Supplementary Information

Synthesis, decoration, and cellular effects of magnetic mesoporous silica nanoparticles

J. L. Nyalosaso et al



SI1: Scheme of each step of the synthesis, from Fe_3O_4 magnetic iron oxide nanocrystals (MIONs) to native core shell Fe_3O_4 @MSN.

Synthesis of Fe₃O₄ nanocrystals (MIONs)

0.18 g of hydrated iron oxide (II) was mixed with 5 g of docosane and 3.2 g of oleic acid in a 100 mL two necked round bottom flask. Prior to reaction, the mixture was stirred under vacuum for 30 min, and then placed under Ar flow for 30 min. The thermal decomposition of iron oxide occurred under reflux at 340 °C for 1h30. After completion of the reaction, the dark mixture was cooled down slowly without stirring to a temperature of 50 °C and then collected in a beaker. At room temperature, 15 mL of pentane and 30 mL of the mixture ether: ethanol (2: 1, v: v) were added to the reaction mixture. This black suspension was ultrasonically dispersed for 2 min and then centrifuged for 10 min at 35,700 g (20 krpm). The collected black solid was washed, sonicated and centrifuged a first time with 2 mL of pentane plus 30 mL of the mixture ether: ethanol (2: 1, v: v), and a last time with 30 mL of the mixture ether: ethanol (1: 1, v: v). Finally, the black magnetic Fe₃O₄ nanocrystals were stored in 15 mL of chloroform and stabilized by adding 200 μ L of oleylamine, to form a homogenous solution.

Synthesis of Fe₃O₄ @MSN

Typically, 0.125 g of CTAB was placed in a 250 mL three-necked round bottom flask containing 60 g of ultrapure water and 0.44 mL of a solution of NaOH (2 M). This micellar solution was then heated to 70 °C under vigorous stirring (750 rpm). After 1 hour stabilization, 0.5 mL of the suspension of MIONs, previously dispersed with ultrasonics for 3h, was injected stepwise (10 x 50 μ L) into the micelle solution during 50 minutes. Between each injection, a partial and brief vacuum of 2 seconds was applied followed by a gas stream (Ar or air) in order to remove the chloroform (MIONs solvent) from the micellar solution. Following these injections, the reaction mixture was heated to 80 °C. At this temperature, a vacuum was one more time applied briefly followed by a gas stream. This was repeated once again before leaving the reaction mixture to stabilize under stirring for 1 hour at 80 °C. Then, an initial amount of 0.1 mL of TEOS was added to the mixture. And finally, after 30 min of reaction, an additional 0.5 mL of TEOS was added to complete the sol-gel reaction. The condensation process was conducted for 1h30.

After reaction, the mixture was slowly cooled down at room temperature while stirring, and then collected in NalgeneTM tubes for centrifugation at 5,200 g (7.5 krpm) for 10 minutes. This first centrifugation at 5.2 kg was carried out in order to extract more dense multicore silica particles that may be present in the sediment. Then, the supernatant was centrifuged a second time at 35,700 g (20 krpm) for 10 minutes in order to collect the solid. To extract efficiently the

surfactant from the nanoparticles, the solid was redispersed in a 100 mL round flask containing 50 mL of an alcoholic solution of nitrate ammonium (6g/L in EtOH) and was refluxed at 80 ° C for 2 h, then at 60 ° C overnight. The sample was centrifuged at 35,700 g for 10 minutes and washed 4 times consecutively with 30 mL of EtOH, twice with water, then EtOH. Each washing step involved 15 minutes of sonication and a centrifugation operation at 35,700 g for 10 minutes. Finally, the sample was dried for 4 hours in vacuum at room temperature.



SI2: TGA of Fe₃O₄ nanocrystals (MIONs).



SI3: Wide-angle X-Ray diffraction pattern of native Fe₃O₄@MSN core-shell nanoparticles.



SI4: N₂ sorption isotherms of native Fe₃O₄@MSN (● Adsorption, O Desorption) and PEG- Fe₃O₄@MSN (■ Adsorption, □ Desorption)



Applied magnetic field / Oe

SI5: Magnetization curve of Fe $_3O_4@MSN$ at 300 K



SI6: Zero field cooling (ZFC) and field cooling (FC) curves of Fe₃O₄@MSN measured with a field of 100 Oe on a superconducting quantum interference device (SQUID) magnetometry.



SI7: TEM images of PEG-grafted Fe $_3O_4@MSN$ core-shell nanoparticles



SI8: STEM images of pristine and DMPC Fe₃O₄@MSN. a) Pristine Fe₃O₄@MSN overlay of TEM black field, iron, silica, phosphorus element cartography (presented separately on images 1, 2, 3, and 4, respectively). Phosphorus appears on the cartography but corresponds to background, because any phosphorus is quantified. b) DMPC- Fe₃O₄@MSN overlay of TEM black field, iron, silica and phosphorus element cartography (presented separately on images 1, 2, 3, and 4, respectively). Phosphorus is located around the silica shell of Fe₃O₄@MSN. Quantitative analysis of STEM images associated with EDX element spectra: for pristine Fe₃O₄@MSN, no phosphorus is quantified, so the phosphorus appearing on the STEM image is in the background. On the contrary, phosphorus is quantified for DMPC- Fe₃O₄@MSN.