Supplementary Information

One-step Solvothermal Synthesis of Highly Water-soluble, Negatively Charged Superparamagnetic Fe_3O_4 Colloidal Nanocrystal Clusters

Jining Gao, Xinze Ran, Chunmeng Shi*, Humin Cheng, Tianmin Cheng and Yongping Su*

Institute of Combined Injury, State Key Laboratory of Trauma, Burns and Combined Injury, Chongqing Engineering Research Center for Nanomedicine, College of Preventive Medicine, Third Military Medical University, 30 Gaotanyan Road, 400038, Chongqing, China. Fax: +86-23-68752009; Tel: +86-23-68752279

E-mail: suyp2003@163.com, shicm@sina.com

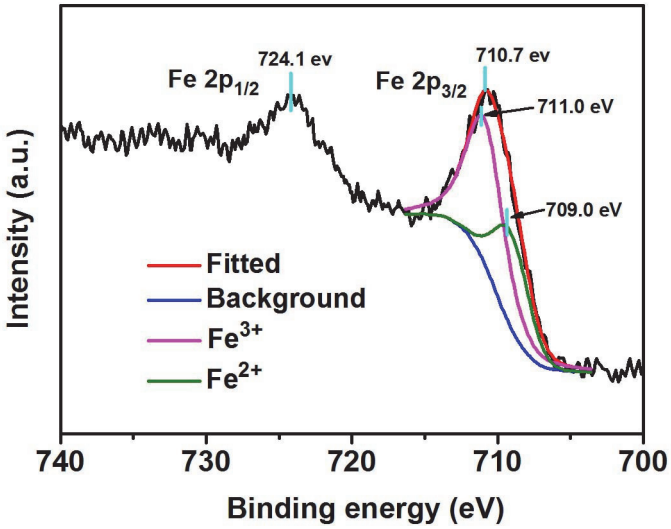


Fig.S1 XPS spectrum of the fitted Fe $2p_{2/3}$ peak from the products. The Fe $2p_{2/3}$ peak for Fe_3O_4 was deconvoluted into two peaks by using the peak positions of Fe^{2+} at 709.0 eV and Fe^{3+} at 711.0eV. The relative areas of the deconvoluted peak assigned to Fe^{2+} and Fe^{3+} were calculated to be 0.35:0.65; this value is clearly that of the stoichiometric of Fe_3O_4 within the uncertainty of calculations. Since Fe_3O_4 could also be expressed to be $FeO \cdot Fe_2O_3$, the Fe^{2+}:Fe^{3+} ratio should be 1:2 or 0.33:0.67.

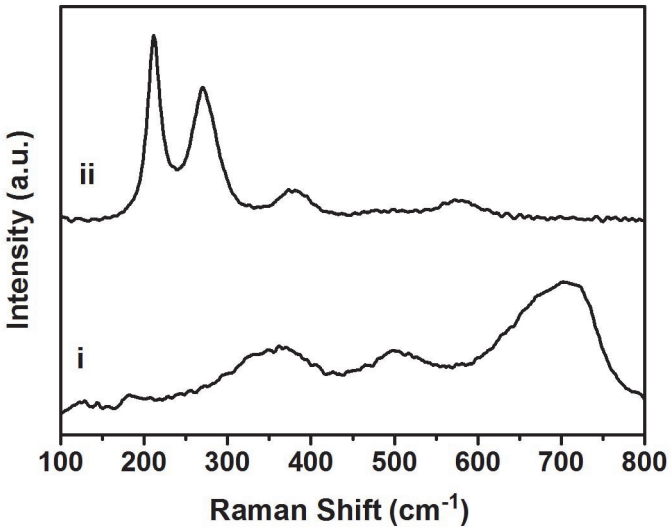


Fig.S2 Raman scattering spectra of the product synthesized with 0.5 g of PSSMA (3:1) obtained with different laser power (i) 0.1 mW and (ii) 1.0 mW. These two spectra were assigned to Fe_3O_4 (i) and α-Fe_2O_3 (ii), respectively. When the laser power was increased, the transformation form magnetite (Fe_3O_4) to into hematite (α-Fe_2O_3) was observed due to the local heating induced oxidation from laser irradiation; this phenomenon was in accordance with that reported in reference 36 and 37.

Table S1 Synthesis condition, corresponding hydrodynamic size and primary nanocrystal size of the MCNCs.

