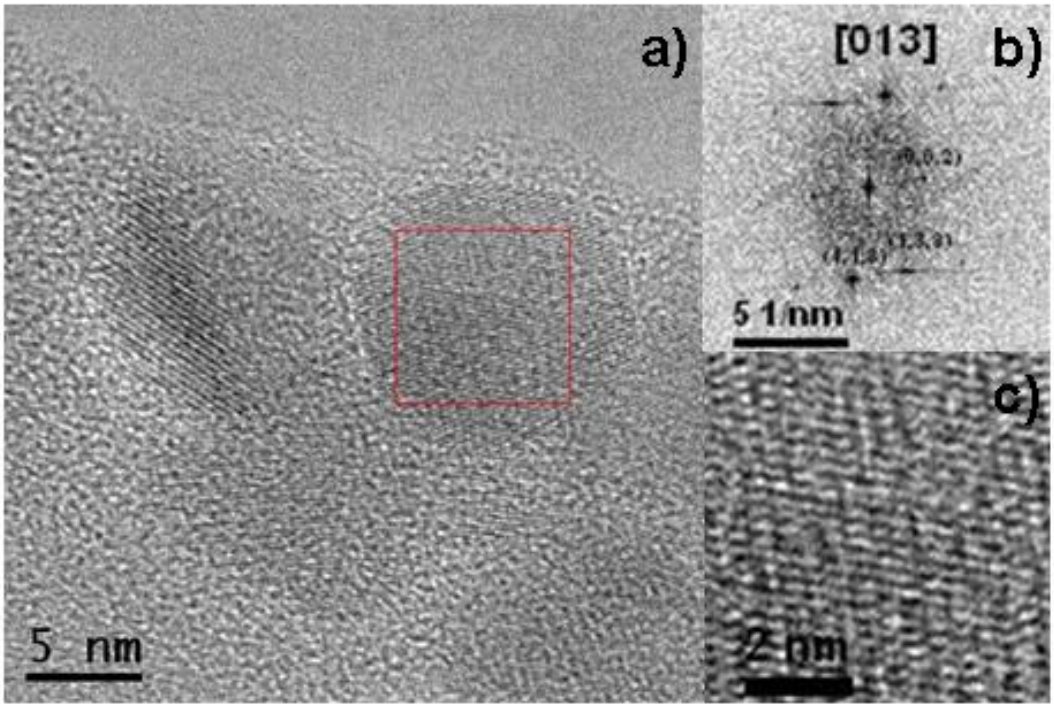
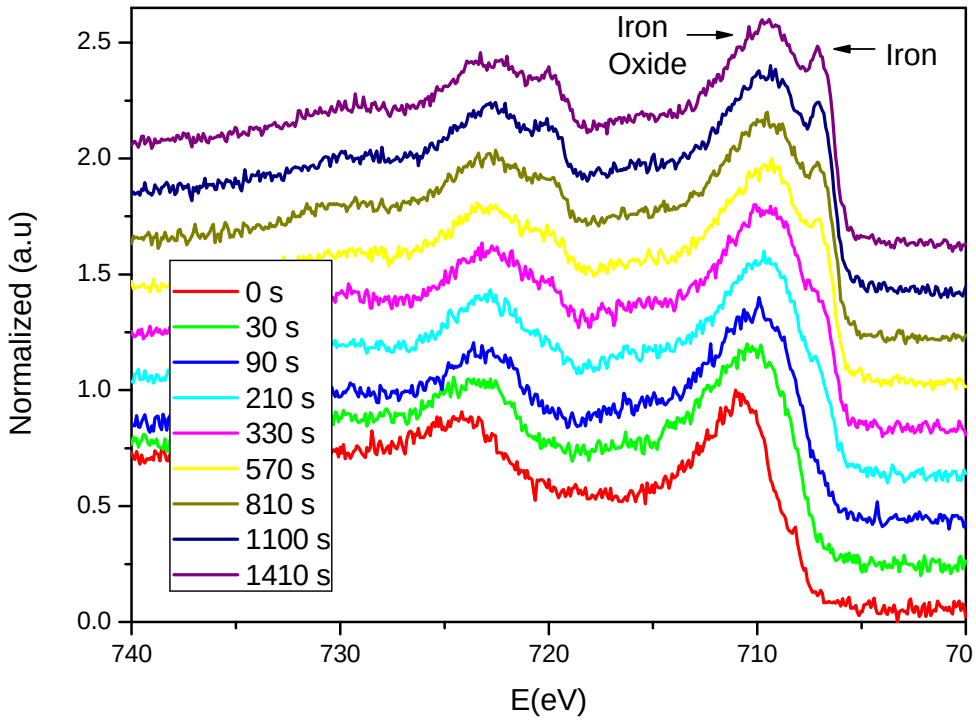


