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Supplementary information

Scale-up approach for the preparation of magnetic ferrite nanocubes and other shapes with benchmark performance for magnetic hyperthermia applications

SUPPLEMENTARY INFORMATION

Scale up approach for the preparation of magnetic ferrite nanocubes and other shapes with benchmark performance for Magnetic Hyperthermia Applications

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Experimental section

MATERIALS

REAGENTS:

- Tolualdehyde (4-methylbenzaldehyde, Sigma-Aldrich, 97%)
- Biphenyl-4-carboxaldehyde (4-phenylbenzaldehyde, Sigma-Aldrich, 99%)
- M-Anisaldehyde (3-methoxybenzaldehyde, Sigma-Aldrich, 97%)
- Oleylamine (Sigma-Aldrich, 98%)
- 4-methyl-2-phenyl-2-pentenal (α-(2-Methylpropylidene)-benzeneacetaldehyde, mixture of cis and trans, Sigma-Aldrich, 88%)
- Phenylacetaldehyde (Sigma-Aldrich, 90%)
- 3-Phenylpropionaldehyde (hydrocinnamaldehyde , Sigma-Aldrich, 90%)
- Decanal (Sigma-Aldrich, 98%)
- Heptaldehyde (Sigma-Aldrich, 95%)
- Pentanal (Sigma-Aldrich, valeraldehyde, 97%)
- Cis-4-Heptenal (Sigma-Aldrich, 98%)
- Trioctylamine (Sigma-Aldrich, 98%)
- Tridodecylamine (Sigma-Aldrich, 97%)
- Dioctylamine (Sigma-Aldrich, 98%)
- Didodecylamine (Sigma-Aldrich , 97%)

SOLVENTS:

- Acetone (Carlo Erba, 99.5%)
- Chloroform (Carlo Erba, 99.8%)
- Isopropanol (Carlo Erba, 99.8%)
  Diethyl ether (Carlo Erba, 99.8%)

Water transfer protocols

BOX S1: *WATER TRANSFER PROTOCOLS OF IRON OXIDE NANOCUBES USING* ***TETRAMETHYLAMMONIUM HYDROXIDE***

MATERIALS

- *Tetramethylammonium hydroxide pentahydrate (TMAOH, Ligand, Merck, ≥97%)*
- *Triethylamine (TEA, base, Merck, 99.5%)*
- *Ethanol Absolute Anhydrous (Carlo Erba, 99.9%)*
- *Milli-Q water filtered through 0.22 µm pore size hydrophilic filters (18.2 MΩ cm) (supplied by a Milli-Q® Integral Water Purification System)*

EQUIPMENT

- *8 mL glass vials*
- *Sonicator (USC 600 THD or USC 500 THD)*
- *Centrifuge Membrane tubes (Amicon® membrane filters of 50,000 MWCO, 15 mL volume)*

PROCEDURE

1. *Centrifuge (4500 rpm, 20 min), 1.4 mL of a solution of nanoparticles in chloroform ([Fe] = 7 mg/mL) after addition of acetone (5 mL) to precipitate and collect the nanoparticles.*
2. *Discard solvents and dry gently the pellet using a nitrogen flux.*
3. *Add 1 mL of a TMAOH solution in anhydrous ethanol (0.27 M) to the nanoparticle powder.*
4. *Sonicate the solution for 30 min at room temperature*
5. *Add Milli-Q water (3 mL) and centrifuge the solution using centrifuge membrane tubes (1500 rpm, 10 min).*
6. *To the nanoparticles solution on the centrifuge tube, repeat step 5 at least two more times to wash out the excess of TMAOH ligands.*
7. *Recover the nanoparticle solution and redisperse in 2 mL Milli-Q water.*

TIMING

Step 1: 20 min
Steps 2-4: 30 min
Steps 5-7: 70 min
Overall reaction time: ca. 2 hours

BOX S2: *WATER TRANSFER PROTOCOL OF IRON OXIDE NANOCUBES USING* ***GALLIC POLYETHYLENE GLYCOL***

MATERIALS

- *Gallic polyethylene glycol (PEG-GA, produced according to reference Guardia et al., J. Mater. Chem. B, 2014, 2, 4426).*
- *Triethylamine (TEA, base, Merck, 99.5%)*
- *Chloroform (Carlo Erba, 99.8%)*
- *Toluene (Carlo Erba, 99.7%)*
- *Milli-Q water filtered through 0.22 µm pore size hydrophilic filters (18.2 MΩ cm) (supplied by a Milli-Q® Integral Water Purification System)*

EQUIPMENT

- *40 mL glass vials*
- *Orbital shaker*
- *Rotary evaporator*
- *Separating funnel*
- *Cellulose dialysis membrane (Spectrum labs) with 50 kDa MWCO*
- *Centrifuge membrane tubes (Amicon® with membrane filters of 100,000 MWCO, 15 mL volume)*

PROCEDURE

1. *Mix 5 mL of a chloroform solution of 14 nm nanocubes at [Fe] = 1 mg/mL with 8.2 mL PEG-GA chloroform solution (0.05 M) and add 0.8 mL of trimethylamine at an excess of ligand molecules per nanocube surface of 300 L/nm^2.*
2. *Stir overnight on the orbital shaker at RT.*
3. *Transfer the chloroform solution to a separating funnel containing water (100 mL) and toluene (40 mL).*
4. *Shake the mixture to form an emulsion*
5. *Add acetone (100 mL) to promote phase separation and, just after, collect the aqueous phase containing the nanocubes.*
6. *Remove traces of organic solvents by purging nitrogen for 2 h.*
7. *Concentrate the sample under reduced pressure in a rotary evaporator at 40 ° C to reach a final volume of 10 mL.*
8. *Remove the excess of PEG-GA by al least one overnight dialysis versus 5 L of de-ionized water using dialysis membrane.*
9. *Concentrate the nanocubes solution by centrifugation on the centrifuge membrane tubes (1500 rpm, 10 min as many times as needed) until reaching a final volume of 1 mL.*
10. *Sonicate the water solution for 30 min at 65 °C.*

TIMING

Step 1-2: 24 h
Steps 3-5: 3 h
Steps 6-7: 2.5 h
Step 8: 16 h
Step 9: 2 h
Step 10: 0.5 h
Overall reaction time: ca. 48 hours

BOX S3:** WATER TRANSFER PROTOCOLS OF IRON OXIDE NANOCUBES USING **α-NITRODOPAMINE-ω-CARBOXY-POLY(ETHYLENE GLYCOL)

MATERIALS

- *α-nitrodopamine-ω-carboxy-poly(ethylene glycol (ND-PEG-COOH, produced according to reference Zyuzin et al., ACS Appl. Mater. Interfaces 2019, 11, 41957-41971)*
- *Triethylamine (TEA, base, Merck, 99.5%)*
- *Chloroform (Carlo Erba, 99.8%)*
- *Milli-Q water filtered through 0.22 μm pore size hydrophilic filters (18.2 MΩ cm) (supplied by a Milli-Q® Integral Water Purification System)*

EQUIPMENT

- *8 mL glass vials*
- *Separating funnel*
- *Orbital shaker*
- *Cellulose membrane (Spectrum labs) with a pore size of 50 kDa*
- *Centrifuge Membrane tubes (Amicon® with membrane filters of 50,000 MWCO, 15 mL volume)*