No.	$FeCl_3 \cdot 6H_2O$ (g)	Solvent (mL)		PSSMA (g) (SS:MA)	Time (h)	Z-Average Diameter (nm)	PDI	Primary Nanocrystal Size (nm)
		EG	DEG					
1	0.54	20	0	0	10	360.0 ± 20.7	0.238 ± 0.009	15.3
2	0.54	20	0	0.1 (3:1)	10	259.8 ± 2.7	0.156 ± 0.018	8.3
3	0.54	20	0	0.5 (3:1)	10	231.3 ± 2.5	0.027 ± 0.007	7.4
4	0.54	20	0	1.0 (3:1)	10	220.5 ± 0.9	0.025 ± 0.017	6.6
5	0.27	20	0	0.5 (3:1)	10	371.5 ± 8.0	0.238 ± 0.011	9.0
6	1.08	20	0	0.5 (3:1)	10	239.9 ± 1.4	0.059 ± 0.006	7.0
7	0.54	20	0	0.5 (3:1)	5	217.6 ± 1.9	0.038 ± 0.015	6.5
8	0.54	20	0	0.5 (3:1)	20	260.8 ± 1.6	0.053 ± 0.037	7.7
9	0.54	20	0	0.5 (1:1)	10	198.9 ± 1.2	0.031 ± 0.003	6.3
10	0.54	10	10	0.5 (3:1)	10	247.1 ± 1.5	0.029 ± 0.013	7.5
11	0.54	10	10	0.5 (1:1)	10	214.8 ± 2.6	0.037 ± 0.024	6.7

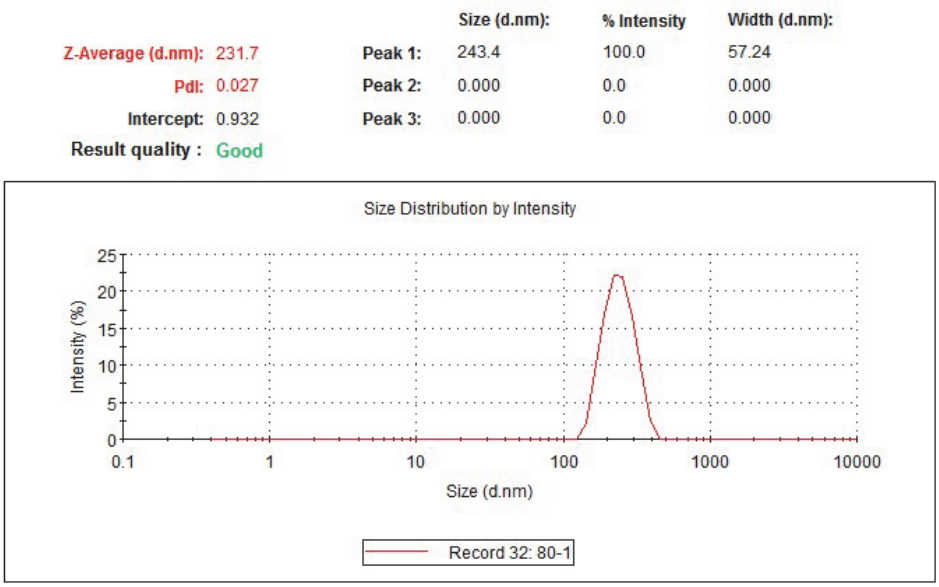
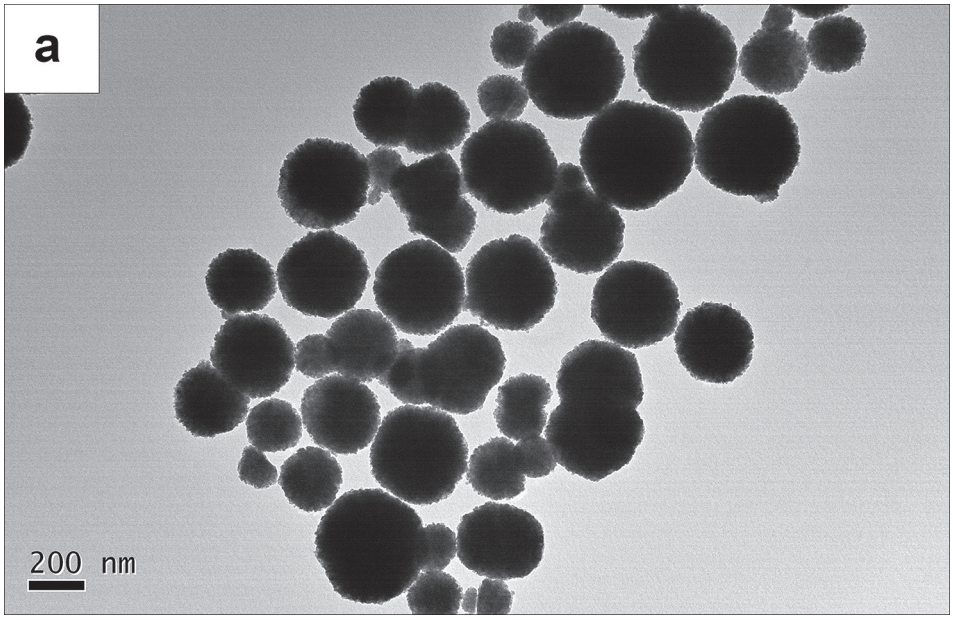


