



Silica rod-shape capsules embedding superparamagnetic iron oxide nanoparticles

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Zaragoza

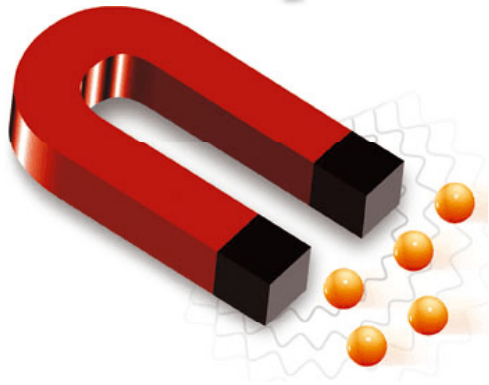
OUTLINE

- ***INTRODUCTION***
- ***PREPARATION OF SILICA COATED MAGNETIC NANOPARTICLES***
 - **Synthesis of magnetic nanoparticles**
 - **Silica coating**
- ***RESULTS***
 - **Structure and colloid characterization**
 - **Magnetic properties**
- **CONCLUSIONS**

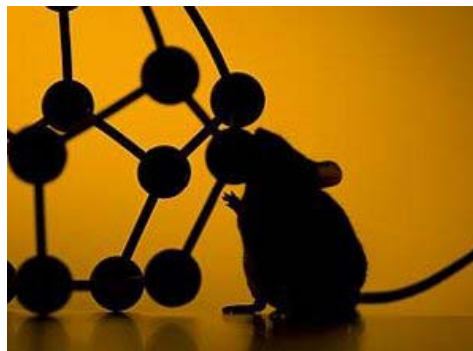
INTRODUCTION

IRON OXIDE NANOPARTICLES

Magnetic properties



Optimum size



Biocompatibility

INTRODUCTION

A great versatility ...

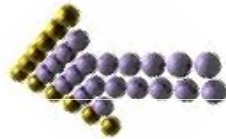
Drug delivery systems



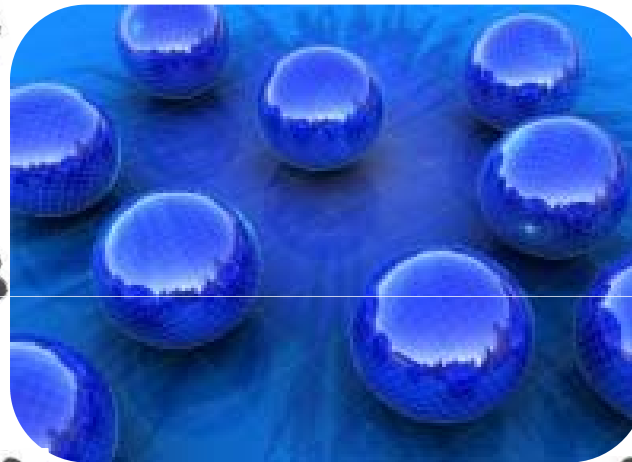
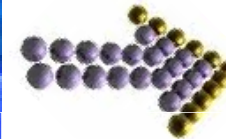
MRI contrast agents



Nano heat power sources



Magnetic immunoassay



Catalysis



Detoxification system



INTRODUCTION



Most drugs are orally or parenterally administered



Conventional administration

Partial degradation of the drug before reaching its destination

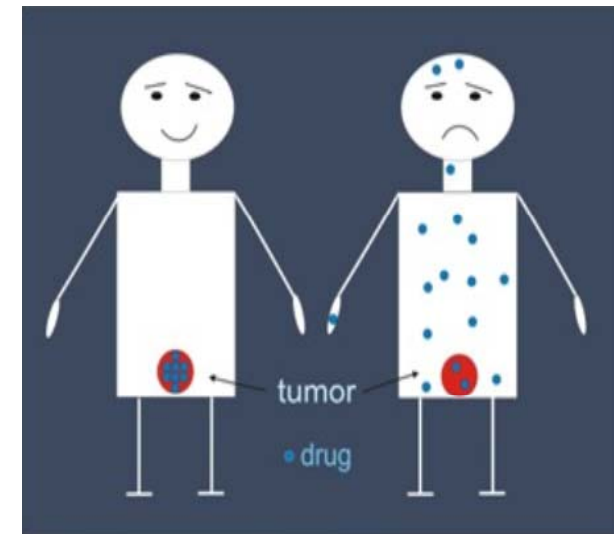
Reduction of the potential effects of the drug

Immediate release of the drug

Probable side or adverse effects

Need to improve the therapeutic properties of drugs

Directed system



INTRODUCTION

However...

 Low colloidal stability at physiological pH

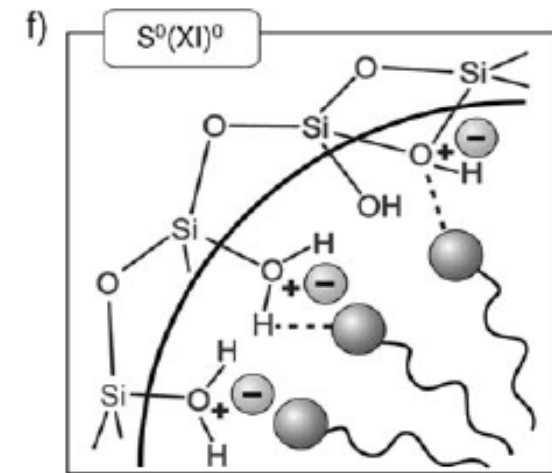
Surface treatment

Chemical stability

Reduced toxicity

Increased reactivity

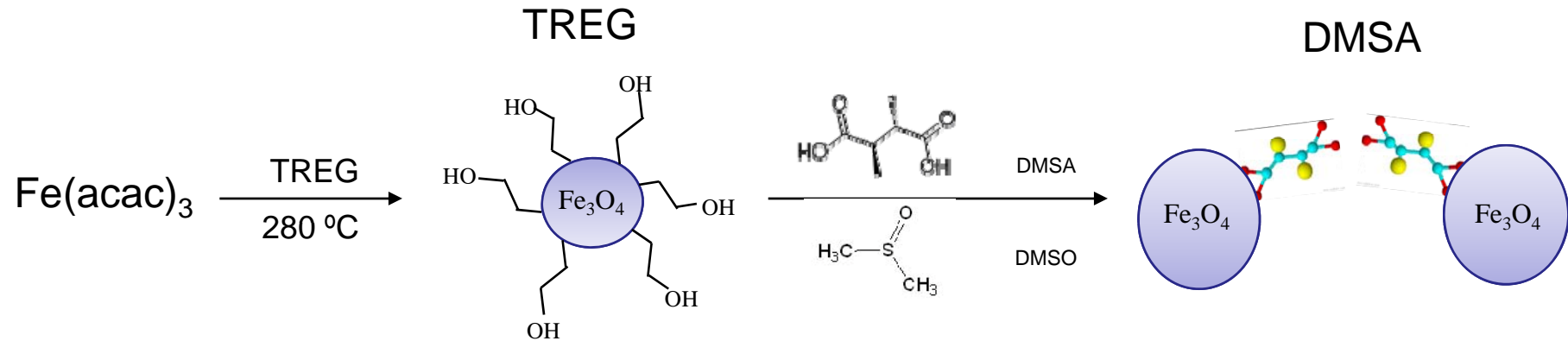
Hydrolysis and condensation of silica alcoxide precursors onto the particle surface provides several advantages