PROCEDURE

1. *Dissolve 148 mg of the polymeric ligand (0.098 mmol) in the 3.1 mL of chloroform to yield a 0.05 M solution.*
2. *Dilute 0.67 mL of the nanocubes ([Fe]=7.5 mg/mL, equivalent to 5 mg Fe) (14±1 nm) in chloroform to reach a final concentration of 1 mg Fe/mL and a ratio of 150 ligands/nm^2.*
3. *Add 0.97 mL (6.9 mmol) triethylamine (70 eq. with regard to mmol of ligands).*
4. *Shake the mixture overnight on an orbital shaker at RT.*
5. *Transfer the solution into a separating funnel with 40 mL of toluene and 100 mL of water.*
6. *Shake the mixture to form an emulsion and wait 2 h*
7. *After phase separation, collect the aqueous phase containing the nanocubes.*
8. *Repeat steps 6-7 at least 1time by adding to the organic phase 50 mL of water at each cycle.*
9. *Combine the extracted aqueous solutions and remove the toluene traces by purging nitrogen for 2 h.*
10. *Concentrate the sample to a final volume of approximately 10 mL by centrifugation using an Amicon centrifuge tube (1500 rpm, 10 min, repeat as many times as needed).*
11. *Sonicate the solution of nanocubes for 30 min at 65 °C.*
12. *Transfer the sample in a dialysis membrane and dialyze the sample for 2 days against 5 L of water for removing unbound and excess ligand (change the water bath at least 2 times).*
13. *Concentrate the sample to a final volume of approximately 2 mL using an Amicon centrifuge tube (10 min. cycle at 1500 rpm, to be repeated as any times as needed).*
14. *Sonicate the final solution of nanocubes for 30 min at 65 °C.*

TIMING

Step 1-4: 24 h
Steps 5-8: 3 h
Steps 9-10: 3.5 h
Step 11: 48 h
Steps 12-13: 1.5 h
Overall reaction time: ca. 80 hours

BOX S4: *WATER TRANSFER PROTOCOL OF IRON OXIDE NANOCUBES USING POLYMER COATING WITH* ***POLY(MALEIC ANHYDRIDE-ALT-1-OCTADECENE)***

MATERIALS

- *poly maleic anhydride-alt-1-octadecene (PMAO, Merck)*
- *Sucrose (Sigma-Aldrich, 99%)*
- *Chloroform (Carlo Erba, 99.8%)*
- *Sodium borate buffer (50mM, pH 9)*
- *Milli-Q water filtered through 0.22 μm pore size hydrophilic filters (18.2 MΩ cm) (supplied by a Milli-Q® Integral Water Purification System)*

EQUIPMENT

- *Round flask*
- *Rotary evaporator*
- *Ultra-centrifuge tubes for speed up to 40k RPM(Beckman Coulter)*
- *Sonicator (USC 600 THD or USC 500 THD)*
- *UV Lamp*
- *Centrifuge Membrane tubes (Amicon® filters of 50,000 MWCO, 15 mL volume)*

PROCEDURE

1. *Add a solution of PMAO polymer in chloroform (137mM in monomeric unit) to a solution of oleic acid-coated nanocubes in chloroform at a concentration of 10nM in order to reach a ratio of polymer monomer units per nm^2 of nanoparticles surface equal to 500 (considering nanocubes of 14±1 nm). The final volume of the mixture is around 70 mL.*
2. *Evaporate slowly and completely the solvent using a rotary evaporator (bath set at 50 °C and slowly reducing the pressure from 800 to 600 mbar).*
3. *Add 20 mL of sodium borate buffer (50 mM, pH 9) to recollect the thin film of nanoparticles from the walls of the flask and sonicate in a water bath set at 65 °C for 1 h).*
4. *Concentrate the nanocubes solution on the centrifuge filters to nearly 3 mL at 1500 rpm at RT.*
5. *Load 0.5 mL of the sample solution on top of a sucrose gradient (prepared by deposition of 3 mL of 60%, 3 mL of 40% and 2 mL of 20%in an ultra-centrifugal tube and centrifuge at 20000 rpm for 45 min at 4°C.*
6. *Recover the nanocubes from the centrifuge tube with a syringe in the 40% to 60% sucrose fraction by discarding first the excess of polymer, visible under a UV lamp, at the top of the sucrose gradient.*
7. *Wash the nanocubes with pure water (Milli-Q), filter three times with centrifuge filter (1500 rpm for 10 min each run at RT) to remove the excess of sucrose.*
8. *Concentrate the sample to a final volume of approximately 4 mL using an Amicon centrifuge tube (1500 rpm, 10 minute per cycle for as many cycles as needed).*
9. *To redisperse the nanocubes, sonicate the final solution for 30 min at 65 °C.*

TIMING

Step 1-3: 4h
Steps 4-5: 2 h
Steps 6-9: 2 h
Overall reaction time: ca. 8 hours

CHARACTERIZATIONS

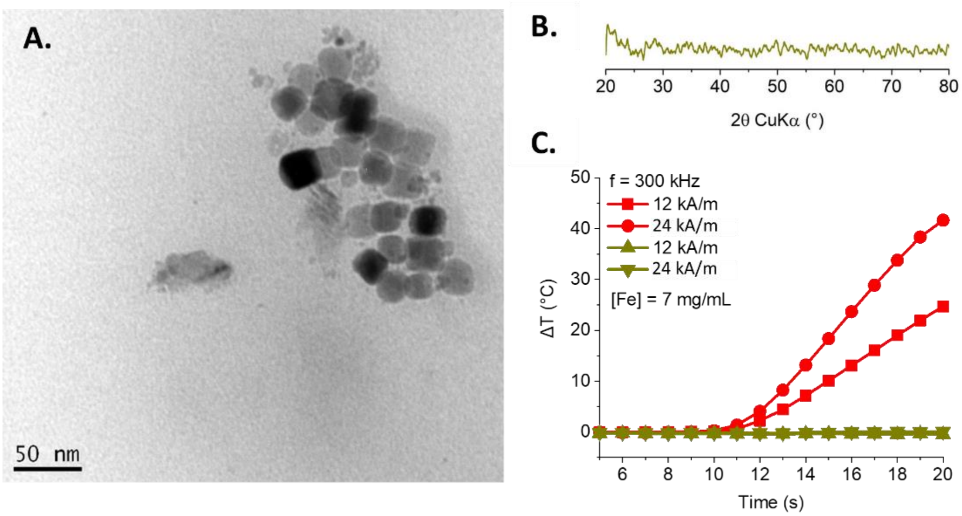
Elemental analysis: Inductively coupled plasma optical emission spectroscopy (ICP-OES) analysis was performed on an iCAP 6000 spectrometer (Thermo Scientific) to quantify the concentration of the elemental elements of interest. Briefly, 25 µL of sample was digested in 2.5 mL of aqua regia ($HCl:HNO_3$ ratio is 3:1 (v/v)) overnight and diluted to 25 mL of Milli-Q water prior to the measurement.

Transmission electron microscopy (TEM): TEM analyses were carried out on a JEOL JEM-1011 instrument, using an acceleration voltage of 100 kV. The sample preparation was conducted by drop-casting few drops of a diluted sample solution onto a 400 mesh ultra-thin carbon-coated copper grid with a subsequent evaporation of the solvent at RT. Size determination involved the use of ImageJ, an open-source freeware, using a sample size of ca. 200 nanoparticles.

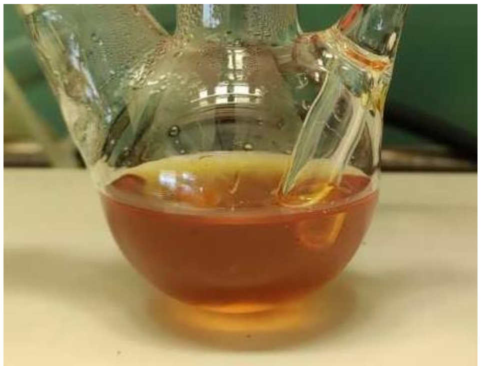
X-Ray diffraction analysis: XRD patterns were recorded on a Malvern PANalytical Empyrean diffractometer machine. The X-ray source is operated at 40 kV and 150 mA. The diffractometer was equipped with a Cu source and a Göbel mirror to obtain a parallel beam as well as to suppress Cu Kβ radiation (1.392 Å). The 2-theta/omega scan geometry was used to acquire the data. The samples were prepared by drop casting a concentrated solution of nanoparticles (ca. 6-8 mg/mL) onto a zero background silicon substrate. The PDXL software of Rigaku was used for phase identification.