Fig. S3 A typical intensity particle size distribution for Fe_3O_4 MCNCs synthesized with 0.5 g of PSSMA (3:1) (Sample 3 in Table S1).



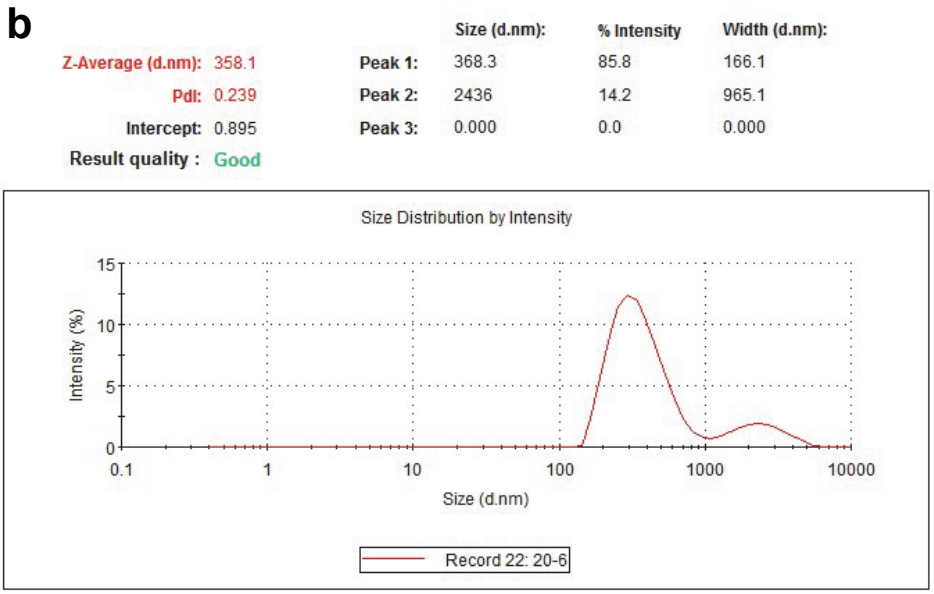


Fig. S4 A typical TEM image (a) and intensity particle size distribution (b) for Fe_3O_4 MCNCs synthesized without PSSMA (Sample 1 in Table S1).

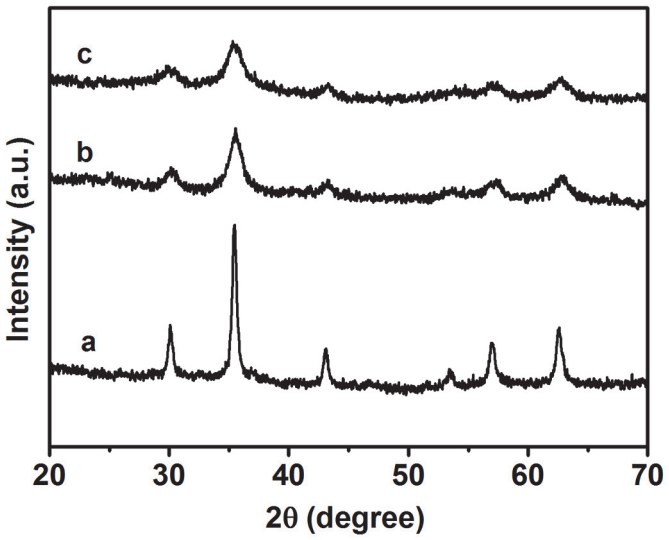


Fig. S5 XRD patterns of the Fe_3O_4 MCNCs obtained using different PSSMA: (a) no PSSMA, (b) 0.5 g PSSMA (3:1), and (c) 0.5 g PSSMA (1:1). (See also sample No.1, No.3, and No.9 in Table S1, respectively).