Final properties will depend on the type of aggregate that they form

PREPARATION OF SILICA COATED NANOPARTICLES

- SYNTHESIS OF MAGNETIC NANOPARTICLES**

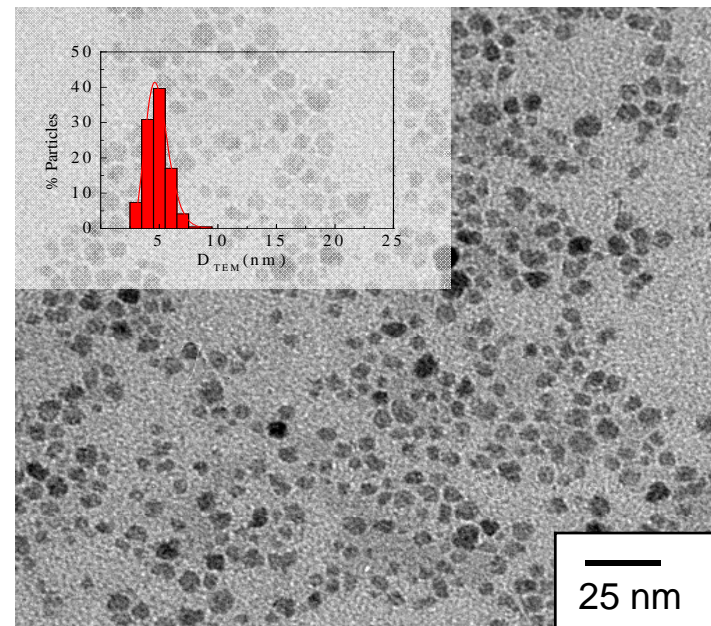


Wan, J.; W.Cai; Meng, X.; Liu, E. *Chem. Commun.* **2007**, 5004-5006

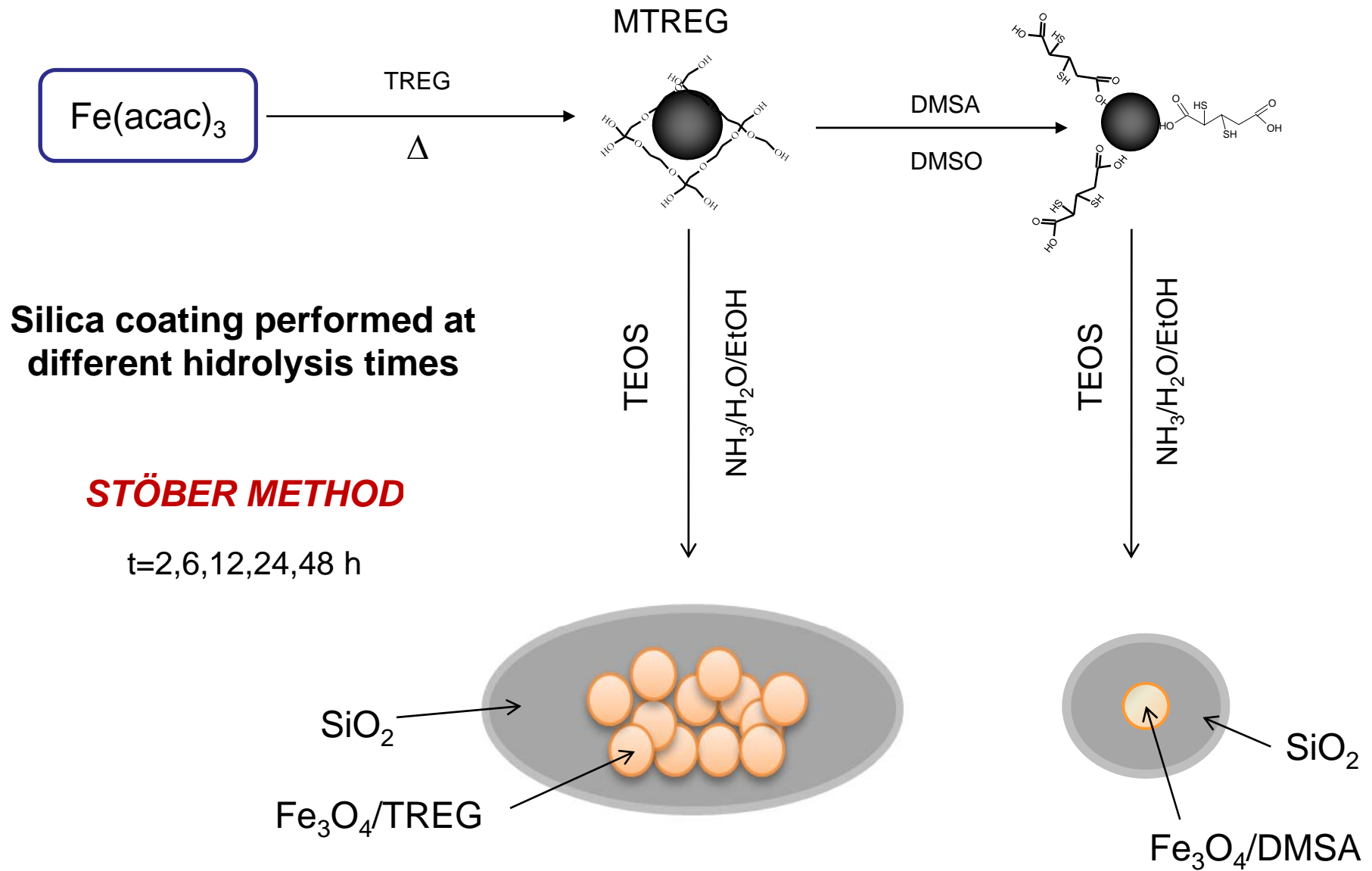
One-step synthesis

THERMAL DECOMPOSITION METHOD

$D_{\text{TEM}} = 4.8\text{ nm}$
 $W = 0.20$



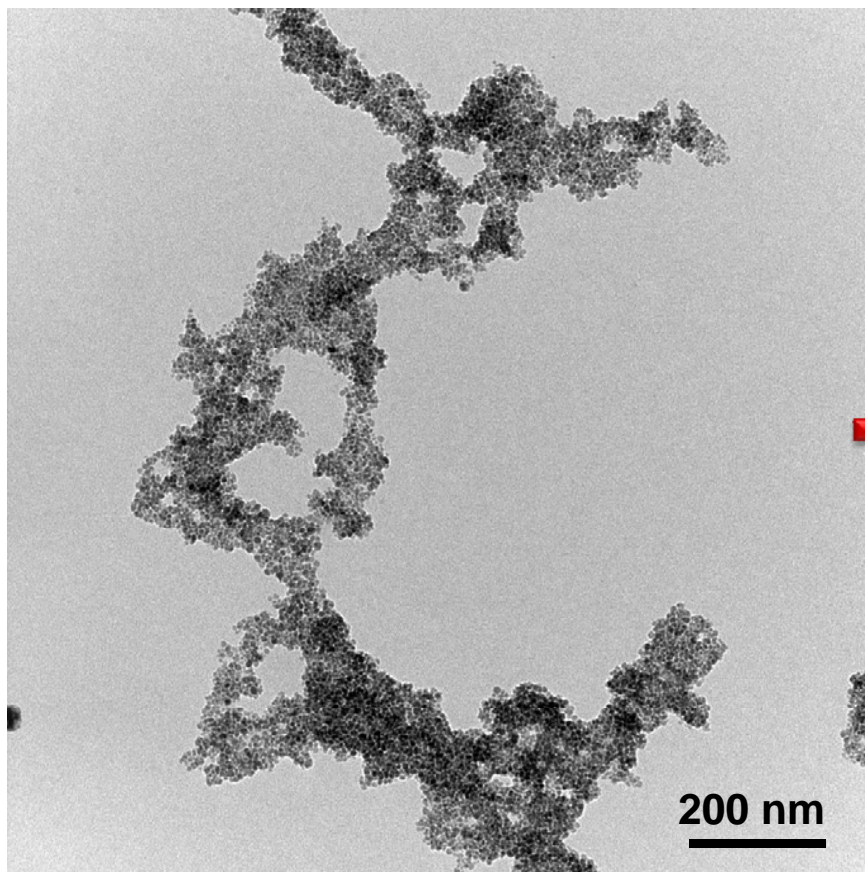
PREPARATION OF SILICA COATED NANOPARTICLES