Dynamic light scattering (DLS): The particle hydrodynamic size distribution was measured using a Malvern Zeta sizer operated in the 173° backscattered mode on a highly diluted (< 0.5 mg/mL) aqueous solution of nanoparticles. The samples were diluted with water whose pH was adjusted to 8. The measurements were performed at 20 °C.

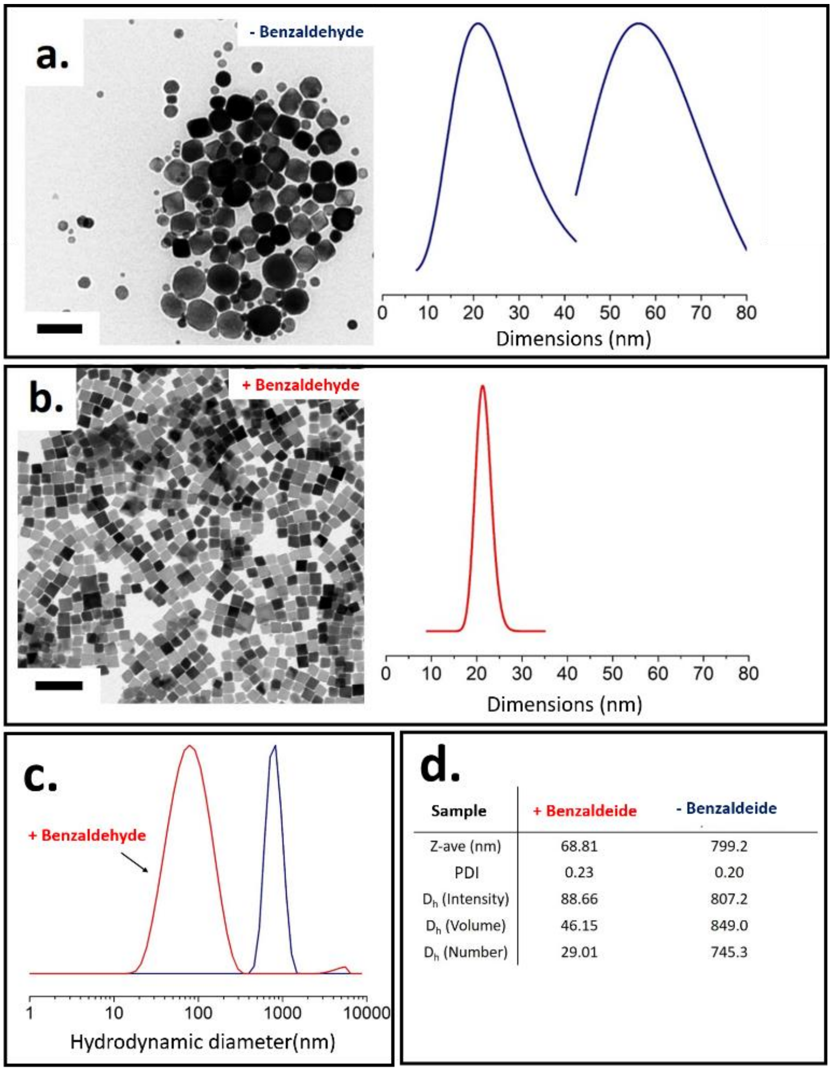
Supplementary Figures



Supplementary Figure 1. Nanocubes reproduced according to J. Phys. Chem. C 2012, 116, 5476-5481. (A) TEM image showing the irregular sample, in which a fraction has cubic shape (the size is 25 nm). (B) pattern-ray diffractogram collected for the sample, showing that the sample is not crystalline. (C) Temperature versus time curves recorded for aqueous solutions of iron oxide nanocubes (IONCs) of 20 nm obtained through our method (red line) and according to J. Phys. Chem. C 2012, 116, 5476-5481 (green line). The samples were characterized at 12 and 24 kA/m and 300 kHz. The nanocubes reproduced according to J. Phys. Chem. C 2012, 116, 5476-5481 are not able to increase the temperature of the solution under AMF exposure at any field condition here tested.

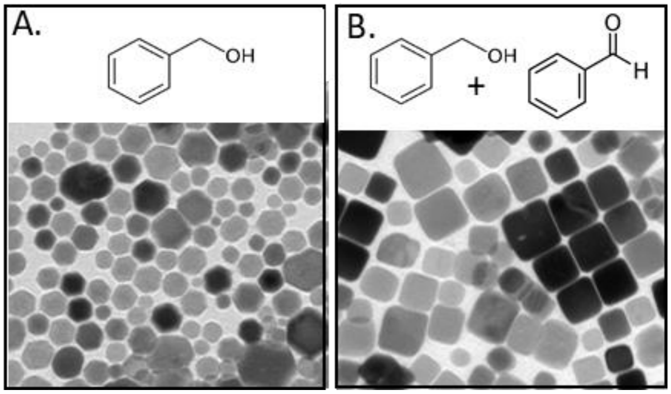


Supplementary Figure 2. Photograph of the homogeneous final stock solution containing all the reagents, including the metal precursor (iron pentacarbonyl) and the shape directing agent (i.e benzaldehyde).

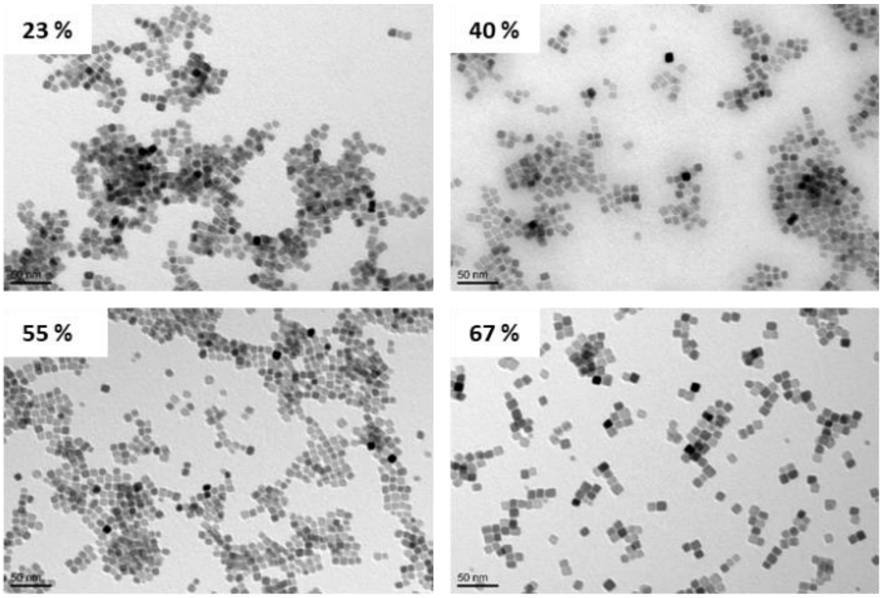


Sample	+ Benzaldeide	- Benzaldeide
Z-ave (nm)	68.81	799.2
PDI	0.23	0.20
D_h (Intensity)	88.66	807.2
D_h (Volume)	46.15	849.0
D_h (Number)	29.01	745.3