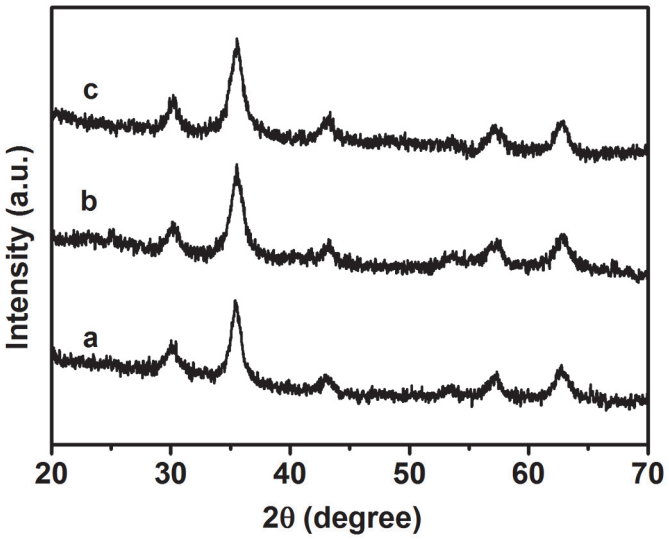


Fig. S6 XRD patterns of the Fe_3O_4 MCNCs obtained using different amounts of $FeCl_3 \cdot 6H_2O$: (a) 0.27, (b) 0.54, and (c) 1.08 g, (See also sample No.3, No.5, and No.6 in Table S1, respectively).

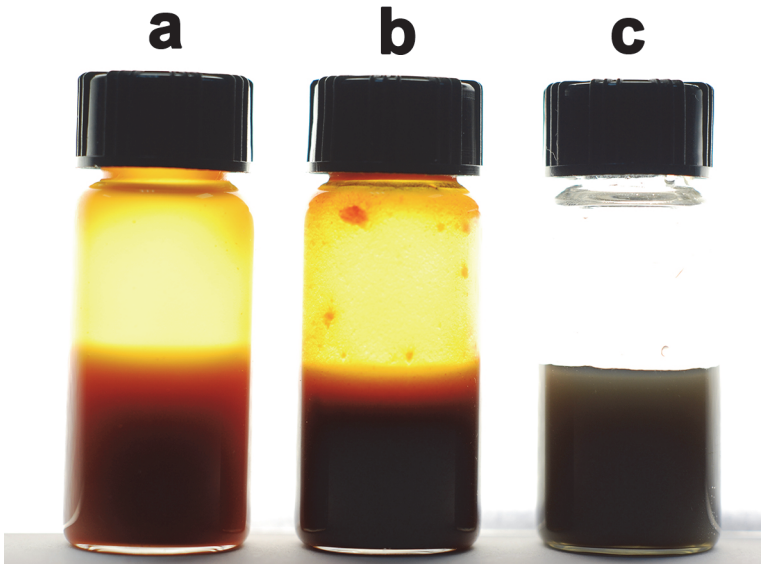


Fig. S7 Digital photographs of the reaction mixtures with the reaction time of (a) 1 h, (b) 3 h, (c) 10 h.

