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Controlled Synthesis of Magnetic Iron Oxide Nanoparticles: Magnetite or Maghemite?

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Table S1. Parameter range according to the design of experiments with 6 parameters.

Sample	A	B	C	D	E	F
1	0	1	0.168	0.72	0.06	-0.169
2	0	1	-0.8	1	1	-1
3	0.5	0.5	-1	-1	-1	1
4	0.484	0.516	1	0	-1	1
5	0.488	0.512	-1	0.8	1	1
6	1	0	-0.05	1	1	-1
7	0.5	0.5	-0.8	1	1	-1
8	0	1	-1	1	-1	-0.03
9	0.494	0.506	1	-0.8	1	-1
10	0.489	0.511	-1	1	-1	0
11	0.505	0.496	-1	-1	1	-0.8
12	0	1	-1	-1	1	-0.84
13	0.503	0.497	0	-1	-1	-1
14	0.25	0.75	-0.238	-0.247	0.13	1
15	1	0	1	0.05	-1	-1
16	0.98	0.02	-1	1	-1	-1
17	1	0	1	0.8	1	1
18	1	0	-1	1	0.76	1
19	1	0	-0.8	-1	1	1
20	0.495	0.505	0.82	-1	1	1
21	0	1	1	0.05	-1	1
22	1	0	0.75	1	-1	1
23	0	1	1	1	1	0.8
24	0	1	-1	-1	-1	1
25	1	0	1	-1	-0.84	1
26	0.231	0.769	1	-0.96	-0.225	-0.02
27	0.498	0.502	1	1	1	0.8
28	0.484	0.516	1	0	-1	1
29	0.508	0.492	1	1	-1	-1
30	0	1	0.04	-1	-1	-1
31	0	1	-1	0.8	1	1
32	1	0	-1	-0.85	-1	1

33	0	1	1	-0.8	1	-1
34	0.498	0.502	1	1	1	0.8
35	0	1	0.81	-1	1	1
36	1	0	-1	-1	-0.02	-1
37	1	0	0.32	-0.06	0.232	-0.09
38	0.114	0.886	-1	-0.03	-0.264	-1
39	0.489	0.511	-1	1	-1	0
40	0	1	1	1	-1	-1
41	1	0	1	-1	1	-1
42	0.503	0.497	0	-1	-1	-1
43	0.5	0.5	-0.8	1	1	-1

Table S2. Absolute values of parameters implemented in the DoE model for all experiments.

Sample	A1: Iron(II)-chloride in g	A2: Iron(III)-chloride in g	B: Sodium hydroxide in g	C: Temperature in °C	D: Stirring rate in rpm	E: Dosing rate in minutes	F: Reaction time in minutes
1	8.64	3.50	14.40	59	860	32	50
2	8.64	3.50	14.40	35	1000	60	0
3	12.93	5.24	10.82	30	0	0	120
4	12.82	5.19	10.91	80	500	0	120
5	12.86	5.21	10.89	30	900	60	120
6	17.28	7.00	7.20	54	1000	60	0
7	12.96	5.25	10.80	35	1000	60	0
8	8.64	3.50	14.40	30	1000	0	58
9	12.91	5.23	10.85	80	100	60	0
10	12.87	5.21	10.88	30	1000	0	60
11	13.00	5.27	10.77	30	0	60	12
12	8.64	3.50	14.40	30	0	60	10
13	12.99	5.26	10.78	55	0	0	0
14	10.80	4.38	12.60	49	376	34	120
15	17.28	7.00	7.20	80	525	0	0
16	17.11	6.93	7.34	30	1000	0	0
17	17.28	7.00	7.20	80	900	60	120
18	17.28	7.00	7.20	30	1000	53	120
19	17.28	7.00	7.20	35	0	60	120
20	12.92	5.23	10.83	76	0	60	120
21	8.64	3.50	14.40	80	525	0	120
22	17.28	7.00	7.20	74	1000	0	120
23	8.64	3.50	14.40	80	1000	60	108
24	8.64	3.50	14.40	30	0	0	120
25	17.28	7.00	7.20	80	0	5	120
26	10.64	4.31	12.74	80	20	23	59

27	12.94	5.24	10.81	80	1000	60	108
28	12.82	5.19	10.91	80	500	0	120
29	13.03	5.28	10.74	80	1000	0	0
30	8.64	3.50	14.40	56	0	0	0
31	8.64	3.50	14.40	30	900	60	120
32	17.28	7.00	7.20	30	75	0	120
33	8.64	3.50	14.40	80	100	60	0
34	12.94	5.24	10.81	80	1000	60	108
35	8.64	3.50	14.40	75	0	60	120
36	17.28	7.00	7.20	30	0	29	0
37	17.28	7.00	7.20	63	470	37	55
38	9.63	3.90	13.58	30	485	22	0
39	12.87	5.21	10.88	30	1000	0	60
40	8.64	3.50	14.40	80	1000	0	0
41	17.28	7.00	7.20	80	0	60	0
42	12.99	5.26	10.78	55	0	0	0
43	12.96	5.25	10.80	35	1000	60	0

Table 3S. XRD results for all samples obtained from 311 reflections and TEM diameters.