Figure S5. a) HRTEM image of a 13 nm iron oxide nanoparticle, b) magnification of the selected area framed in a), and c) Fourier transform of the latter, where the zone axis is the [013].



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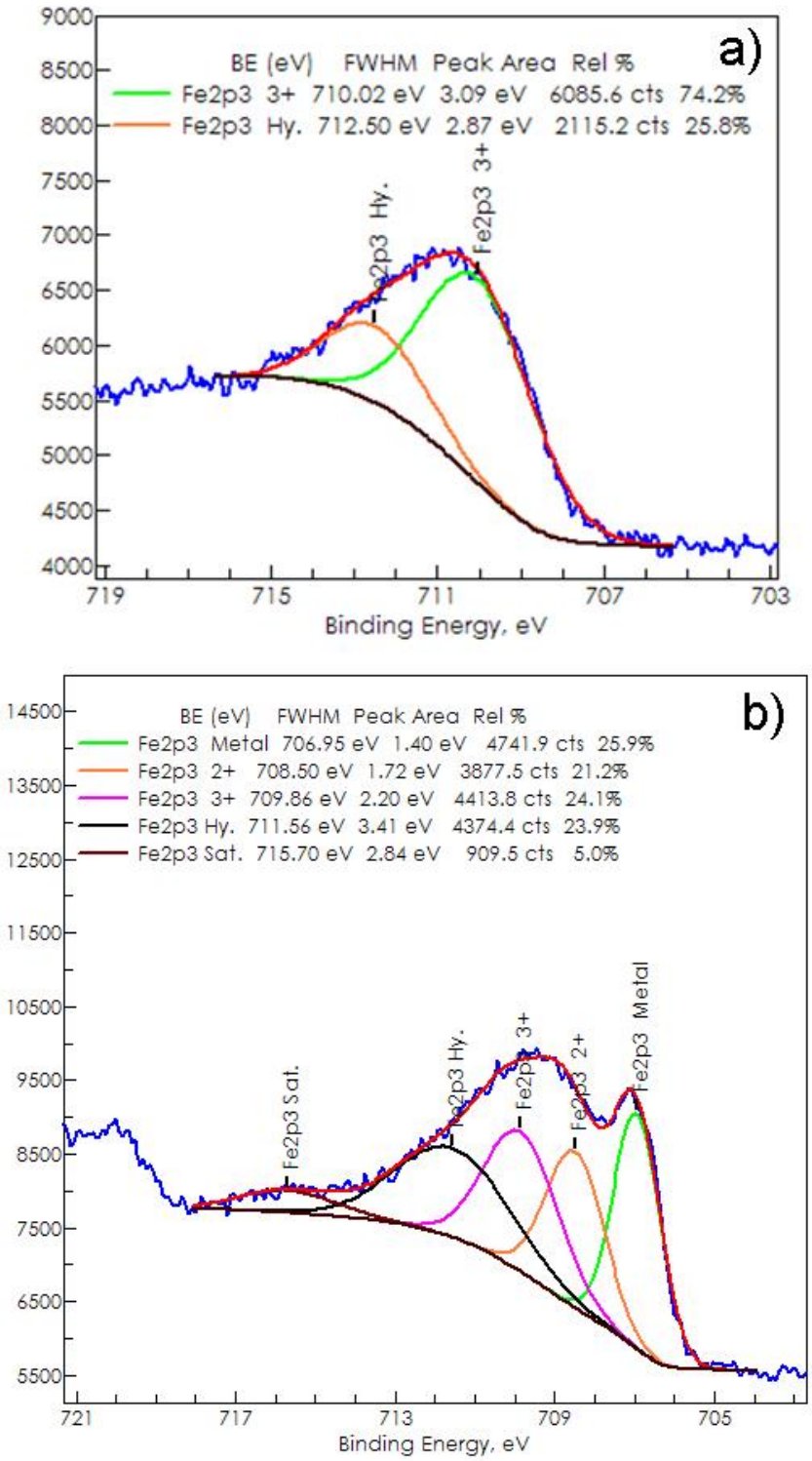