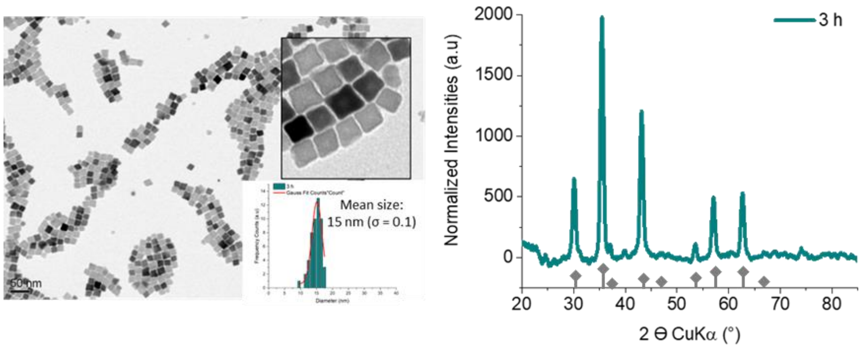
Supplementary Figure 3. Control synthesis to assess the role of the aldehyde as shape-directing agent. Two syntheses having the same stock solution composition (as described for the typical synthesis of Fe_3O_4 nanocubes paragraph) with the only exception of the absence or presence of benzaldhyde within the mixture were annealed in the oven for solvothermal crystallization. A) TEM image of the NPs obtained in the absence of the aldehyde. The nanoparticles have an irregular shape and wide multimodal size distribution. B) TEM image of the NPs obtained in the presence of benzaldehyde, having a well-defined nanocube shape and narrow size distribution of 21 ±2 nm with quite fair low polydispersive index (PDI). C) Hydrodynamic sizes diagram by intensity weight peaks of the corresponding samples after water transfer protocol with tetramethylammonium hydroxide (TMAOH) molecule. The nanocubes have a monomodal size distribution with a peak in intensity weight centred at 89±20 nm. Instead, the sample prepared in absence of benzaldehyde are not stable and have the main peak centred at 800±160 nm. D) Table summarizing the colloidal properties of both samples.



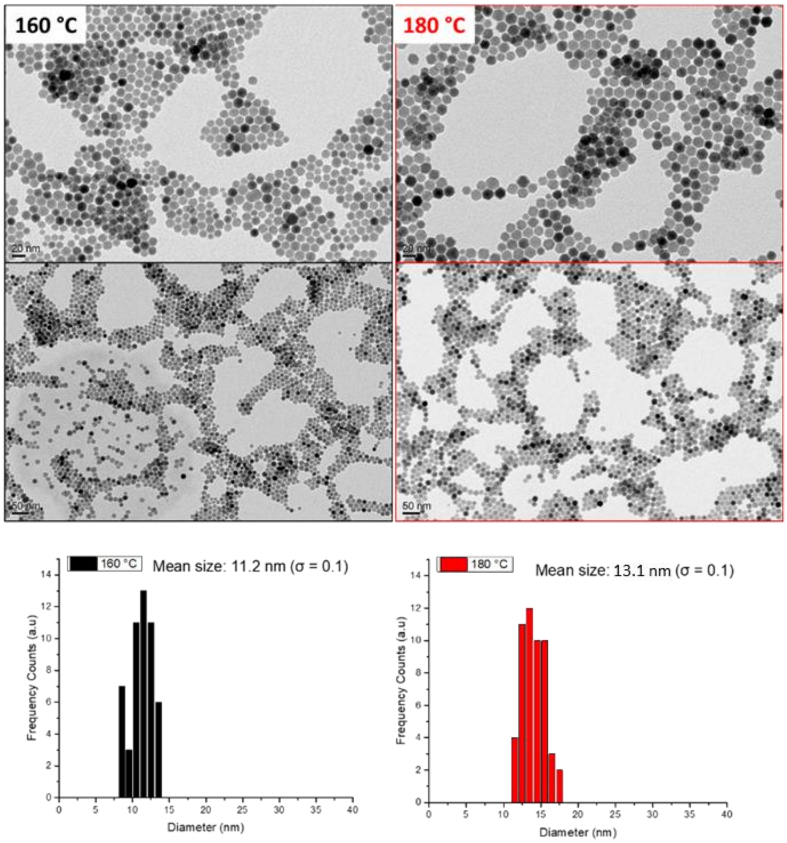
Supplementary Figure 4. Control synthesis to assess the role of the aldehyde as shape-directing in the synthesis. We attempted syntheses using only benzyl alcohol A) or benzyl alcohol + benzaldehyde B).



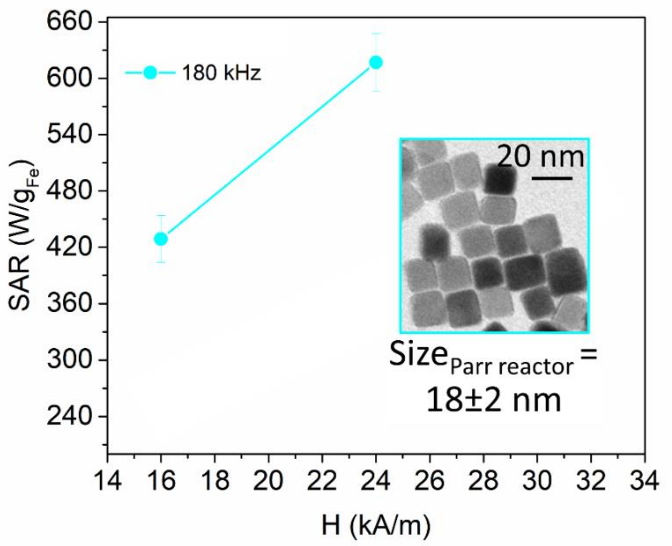
Supplementary Figure 5. Tuning of the size of the iron oxide nanocubes (IONCs) varying the filling percentage of a 50 mL autoclave vessel from 23, 40, 55 and 67% resulting in nanocubes with the size of tuned from 8 ±1 to 12 ±1nm.



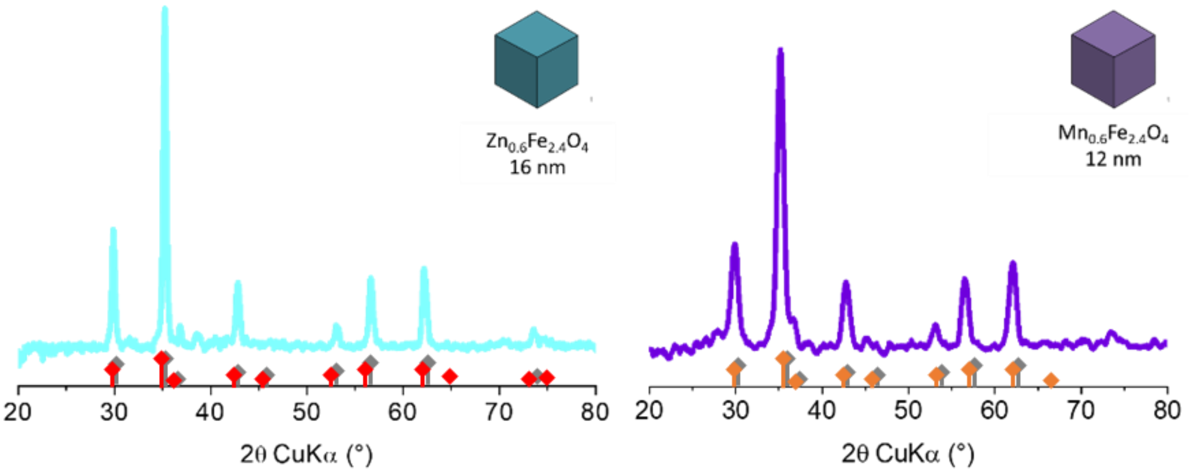
Supplementary Figure 6. TEM image of the nanoparticles obtained after a solvothermal crystallization time in the oven of only 3 h (left); XRD diffractogram of the nanoparticles (right), showing that despite the shorter annealing times, the nanoparticles have the typical peaks of magnetite and possess high crystallinity. In grey, the theoretical peaks for the inverse spinel of Fe_3O_4 powder (JCPDS card no. 00-001-1111).



