

Supporting information

One-pot Solvothermal Synthesis of FePt/Fe₃O₄ Core-Shell

Nanoparticles

By Chih-Wei Lai, Yu-Hsiu Wang, Borade Prajakta Uttam, Yu-Chun Chen, Jong-Kai Hsiao*, Chien-Liang Liu, Hon-Man Liu, Chun-Yen Chen* and Pi-Tai Chou*

Experimental section

Solvothermal synthesis FePt and FePt/Fe₃O₄ MNPs:

According to the synthesis procedure published by Li and Sun *et al.*, the spherical shapes of FePt and FePt/Fe₃O₄ MNPs are well controllable. Based on a colloidal template, we prepared FePt nanoparticles through mixing Fe(CO)₅ (0.065 ml, 0.5 mmole) and Pt(acac)₂ (49.2 mg, 0.125 mmole), precursors for Fe and Pt, respectively. Pt(acac)₂ was further reduced by hexadecane-1,2-diol (258.5 mg, 1 mmole). Sequentially, 8 mL phenyl ether solvent was added, followed by the injection of oleic acid (0.08 ml, 0.6 mmole) and oleylamine (0.08 ml, 0.6 mmole). These steps were all accomplished under room temperature in a glovebox. The colloidal precursor solution was then placed into a 10 mL Teflon-line stainless steel autoclave and gradually heated to 250 °C. Three different heating times were performed: 1hr, 3hr, and 6hr. After the heating process was completed, the autoclave was slowly cooled down to room temperature to yield final product. Centrifugation and re-precipitation from ethanol was executed twice for further modification.

Synthesis of water-soluble FePt/Fe₃O₄ MNPs:

The water-soluble FePt/Fe₃O₄ MNPs were prepared by using modified version of the stepwise procedures reported by Chen *et al.* Briefly, oleic acid/oleylamine-capped FePt/Fe₃O₄ MNPs (20 mg) were mixed with (200 mg, 1.1 mmole) 2, 3-dimercaptosuccinic acid (DMSA) in methanol (15 mL, 99%) in a reaction vessel under room temperature. After the pH value was adjusted to > 10 by using tetramethyl ammonium hydroxide pentahydrate, the original FePt/Fe₃O₄ MNPs was stirred in the mixture under N₂ flow and dark conditions. The mixture was continuously heated by refluxing at 65 °C overnight. After the reaction was terminated, the mixture was slowly cooled down to room temperature. The DMSA-capped nanoparticles were then

precipitated with diethyl ether. For further purification, methanol was added to dissolve the precipitant, followed by the addition of diethyl ether to reprecipitate the nanoparticles. The final product, DMSA-capped FePt/Fe₃O₄, possessed great water-solubility.

Measurement:

The as-prepared nanoparticles were characterized with a Hitachi H-7100 transmission electron microscope, a powder X-ray diffractometer (model PANalytical X'Pert PRO), and a high-resolution transmission electron microscope (FEI Tecnai G2) including EDX spectroscopy and a CCD camera with Diffpack program. SQUID (Quantum Design MPMS-XL7) was used for detecting the magnetic properties. Hydrodynamic radius was measured with a DLS instrument (Malvern Zetasizer 3000 HS).

Culture of human mesenchymal stem cell line: The cell line was cultured in Dulbecco's modified Eagle's medium (DMEM) (Cellgro, Herndon, VA, USA), supplemented with 10% heat-inactivated fetal bovine serum (FBS), penicillin (50 U/ml), and streptomycin (0.05 mg/ml). All cultures were kept in atmosphere of 5% CO₂, 95% air at 37°C.

MR imaging in vitro: MR imaging was performed using clinical 1.5 T MR System (Signa excite, GE Healthcare, USA). The cell samples were centrifuged in 200 μ L test tubes and placed in a water tank. The tank was then placed in an 8 channel head coil. Two dimension T2-weighted gradient echo pulse (GRE) sequences were used (TR/TE= 550/15 ms, FA=15). The slice thickness was 1.4 mm with .03 mm gap and the field of view (FOV) was 14*10.5cm. Total scan time was 4 minutes and 43 seconds at the NEX of 3. The images were then analyzed at the workstation provided by GE healthcare.