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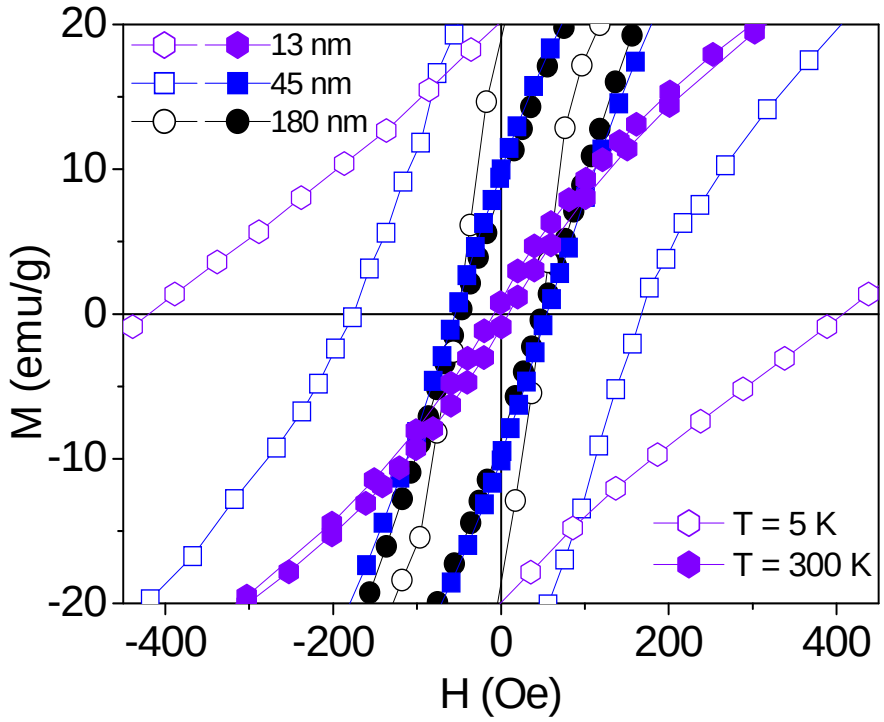

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	Level 1 30 s	**Level 2 90 s**	**Level 3 210 s**	**Level 4 330 s**	**Level 5 570 s**	**Level 6 1110 s**	**Level 7 1410 s**
Fe Metal (%)	0	2.11	6.29	6.70	12.56	15.44	16.06
Fe Oxide (%)	100	97.89	93.71	93.30	87.44	84.56	83.94

Table S2. Saturation magnetization, M_s, for the 13, 45, 67, 100, 124 and 180 nm iron oxide nanoparticles.

Heating rate (ºC/min)	**Size (nm)**	**M_S(5 K) / (emu/g)**	**M_S(300 K) / (emu/g)**
5.2	13 ± 1	63.8 ± 0.2	54.7 ±0.5
3.5	45 ± 4	97.6 ± 0.2	89.9 ±0.3
2.6	67 ± 7	97.8 ± 0.2	89.0 ±0.3
1.75	100 ± 13	103.2 ± 0.4	92.8 ±1
1.2	124 ± 12	94.0 ± 0.4	86.0 ± 0.4
0.8	180 ± 56	89.8 ± 0.2	81.9 ± 0.3