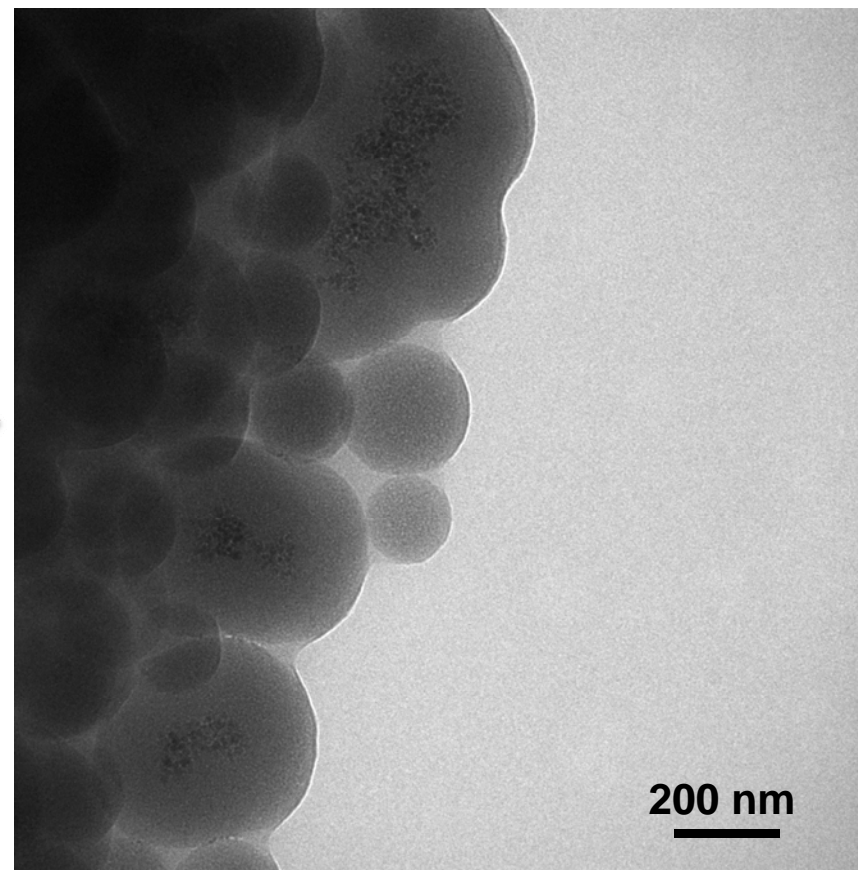
RESULTS

- **STRUCTURAL CHARACTERIZATION**

Maghemite/TREG



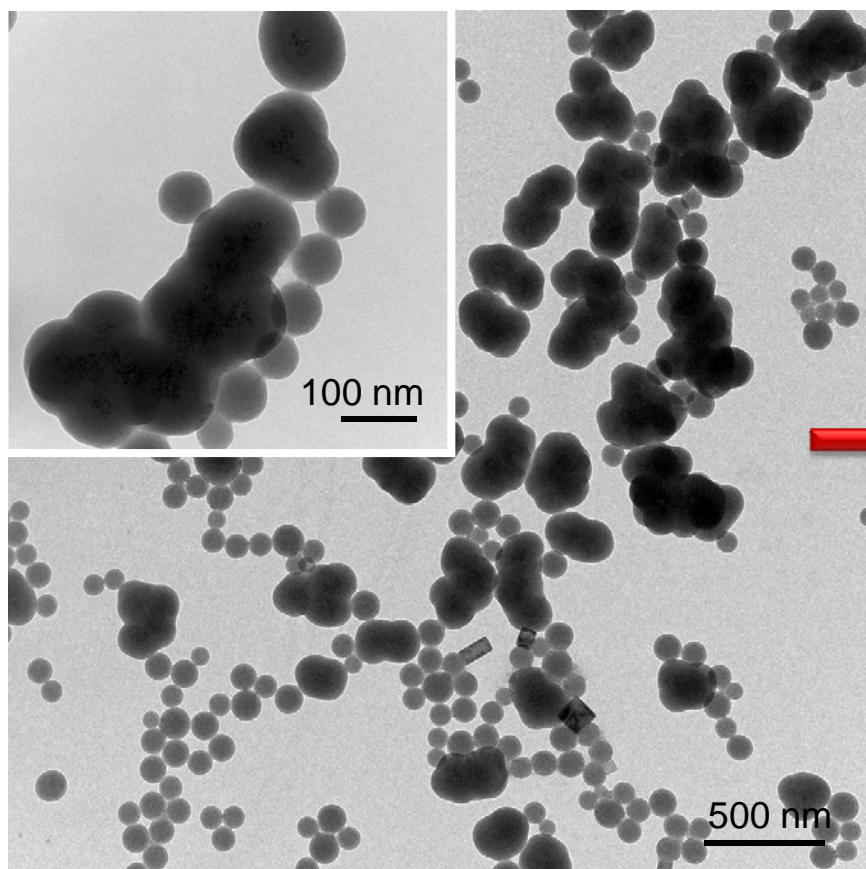
T2h



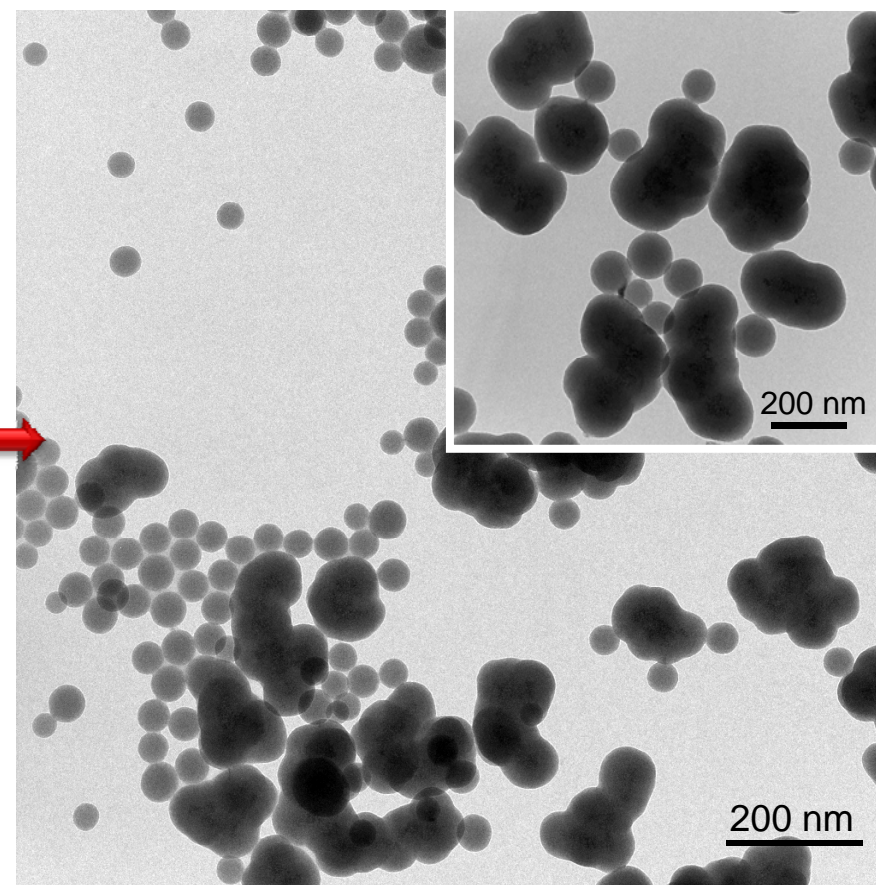
RESULTS

- **STRUCTURAL CHARACTERIZATION**

T6h



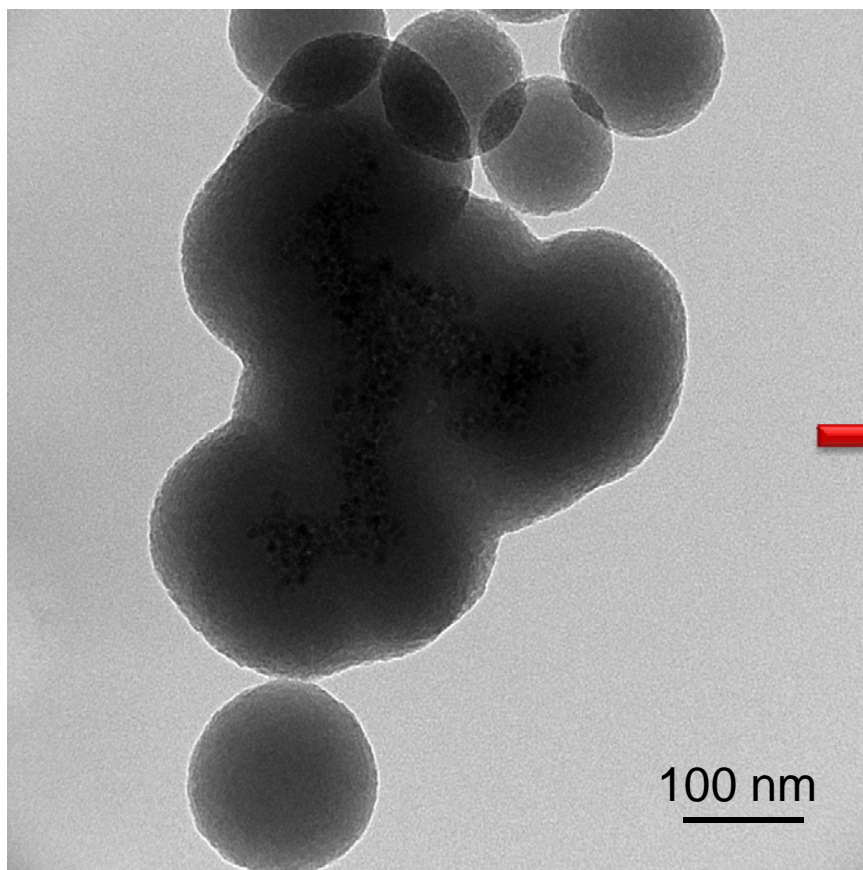
T12h



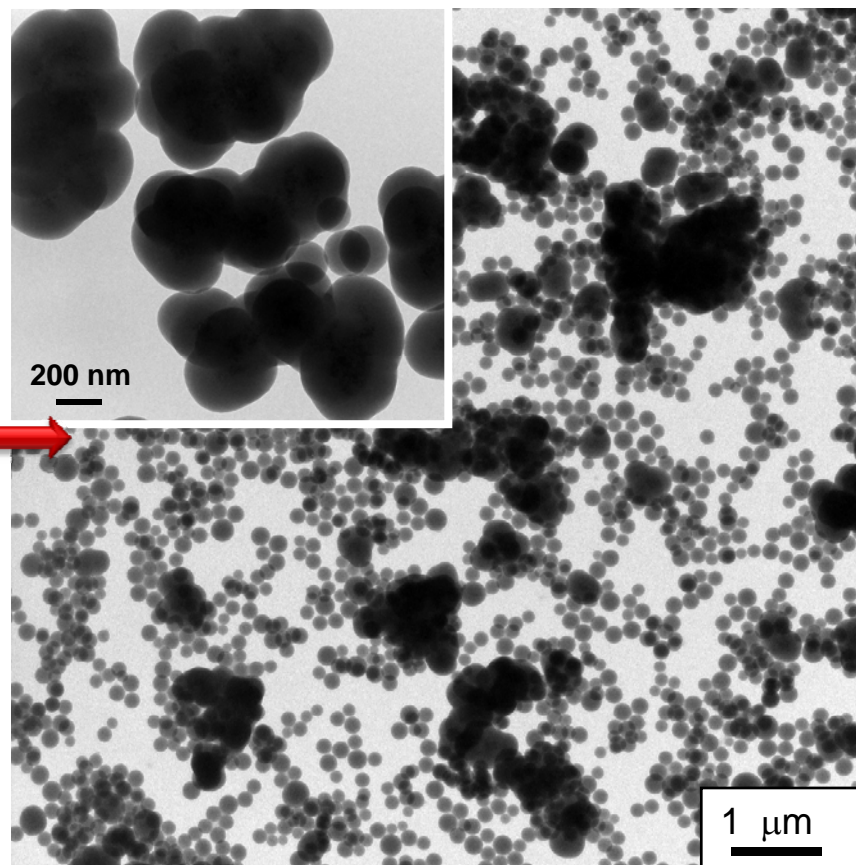
RESULTS

- STRUCTURAL CHARACTERIZATION**

T24h



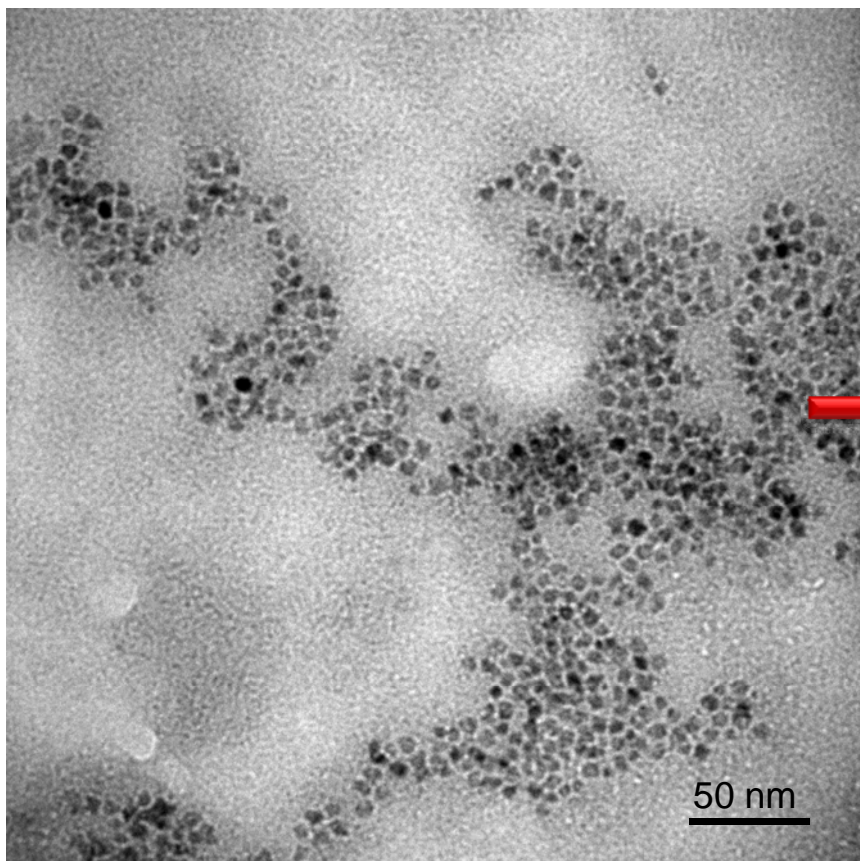