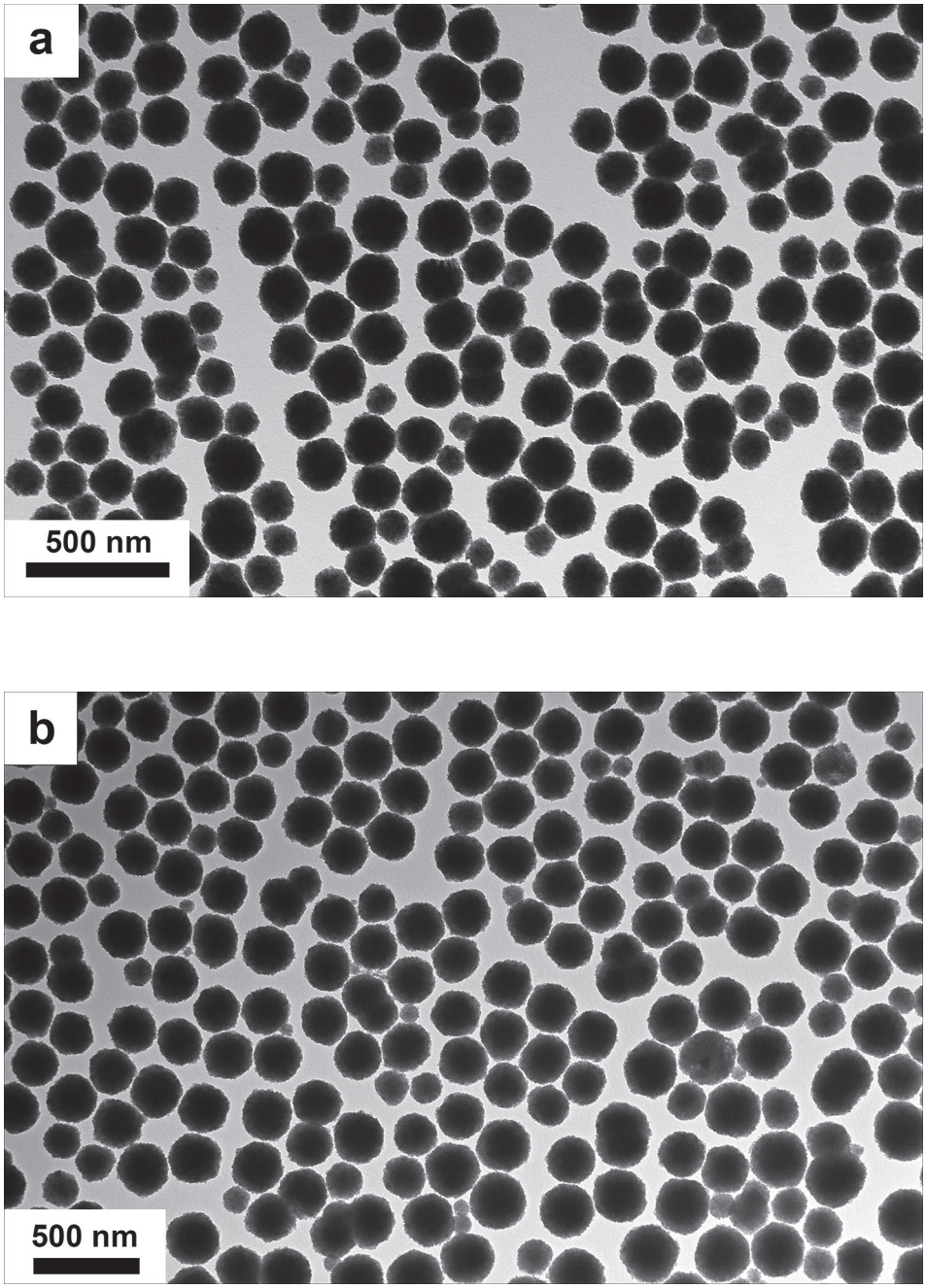


Fig. S8 TEM images of Fe_3O_4 MCNCs synthesized with 0.5 g PSSMA (3:1) with the reaction time of (a) 5 h, (b) 20 h.

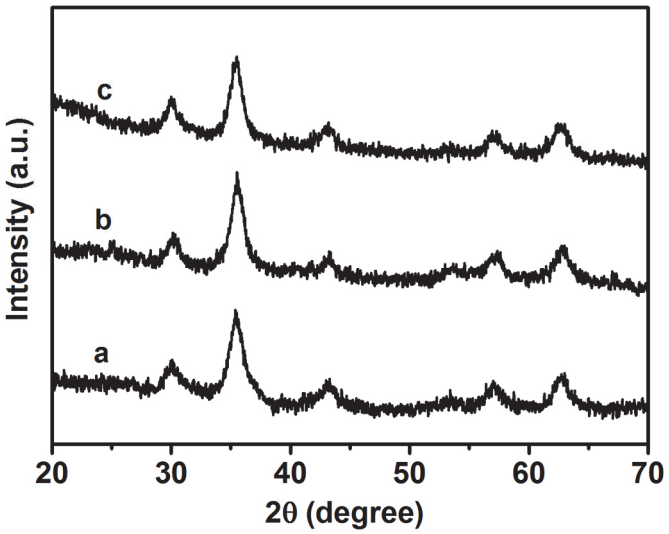


Fig. S9 XRD patterns of the Fe_3O_4 MCNCs obtained with different reaction time: (a) 5, (b) 10, and (c) 20 h. (See also sample No.7, No.3, and No.8 in Table S1, respectively).