Sample	FWHM (311)	2θ in ° (311)	Scherrer diameter (nm)	TEM diameter (nm)	σTEM (nm)	Lattice constant a (Å)
1	0.47	16.16	7.79	8.00	1.30	8.37
2	0.41	16.085	8.83	9.04	1.99	8.41
3	0.31	16.13	11.93	12.43	2.13	8.38
4	0.35	16.13	11.27	11.24	1.64	8.38
5	0.59	16.145	6.19	6.59	1.13	8.38
6	0.43	16.13	8.49	8.98	1.33	8.38
7	0.37	16.04	9.97	10.40	1.36	8.43
8	0.41	16.1	8.82	9.55	1.26	8.40
9	0.31	16.085	11.65	11.84	1.71	8.41
10	0.37	16.085	9.75	10.21	1.76	8.41
11	0.36	16.115	10.29	10.93	1.77	8.39
12	0.38	16.115	9.72	10.60	2.02	8.39
13	0.33	16.1	11.1	11.11	1.31	8.40
14	0.40	16.13	9.1	10.16	1.10	8.38
15	0.49	16.115	7.43	8.099	1.43	8.39
16	0.70	16.055	5.22	6.068	0.85	8.42
17	0.36	16.085	10.16	10.12	1.52	8.41
18	0.42	16.175	8.74	8.89	1.14	8.36
19	0.40	16.07	9.24	10.08	1.28	8.42
20	0.27	16.1	13.55	14.52	1.82	8.40
21	0.39	16.085	9.31	9.97	1.20	8.41
22	0.56	16.1	6.57	7.29	1.39	8.40
23	0.51	16.145	7.2	7.53	1.26	8.38
24	0.36	16.1	10.17	10.73	1.16	8.40
25	0.30	16.175	12.34	12.8	2.61	8.36
26	0.27	16.1	13.51	14.3	2.10	8.40
27	0.39	16.085	9.3	10.02	1.15	8.41
28	0.24	16.085	15.17	15.72	2.74	8.41
29	0.32	16.115	11.33	11.57	1.65	8.39
30	0.34	16.1	10.86	11.28	1.56	8.40
31	0.36	16.145	10.14	10.59	1.62	8.38
32	0.40	16.055	9.16	10.25	1.32	8.42
33	0.40	16.1	9.24	9.81	0.87	8.40
34	0.36	16.085	10.15	10.12	2.61	8.41
35	0.28	16.115	13.3	14.95	1.80	8.39
36	0.28	16.13	13.29	13.35	2.95	8.38
37	0.34	16.115	10.72	10.99	2.12	8.39
38	0.42	16.115	8.8	9.17	1.23	8.39
39	0.41	16.115	8.98	9.51	1.64	8.39

40	0.41	16.085	8.86	8.89	1.67	8.41
41	0.27	16.115	13.58	13.87	1.97	8.39
42	0.40	16.085	9.04	9.89	1.21	8.41
43	0.46	16.145	7.94	8.34	1.30	8.38

Table S4. Ratio of deconvoluted A_{1g} bands and magnetite share obtained from Raman and XRD (440) reflections.

Sample	Ratio (660cm^{-1}/710cm^{-1})	Magnetite % (Raman)	Magnetite % (XRD)
1	3.63	72.5	58.2
2	2.19	54.4	18.6
3	2.96	66.2	18.7
4	11.63	91.4	67.6
5	2.78	64.1	41.6
6	1.83	45.3	43.3
7	1.84	45.7	57.4
8	3.02	66.9	46.4
9	2.99	66.6	31.3
10	6.52	84.7	27.1
11	5.97	83.2	54.8
12	4.60	78.3	0.0
13	3.77	73.4	42.1
14	5.58	82.1	56.9
15	1.89	47.2	58.8
16	1.30	23.2	82.2
17	1.12	10.3	14.0
18	1.61	38.0	20.6
19	1.66	39.8	52.5
20	2.20	54.6	51.1
21	3.24	69.2	77.3
22	0.87	0.0	57.8
23	1.63	38.7	18.9
24	8.64	88.4	54.9
25	1.06	5.9	0.0
26	3.64	72.5	50.5
27	2.47	59.5	51.7
28	2.83	64.7	74.5
29	1.92	48.0	70.1
30	2.49	59.9	80.1
31	1.68	40.4	31.6
32	1.31	23.5	27.1
33	1.22	18.0	55.2
34	1.41	28.8	30.0
35	2.03	50.7	35.3

36	1.01	1.5	42.9
37	1.30	23.3	41.3
38	1.07	6.7	35.3
39	1.06	5.8	45.4
40	1.08	7.2	38.9
41	1.09	8.3	38.0
42	1.08	7.5	30.7
43	0.91	0.0	32.9