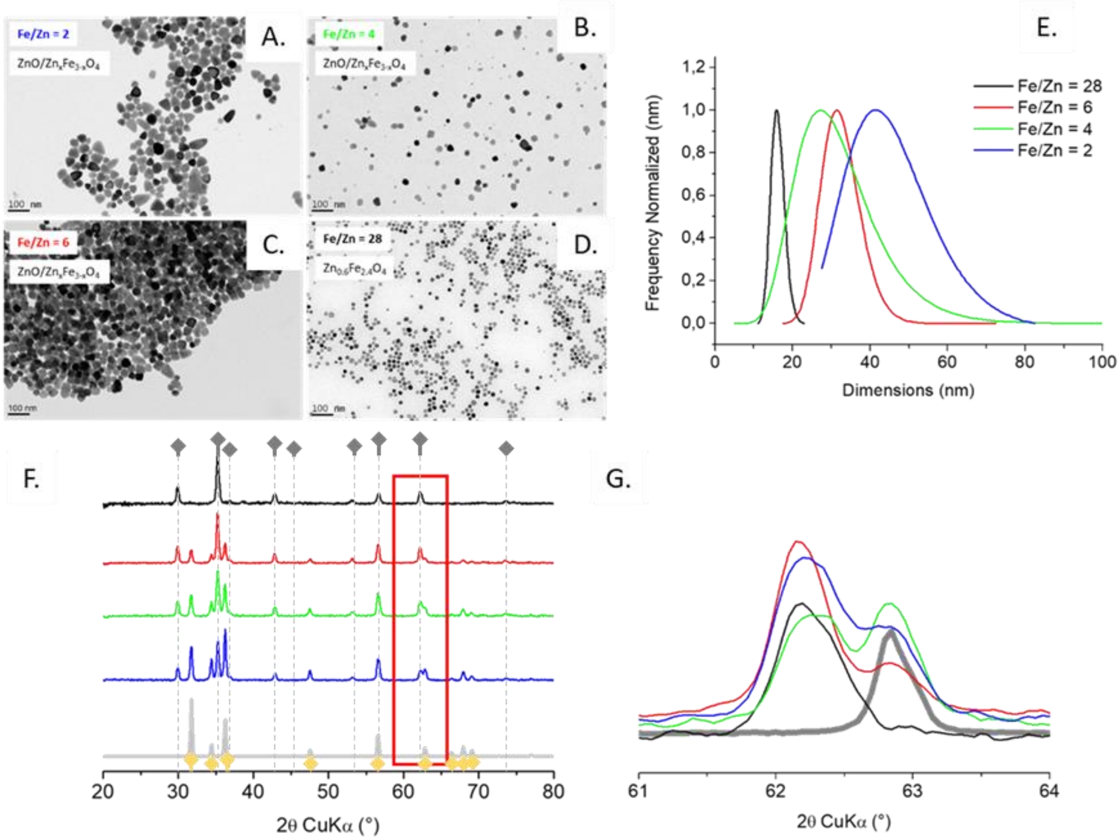
Supplementary Figure 7. TEM images of the nanoparticles obtained at 160 °C (framed in black) and 180 °C (framed in red). A reduction in the annealing temperature leads to a reduction of the size but also a change in the shape, passing from well-defined 20 nm nanocubes (as those obtained at 200 °C), to 13 and 11 nm (σ = 0.1) truncated octahedra (obtained at 180 and 160 °C, respectively).



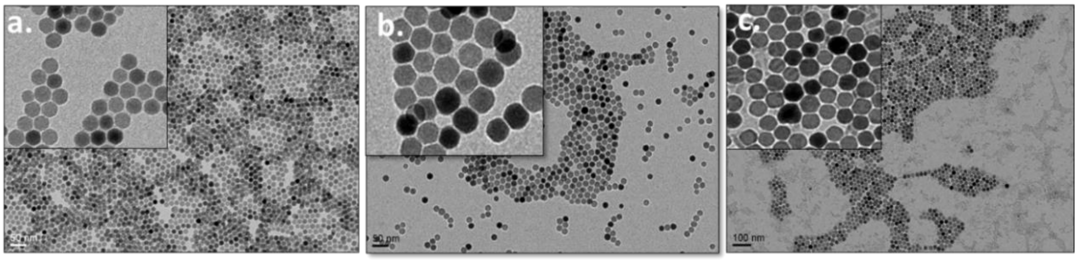
Supplementary Figure 8. SAR data acquired for the iron oxide nanocubes of 18±2 nm using the AMF conditions of 180 kHz and 16, 24 kA/m. The SAR values are in the range of 420-630 W/gFe.



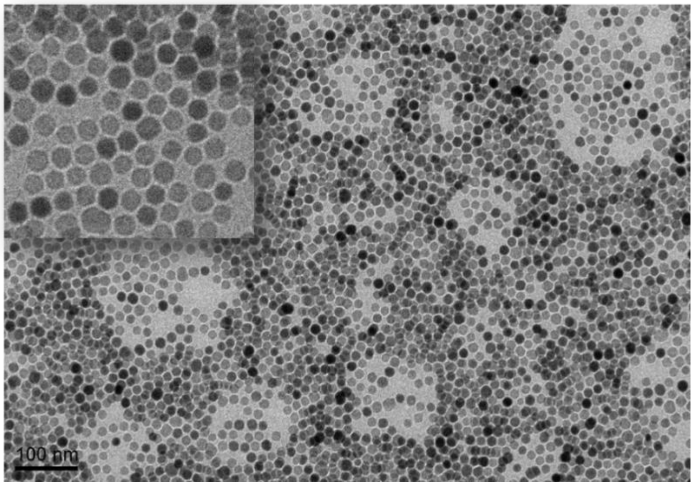
Supplementary Figure 9. XRD patterns collected for Zn-ferrite and Mn-ferrite nanocubes. On the lower part in grey, the peaks for the inverse spinel Fe_3O_4 powder (JCPDS card no. 00-001-1111), in red the theoretical peaks for $ZnFe_2O_4$ powder (JCPDS card no. 00-022-1012) and in orange the theoretical peaks for $MnFe_2O_4$ powder (JCPDS card no. 00-074-2403) are represented, respectively.