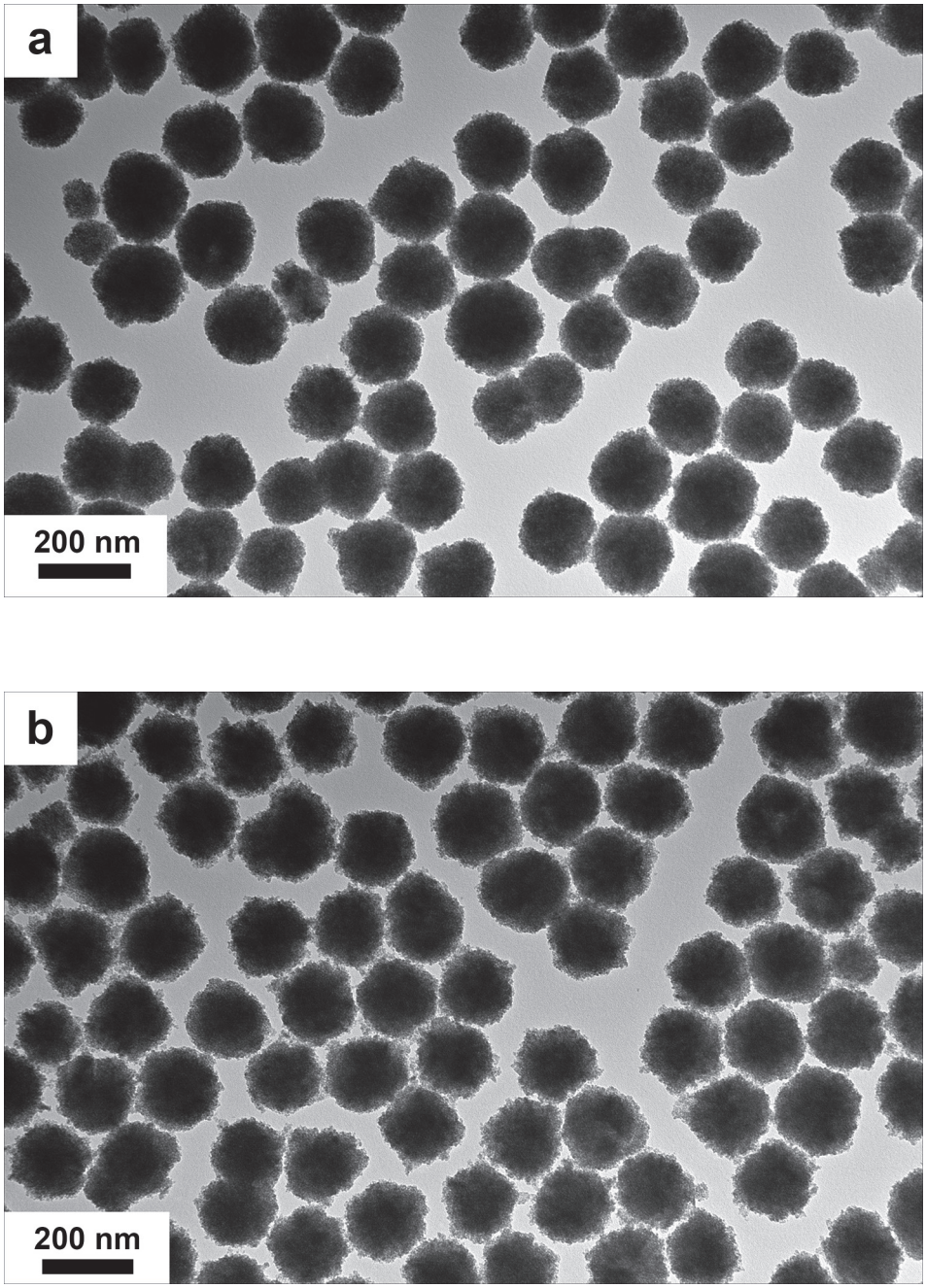


Fig. S10 TEM images of Fe_3O_4 MCNCs synthesized in solvent mixtures with EG/DEG ratio (v/v) of 10/10 using 0.5 g of (a) PSSMA 3:1 and (b) PSSMA 1:1.