T48h



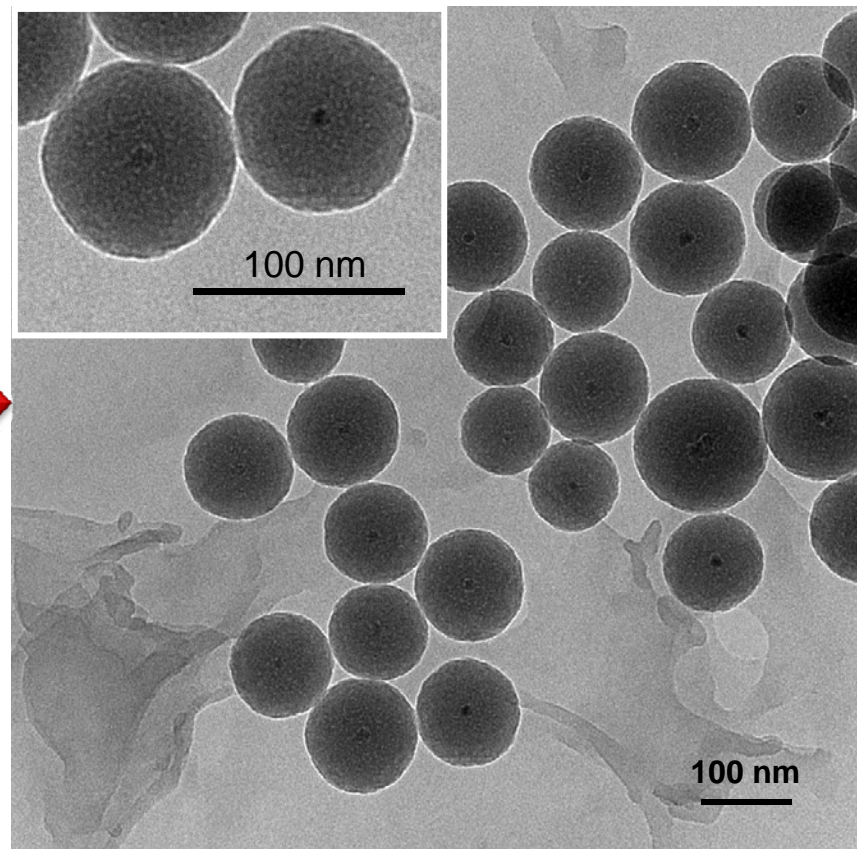
RESULTS

- **STRUCTURAL CHARACTERIZATION**

Maghemite/DMSA

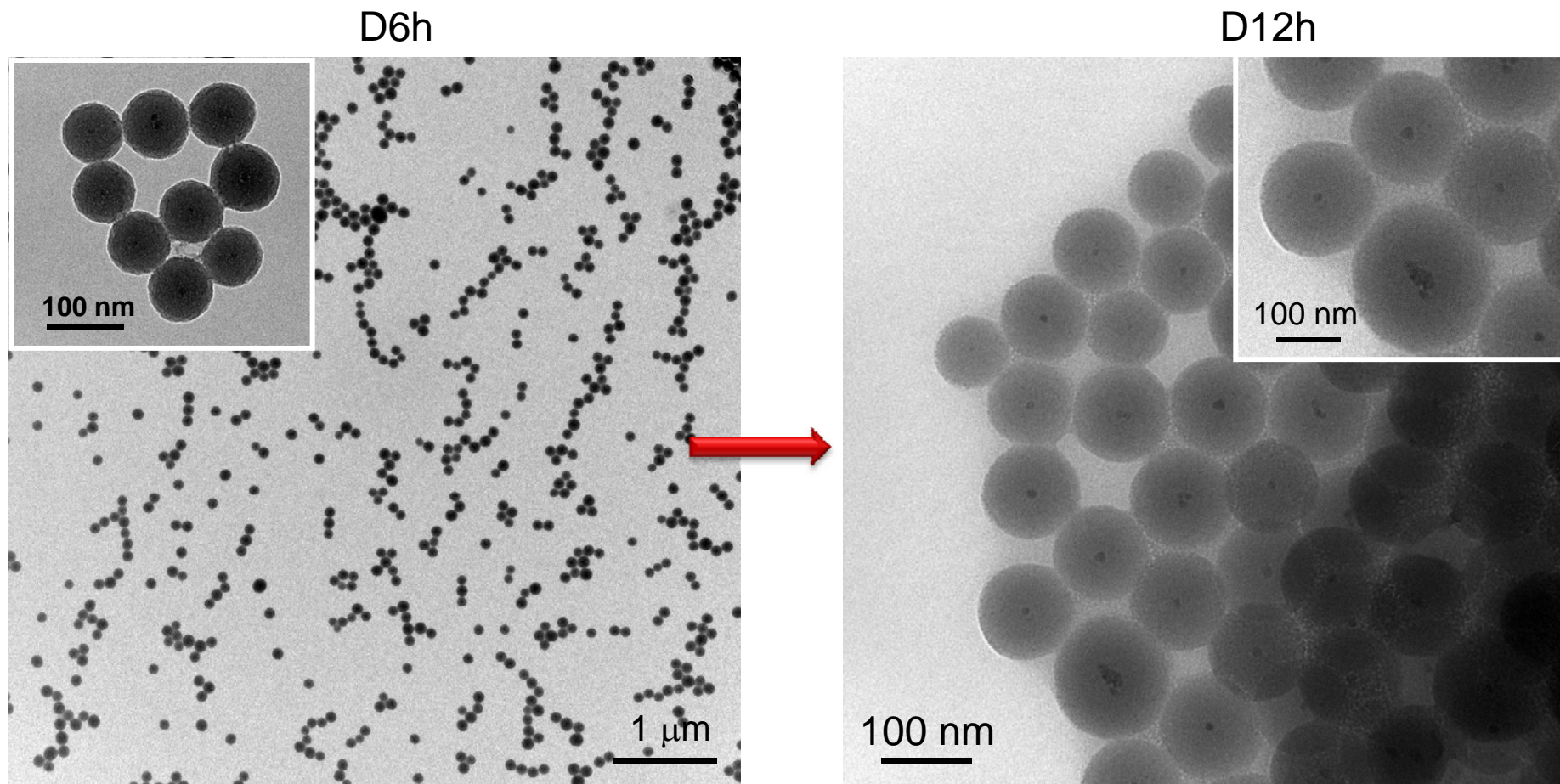


D2h



RESULTS

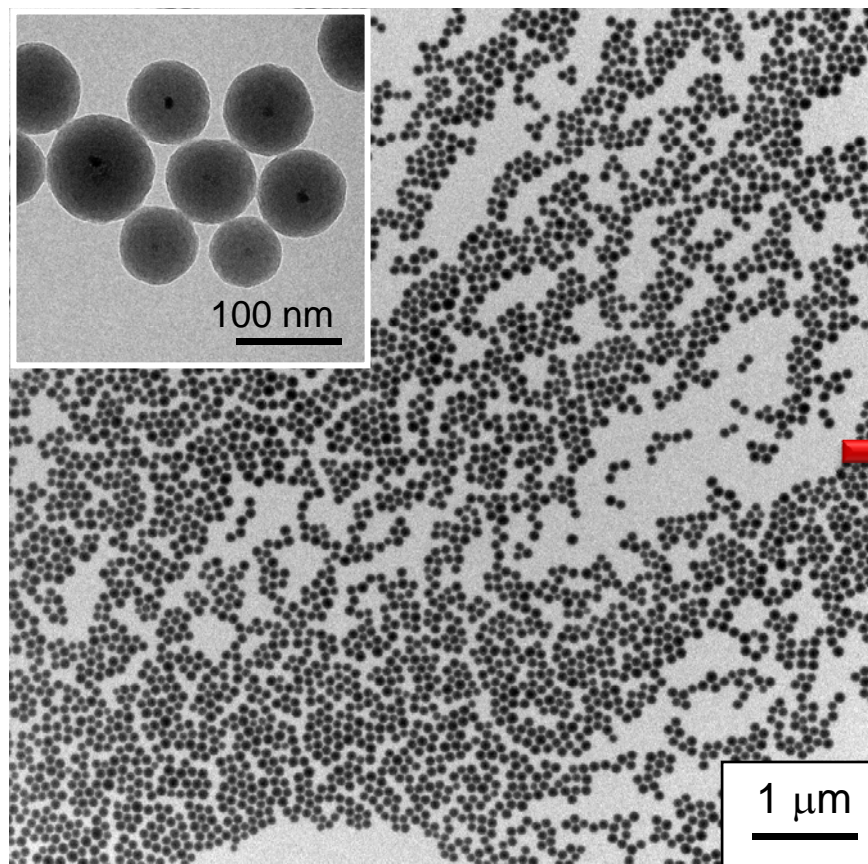
- **STRUCTURAL CHARACTERIZATION**



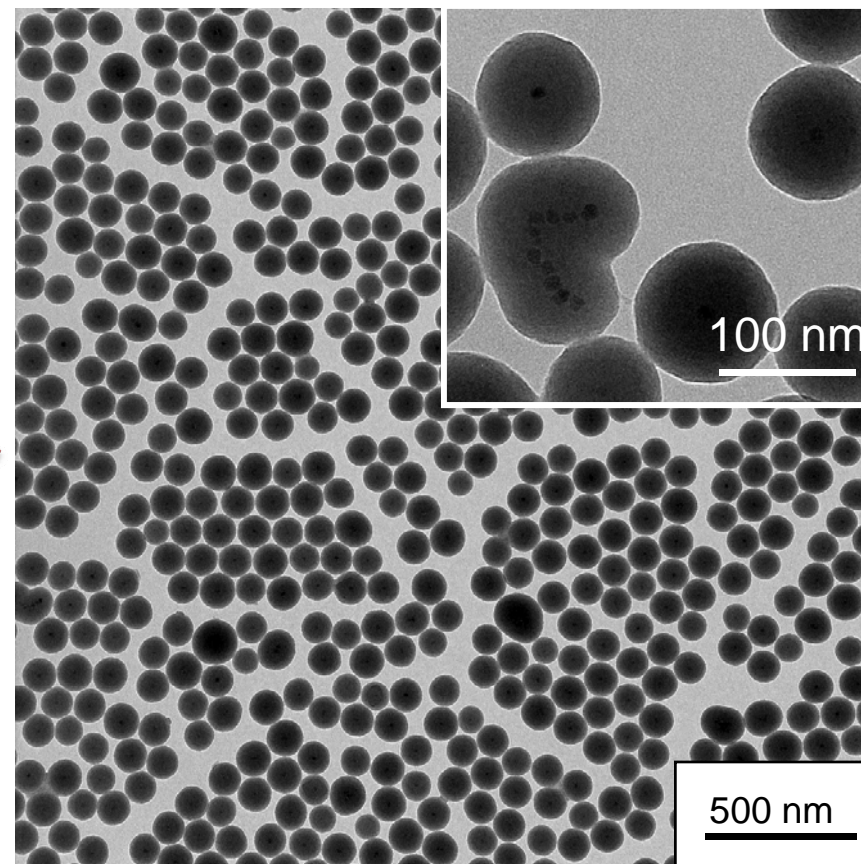
RESULTS

- STRUCTURAL CHARACTERIZATION**

D24h

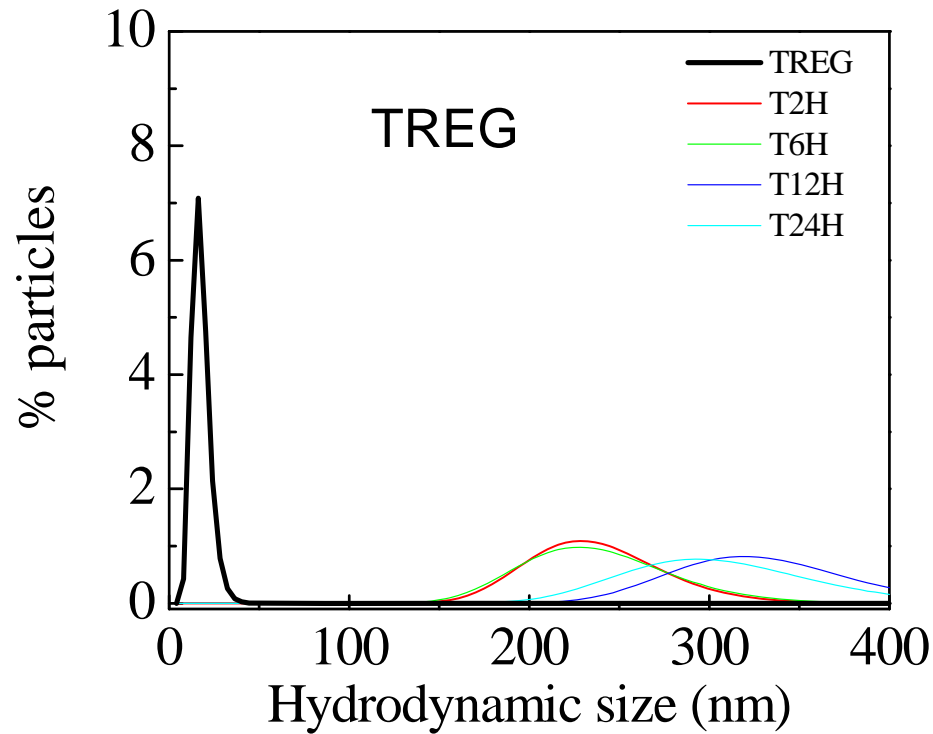


D48h

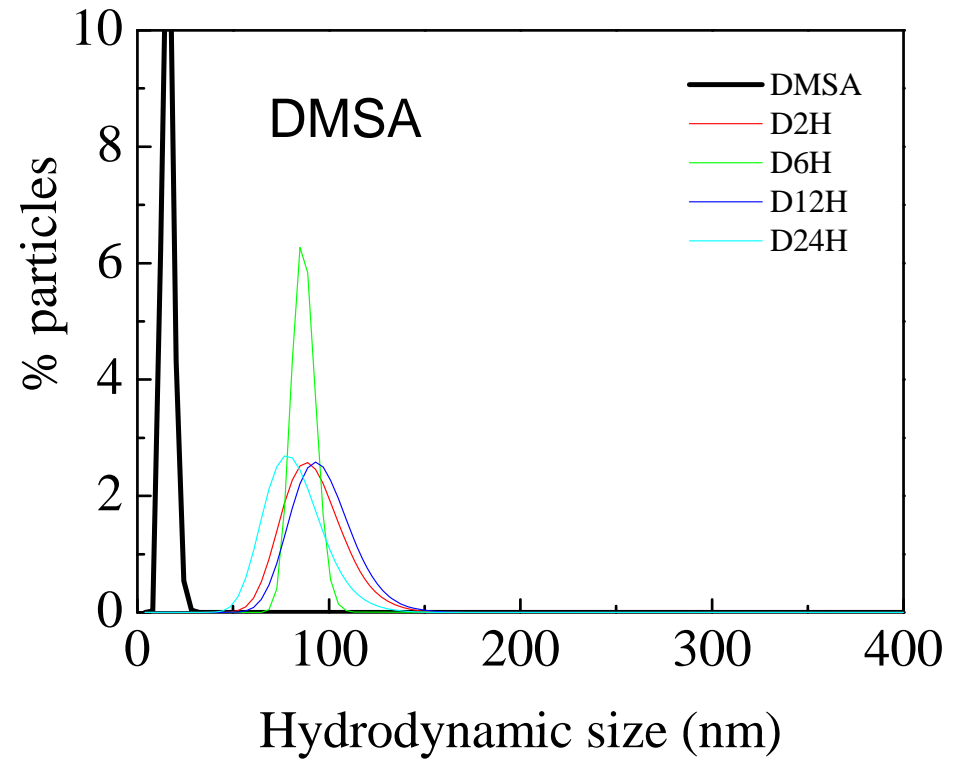