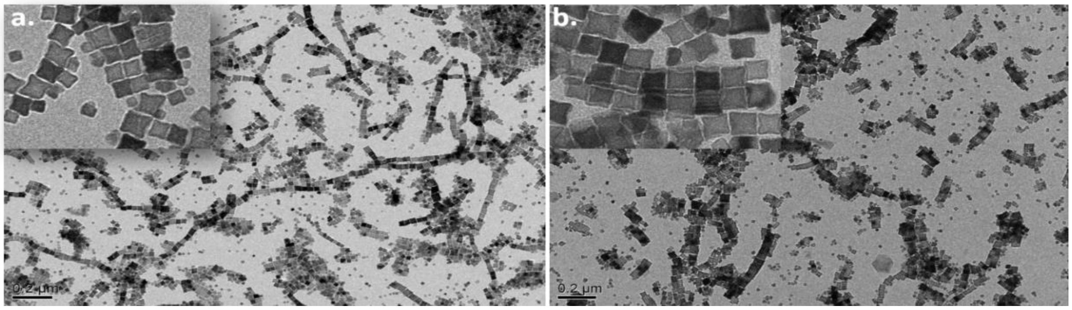
Supplementary Figure 10. Optimization of synthesis of the Zn-ferrite nanocubes- TEM images showing the nanoparticles obtained when varying the Fe/Zn feed ratio: A) 1:2; B) 1:4; C)1:6 and D) 1:28. The formation of polydispersed "bullet-like" nanoparticles progressively disappears forming monodisperse nanocubes only at the highest feed ratio (28). E) Corresponding size distributions obtained by adjusting the TEM analysis of the size to a log-normal curve. F-G) XRD patters obtained for the nanoparticles obtained when using the different Fe/Zn feed ratio. It is observed that only at a feed ratio of 1:28 peaks that can be attributed to a single inverse spinel structure. At lower feed ratios, Bragg peaks attributed to ZnO are present. On the upper part, in red the theoretical peaks for $ZnFe_2O_4$ powder (JCPDS card no. 00-022-1012) and in the lower part in yellow the theoretical peaks for ZnO powder (JCPDS card no. JCPDS No. 00-036-1451) are represented, respectively.



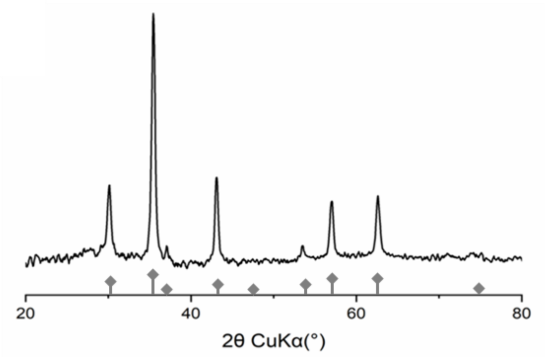
Supplementary Figure 11. TEM images of the quasi-spherical/faceted iron oxide nanoparticles obtained in the presence of aliphatic aldehydes and tuning the experimental conditions according to **Table 2** in order to obtain: a) 15±2 nm NPs (pentanal); b) 17±2 (heptanal) and c) 18±2 nm with decanal. Insets: TEM image at higher magnification reported to appreciate the particles morphology.



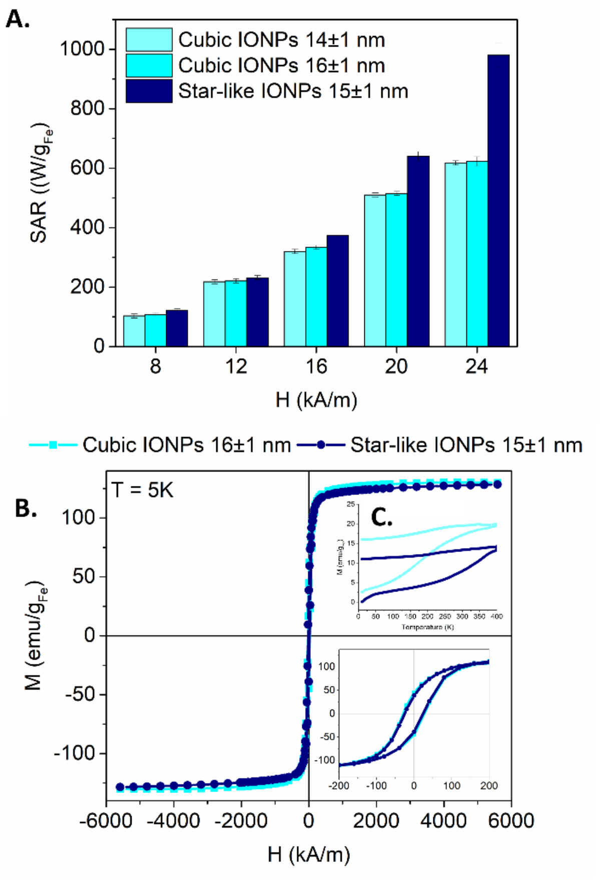
Supplementary Figure 12. TEM image of the MNPs obtained in the presence of cis-4-hepten-1-al (see experimental conditions in **Table 2**).



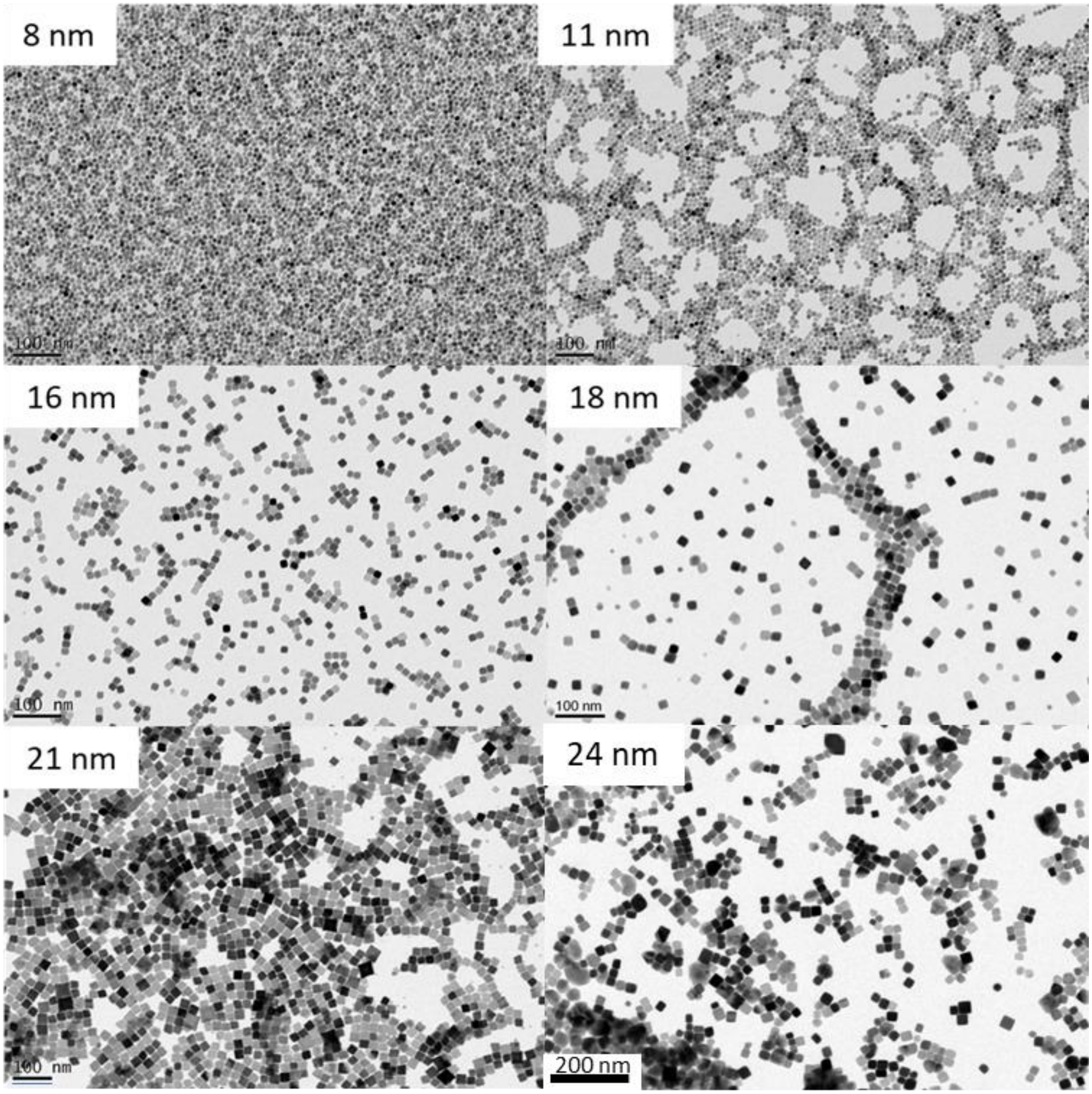
Supplementary Figure 13. TEM images of the MNPs obtained by replacing the amine hexadecylamine with secondary amines, i.e., dioctylamine (a) and didodecylamine (b). The shape of the MNPs evolves from a nanocube to star-like but the edges are not as sharp as in the case of synthesis in presence of tertiary amines as reported in **Figure 8**.