In vitro cytotoxicity test: cells were seeded in a 24-well plate at 1×10^4 cells/well density in 500 μ L culturing medium for 24 hours prior to particles feeding. Different amount of particles were given to each well to reach the final concentration of 0, 6.25, 12.5, 25, 50, and 100 μ g/mL. After 48 hours of incubation, each well was washed with phosphate buffer saline (PBS: 137 mM NaCl, 2.68 mM KCl, 10 mM Na₂HPO₄, 1.76 mM KH₂PO₄, pH 7.4) for two times, and replenished with 500 μ L culturing medium containing 10% MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) agent (Sigma). The medium was removed 2 hours later. Newly formed purple MTT-formazan was dissolved in 200 μ L dimethyl sulfoxide (Sigma) and the

absorbance was measured at 570 nm by a microplate reader (Tecan Infinite F200).

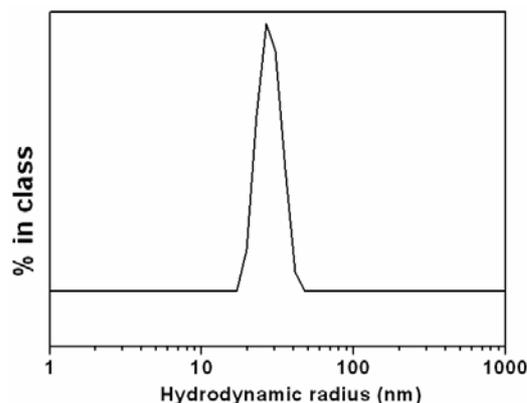


Figure S1. Dynamic light scattering of FePt/Fe₃O₄/DMSA nanoparticles suspended in DI water.

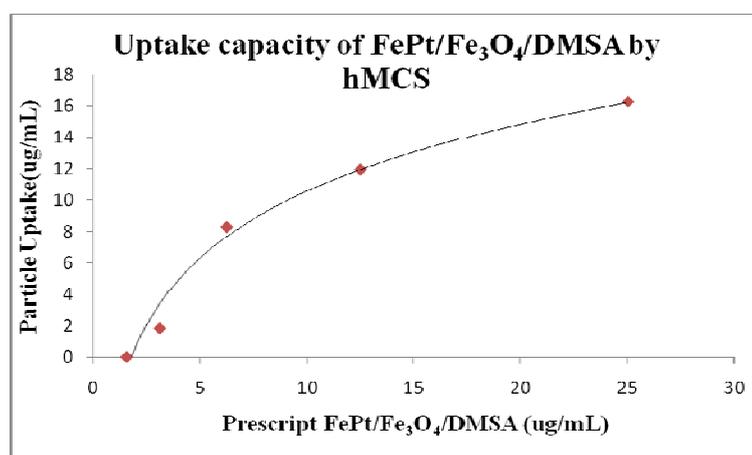


Figure S2. Uptake capacity of FePt/Fe₃O₄/DMSA by hMCS.

Cellular uptake in vitro: Capacity of FePt/Fe₃O₄/DMSA internalization by hMCS was quantified by measuring the iron content. Total of 4 cell samples were respectively pre-treated with 0, 6.25, 12.5, and 25 μg/mL of FePt/Fe₃O₄/DMSA MNPs for 24 hours, where 1×10^4 cells per sample was adopted. Cell samples were then harvested through trypsinization and lysed by heating with 2% nitric acid on a 97 °C heatblock overnight. After centrifuging at the speed of 1,000 rpm for 3 minutes, the pellet portions containing the cell debris were discarded and the supernatant collected were further centrifuged at the speed of 10,000 rpm for 30 minutes to collect the particles. The resulting pellets were re-suspended in deionized water, and the iron content in each sample was determined by taking the absorbance at wavelength 247.3 nm using a UV/Visible spectrophotometer (Beckman DU 800). External standards with concentration of 0, 6.25, 12.5, and 25 μg/mL were employed for calibration, with coefficient of determination being 0.998. Clearly, a rising exponential-like plot for

particle uptake versus prescript FePt/Fe₃O₄/DMSA was obtained, the result of which qualitatively correlates with the dose dependent manner observed in MRI study.