RESULTS

- COLLOIDAL CHARACTERIZATION**



Aggregation of the SiO₂ capsules with the hydrolysis time

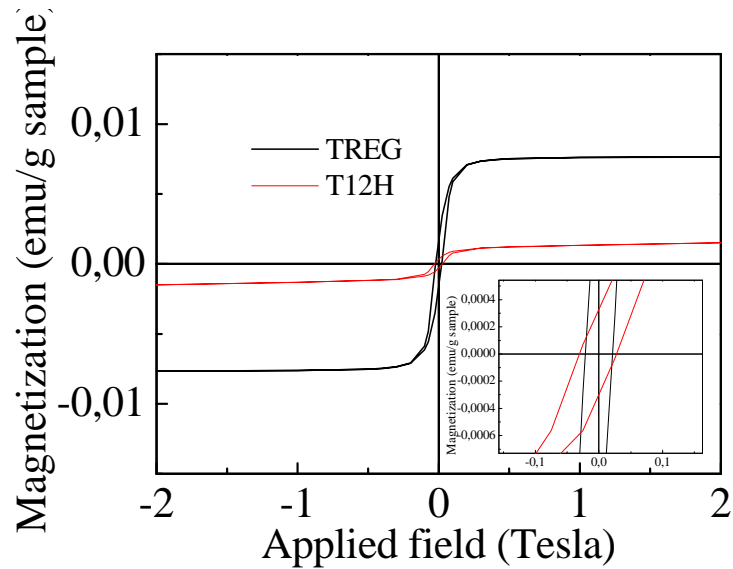


No variation of the hydrodynamic size with the hydrolysis time

RESULTS

- MAGNETIC PROPERTIES**

MAGNETIZATION CURVES IN SUSPENSION COOLED TO 5 K

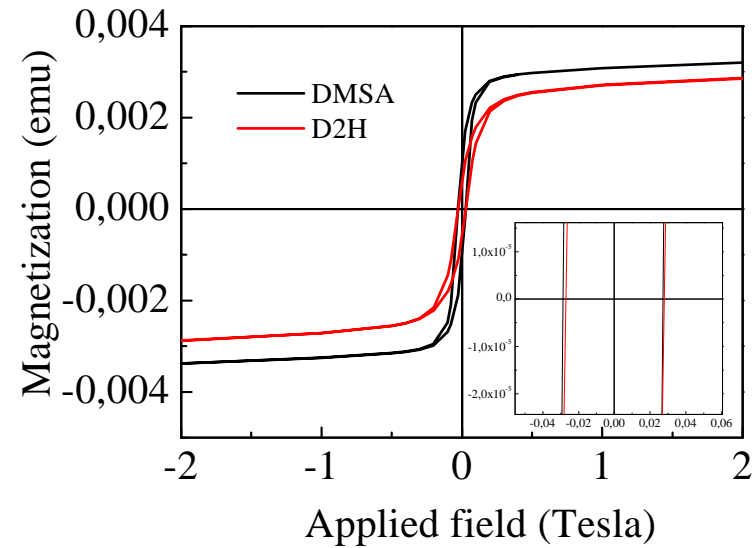


$$H_C(\text{T12H})=300 \text{ Oe}$$

$$H_C(\text{TREG})=150 \text{ Oe}$$