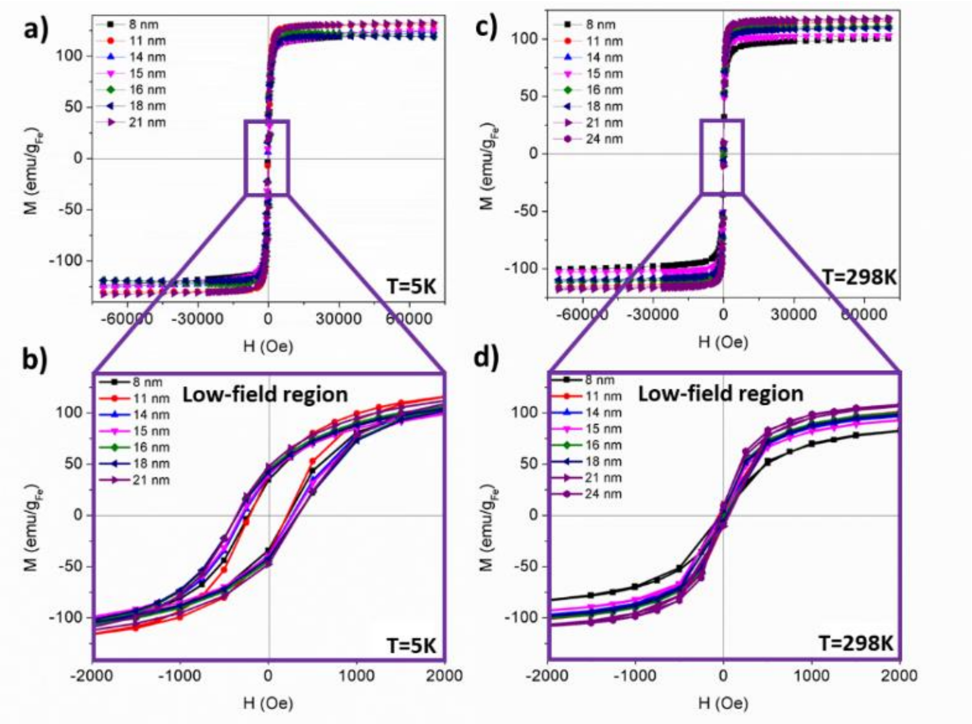
Supplementary Figure 14. XRD of star-like MNPs obtained by replacing hexadecylamine with oleylamine, showing their high crystallinity and ferrite structure. In grey, the theoretical peaks for the inverse spinel of Fe_3O_4 powder (JCPDS card no. 00-001-1111).



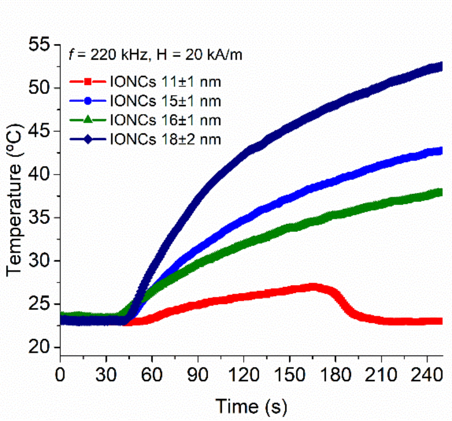
Supplementary Figure 15. Star-like shape nanoparticles through the use of benzaldehyde and different alkyl amines combinations: a) Comparative SAR values at different field conditions (12, 16 and 24 kA/m at f=200 kHz) for star-like nanoparticles and similar size nanocubes: 14 and 16±1 nm cubic IONPs against star-like IONPs of 15±1 nm (turquoise and blue, respectively). The star-like nanoparticles have higher SAR values than the nanocube of similar size. b) Hysteresis cycles at 5K recorded for the samples. Inset ZFC/FC curves recorded for these samples.



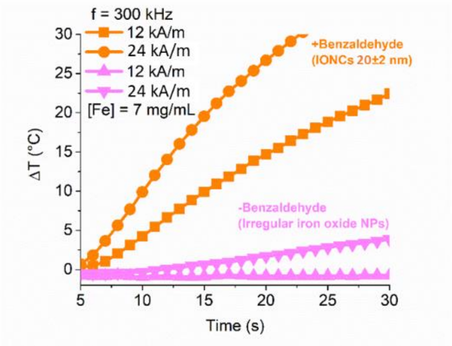
Supplementary Figure 16. TEM images at low magnification of the series of IONCs obtained according to **Table 3** and **Figure 3D**, including selected IONCs of the following sizes: 8 ±1 nm, 11±1 nm, 16±1 nm, 18±2 nm, 20±2 nm, 24±3 nm.



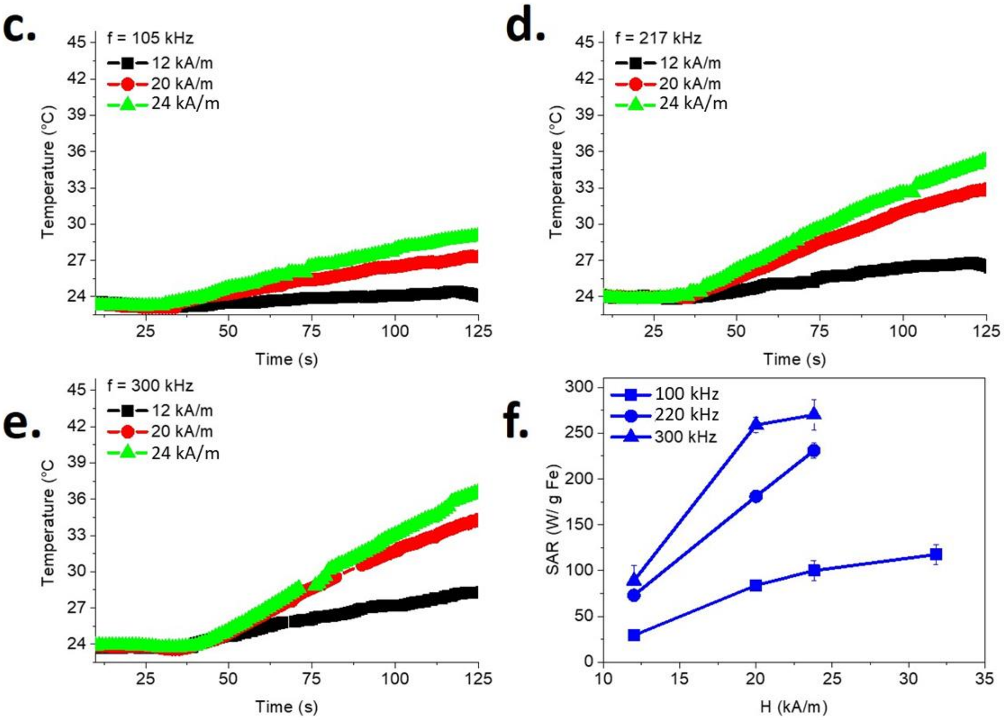
Supplementary Figure 17. SQUID recorded hysteresis cycles of the series of iron oxide nanocubes (IONCs) in the size range of 8-24 nm obtained at 5 (a,b) and 298 K (c-d). Graphs b and d show the low-field region (from -2000 to 2000 Oe) at 5 and 298 K, respectively. IONCs are superparamagnetic at room temperature (RT) and present high values of saturation magnetization (98-117 emu/gFe at RT and ~120 emu/gFe at 5 K).