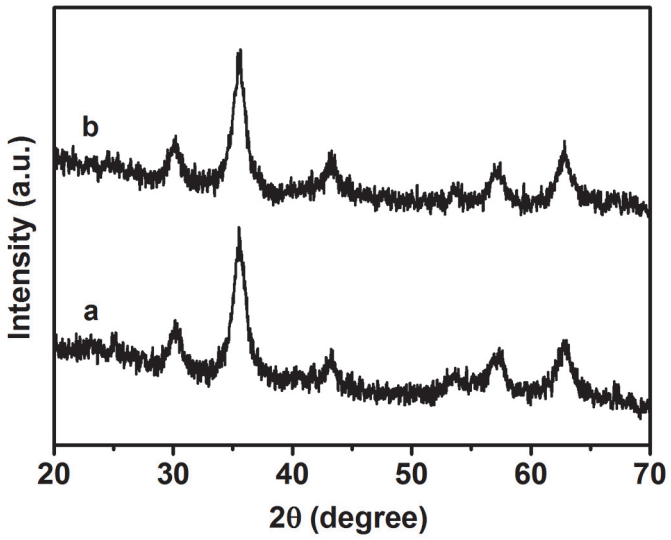


Fig. S11 XRD patterns of the Fe_3O_4 MCNCs obtained using different solvent composition in the presence of 0.5 g PSSMA (3:1): (a) 20 mL EG, (b) 10 mL EG and 10 mL DEG. (See also sample No.3, No.10 in Table S1, respectively).

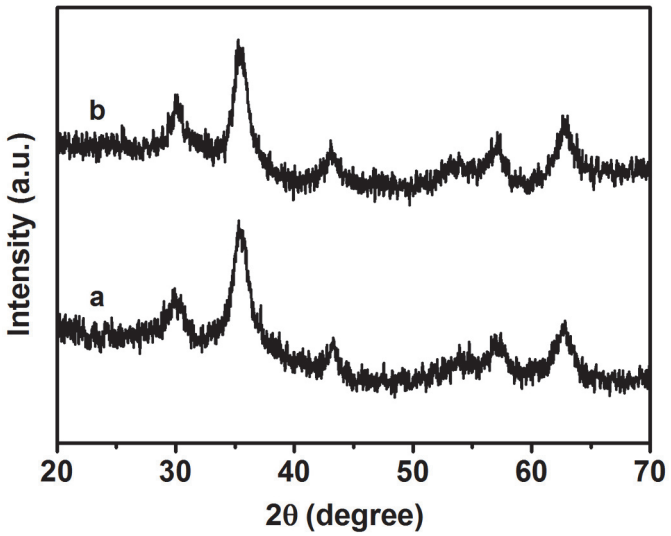


Fig. S12 XRD patterns of the Fe_3O_4 MCNCs obtained using different solvent composition in the presence of 0.5 g PSSMA (1:1): (a) 20 mL EG, (b) 10 mL EG and 10 mL DEG. (See also sample No.9, and No.11 in Table S1, respectively).

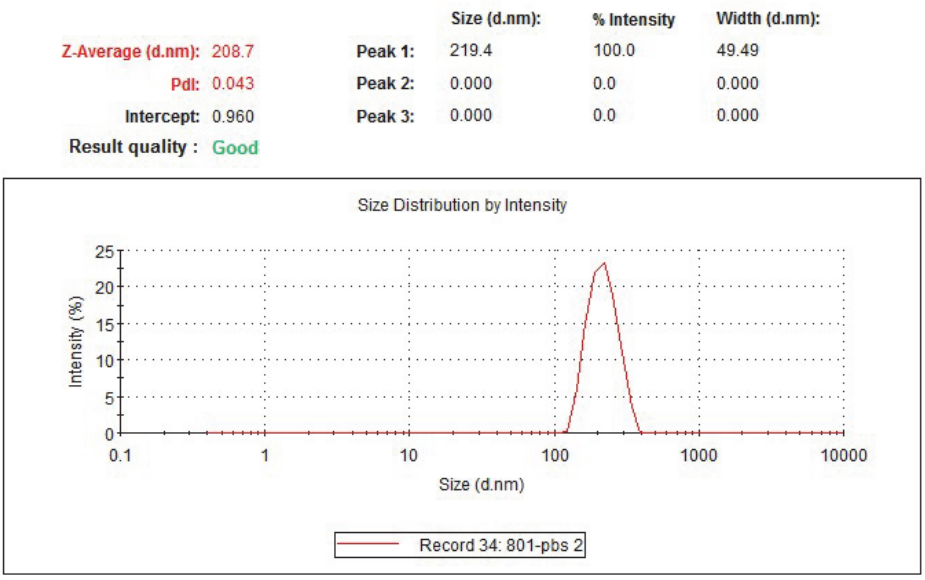


Fig. S13 A typical intensity particle size distribution for Fe_3O_4 MCNCs dispersed in PBS.