$$H_C(\text{T12H}) \gg H_C(\text{TREG})$$

Interactions between particles
increase after silica coating

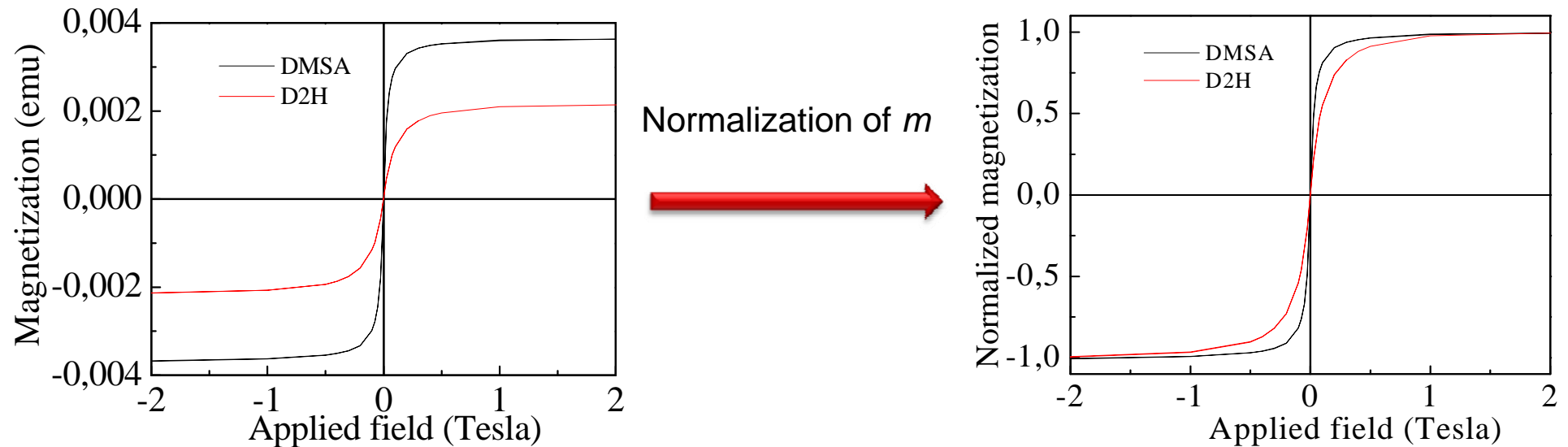


$$H_C(\text{DMSA}) \sim H_C(\text{D2H})=420 \text{ Oe}$$

RESULTS

- **MAGNETIC PROPERTIES**

MAGNETIZATION CURVES IN SUSPENSION COOLED TO 250 K

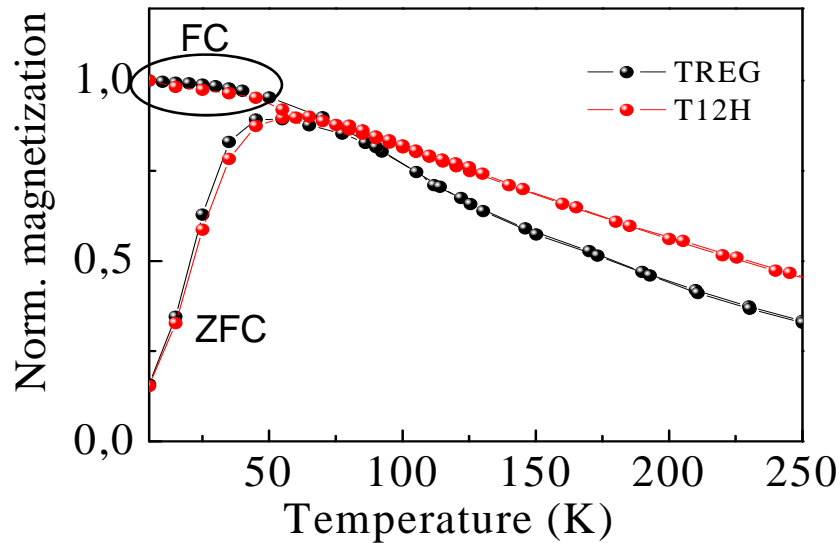


- Both samples have superparamagnetic behavior at 250 K