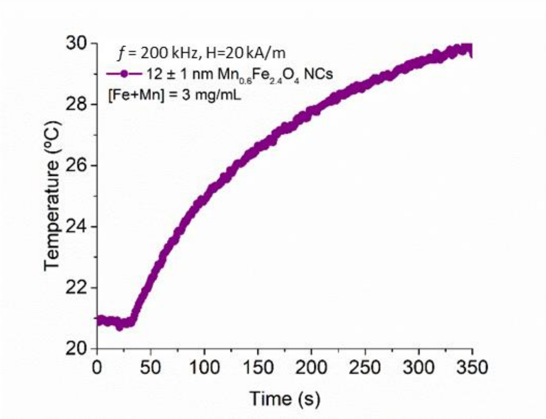
Supplementary Figure 18. Temperature versus time curve recorded for aqueous solutions of IONCs of different size (11-18 nm) under AMF of f= 220 kHz and H = 20 kA/m recorded on 300 µL of sample at [Fe] = 3-7 mg/mL.



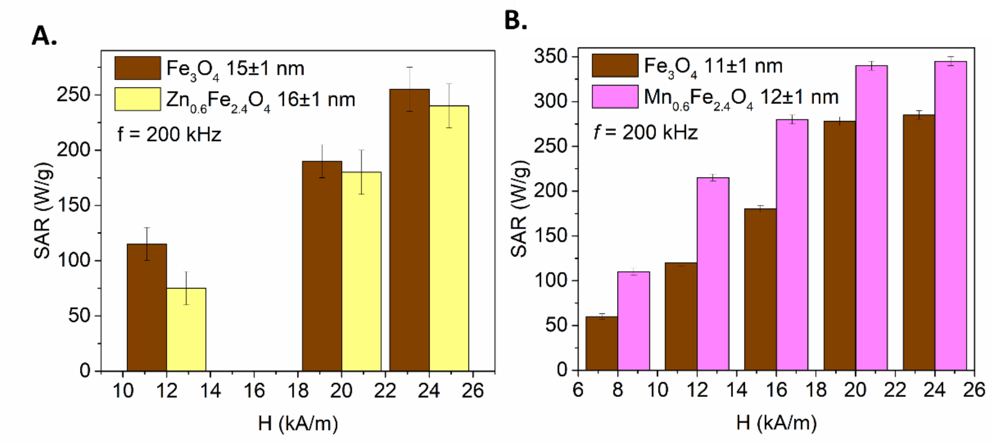
Supplementary Figure 19. Temperature versus time curves recorded for solutions of iron oxide nanoparticles ([Fe]=7mg/mL) obtained in the absence of benzaldehyde (pink) or in the presence of benzaldehyde (orange) under the exposure of AMF (H = 12-24 kA/m and *f*=300 kHz).



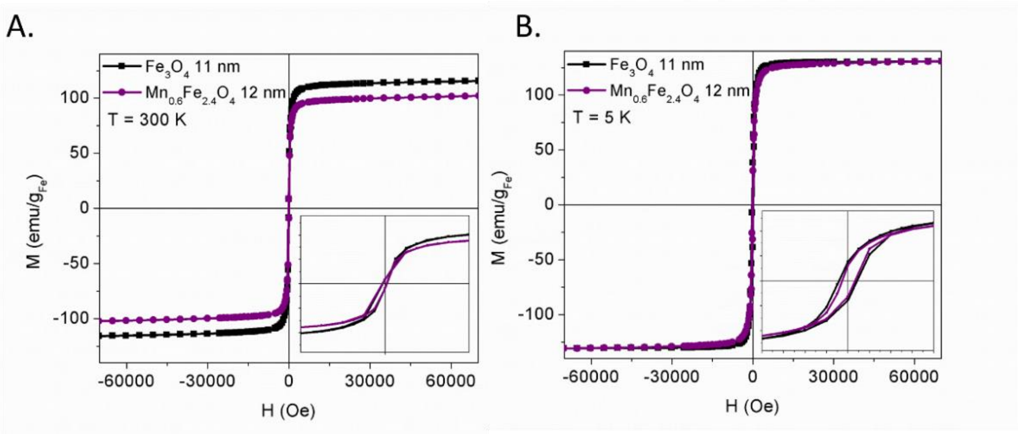
Supplementary Figure 20. (a-c) Temperature versus time curves recorded for aqueous solution of Zn-ferrite nanocubes of 16 ±1 nm under AMF of f= 105, 217 and 300 kHz and H = 12, 20 and 24 kA/m. (d) SAR values determined for each field conditions at nanocube concentration of [Fe+Zn] = 2.45 mg/mL.



Supplementary Figure 21. Temperature versus time curve recorded for aqueous solution of Mn-ferrite nanocubes of 12 ±1 nm under AMF of f= 220 kHz and H = 20 kA/m and nanocube concentration of [Fe+Mn] = 3 mg/mL.



Supplementary Figure 22. SAR values determined for aqueous solution of Zn-ferrite nanocubes (yellow bars) of 16 ±1 nm and Mn-ferrite nanocubes (pink bars) of 12 ±1 nm under AMF of f= 200 kHz and H = 8-24 kA/m. As a comparison, iron oxide nanocubes of similar size have been characterized using identical AMF conditions (brown bars).



Supplementary Figure 23. SQUID recorded hysteresis cycles of Mn-ferrite and IONCs of similar cube edge recorded at 300 K (A) and 5 K (B).

Tables

Table S1. Magnetic material mass for protocols at different experimental conditions.

Nanoparticles shape and size	Autoclave volume (mL)	Temperature (°C)	Autoclave filling percentage (%)	Mass Fe_3O_4 per autoclave (g)	Nº autoclaves per oven/Total Mass of MNPs in Fe_3O_4 produced (g)
Faceted/11±1nm	25	160	46	0.04	10/0.4
Faceted/13±1nm	25	180	46	0.08	10/0.8
Nanocube/ 20±2nm	25	200	46	0.1	10/1.1
Nanocube/ 24±3nm	25	240	46	0.4	10/4.4
Nanocube/ 8±1nm	50	200	23	0.3	8/2.5
Nanocube/ 10±1nm	50	200	40	0.26	8/2.1
Nanocube/ 10±1nm	50	200	55	0.25	8/2.0
Nanocube/ 12±1nm	50	200	67	0.22	8/1.7
Nanocube/ 10±1nm	100	200	46	0.70	6/4

Table S2. Comparison of the size of different samples of iron oxide nanocubes determined through TEM (DTEM) and XRD (DXRD), these latter cases by means of the Scherrer's equation according to Lak et al.4

D_{TEM} (nm)	**D_{XRD} (nm)**
20±2	21±3
16±1	15±4
11±1	13±3
8±1	8±1

Table S3. SAR determination through calorimetric (SAR Calor) and magnetometer (SARMagnet) measures at 200 kHz and 20 kA/m.

Sizes (nm)	**$SAR_{Calor.}$ (W/gFe)**	**$SAR_{Magnet..}$ (W/gFe)**
11	54 ±8	263 ±2
15	205 ±20	484 ±3
16	314 ±24	494 ±3
18	500 ±30	588 ±3

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