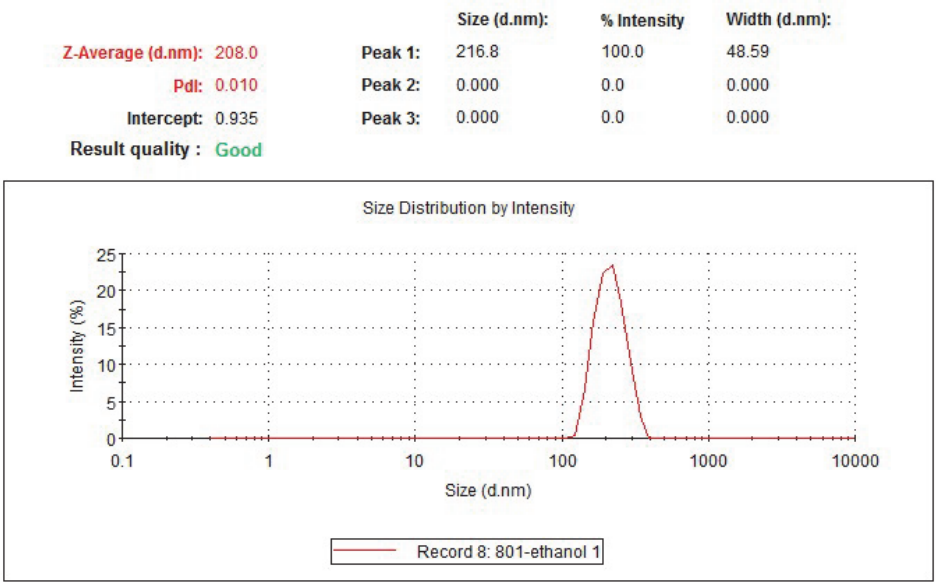


Fig. S14 A typical intensity particle size distribution for Fe_3O_4 MCNCs dispersed in ethanol.

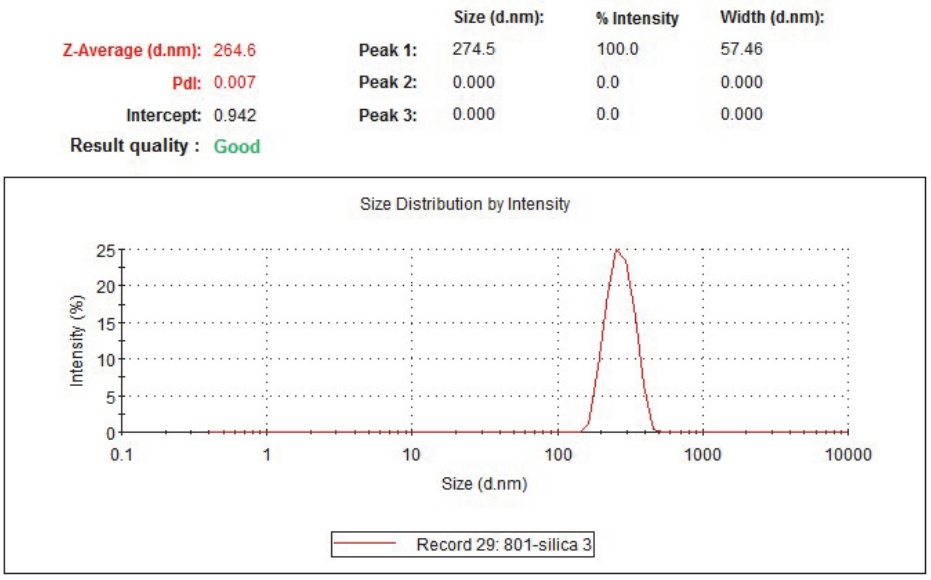


Fig. S15 A typical intensity particle size distribution for silica coated Fe_3O_4 MCNCs dispersed in water.