- Magnetization saturates at higher fields in D2H than DMSA due to the decrease of interactions

RESULTS

MAGNETIC PROPERTIES

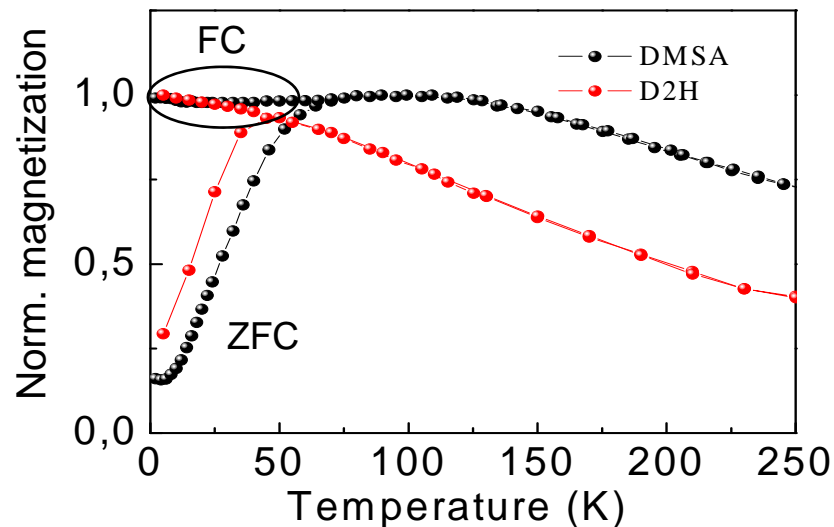
ZERO FIELD COOLING-FIELD COOLING



- Similar T_B for TREG and T12H
- Decrease in magnetization larger in TREG than T12H in the high-temperature region



Interactions



- D2H: Decrease of the T_B from 75 to 50 K
- Sharp magnetization decrease at low and high-temperature region in FC branch

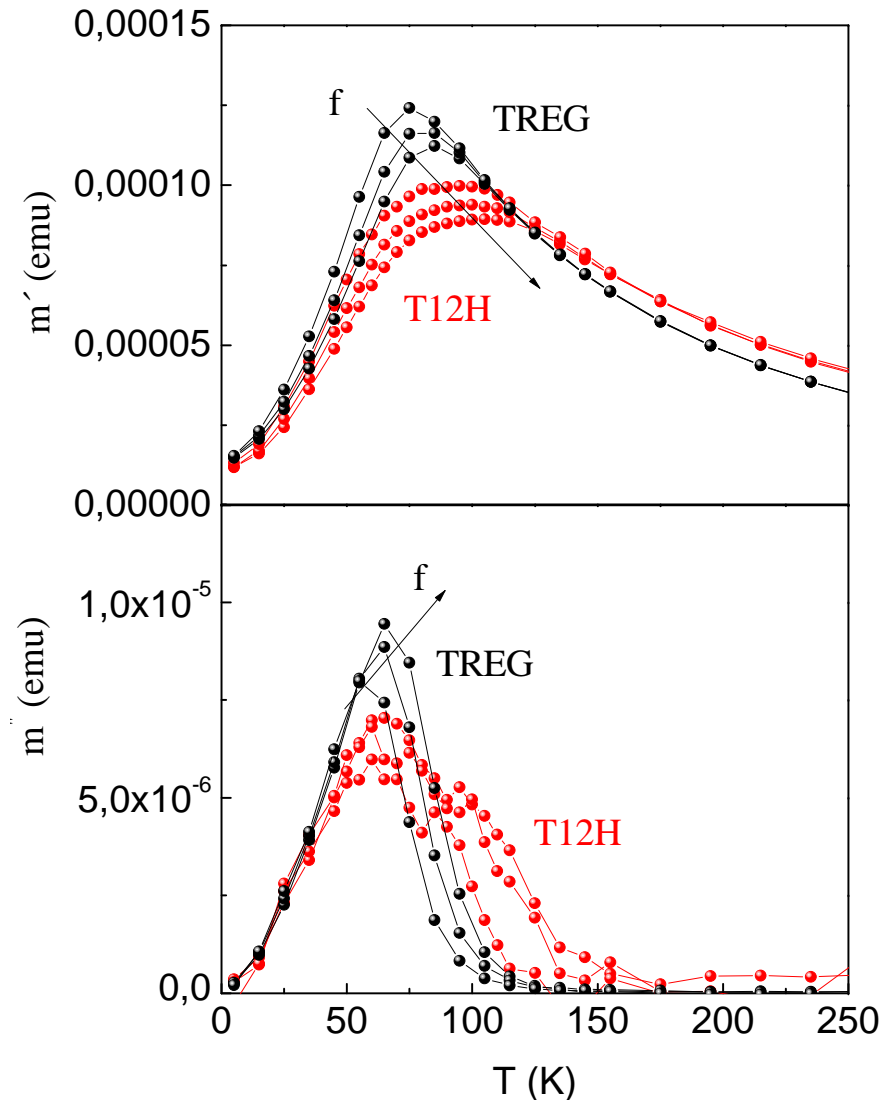


Interactions

RESULTS

MAGNETIC PROPERTIES

TREG & T12H



AC SUSCEPTIBILITY

χ_{ac} vs T f (5,50,240 Hz)

- Superparamagnetic behavior
- T_M increases with f

INTERACTION PARAMETER

RELATIVE SHIFT OF THE BLOCKING TEMPERATURE PER A FREQUENCY INTERVAL*

$$\phi = \frac{\Delta T_f}{T_f \Delta \log_{10}(f)}$$

$\phi(\text{TREG}) = 0,06$

$\phi(\text{T12H}) = 0,04$

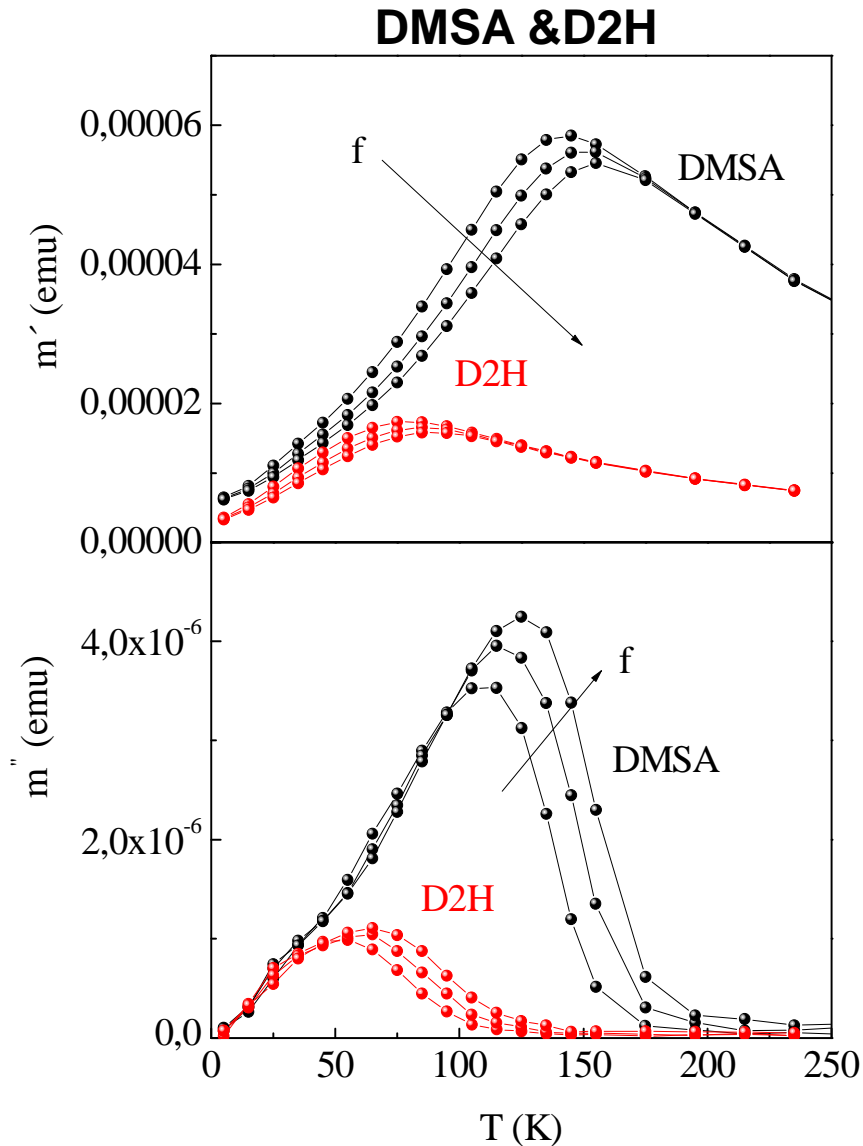
$\phi=0,1$ for superparamagnetic systems
 $\phi \sim 0,01-0,03$ for spin glass

Interactions increase after silica coating.
 The aggregate size of the particles is larger

*G.F. Goya, T.S. Berquo, F.C. Fonseca, M.P. Morales, Journal of Appl. Phys. 94, 3520 (2003)

RESULTS

MAGNETIC PROPERTIES



AC SUSCEPTIBILITY

χ_{ac} vs T f (5,50,240 Hz)

- Superparamagnetic behavior
- T_M decreases after the SiO_2 coating

$$\phi(\text{DMSA}) = 0,04$$

$$\phi(\text{D2H}) = 0,08$$

$$\phi = \frac{\Delta T_f}{T_f \Delta \log_{10}(f)}$$

LESS INTERACTIONS



D2H collective magnetic behavior is superparamagnetic due to the individual coating of the particles by silica

Opposite behavior than TREG sample!!!

CONCLUSIONS

- SiO₂-coated maghemite nanoparticles with spherical and rod-like nanostructures are synthesized using a reproducible method
- From TREG-coated maghemite nanoparticles, we obtained condensed SiO₂ spheres filled with magnetic nanoaggregates
- From DMSA-coated maghemite nanoparticles, we obtained isolated SiO₂ spheres filled with 1-5 magnetic nanoparticles
- The nature of the magnetic interactions between the Maghemite-coated SiO₂ particles depends on the molecule coating of the magnetic particles:
 - TREG-coated → Increasing interactions
 - DMSA-coated → Decreasing interactions

NANOPOROUS FILMS AND PARTICLES GROUP



Nuria Miguel



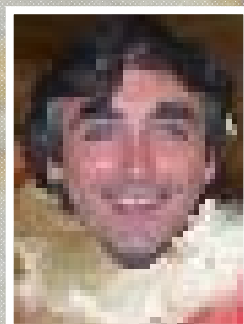
Daniel Carmona



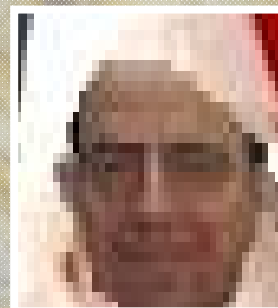
Alejandro G. Roca



Francisco Balas



Jesús Santamaría



Thank you

Gracias

Obrigado