UNIFORM MAGNETITE NANOPARTICLES PREPARED BY HIGH TEMPERATURE DECOMPOSITION IN A ORGANIC SOLVENT

Alejandro Gómez Roca, M. Puerto Morales, Carlos J. Serna
Instituto de Ciencia de los Materiales de Madrid, CSIC, Cantoblanco 28049, Madrid, Spain
E-mail: alexgr@icmm.csic.esT

Magnetic nanoparticles are currently one of the most fashionable and promising materials because of their multiple applications in computing, electronic devices or biomedicine [1-3]. In particular in the biomedicine field, magnetic nanoparticles in the form of colloidal aqueous suspensions, have found applications as magnetic separators, contrast agents for NMR imaging, drug delivering and hyperthermia treatments [4]. The performance of this material will be strongly dependent on the chemical and physical characteristics of the nanoparticles and its surface. The control of particle size and shape and their distribution, the crystallinity, and the colloidal and magnetic properties, such as saturation magnetization and susceptibility are of crucial importance and depend, at a great extend, on the synthesis method chosen to prepare the particles [5]. Finally it should be emphasized that the use of uniform particles for in vivo applications allows the reduction of the doses injected to get a certain signal and avoids secondary effects coming from a wide particle size distribution [5].

The aim of this work is to obtain uniform and crystalline magnetic nanoparticles with high saturation magnetisation values and high susceptibilities and the preparation of stable aqueous suspensions of the particles, suitable for biomedical applications. We have synthesized magnetite nanoparticles (Fe_3O_4) of three different sizes below the limit for single domain magnetic behaviour using a new synthesis route based on the high temperature decomposition of an iron precursor in an organic media and in the presence of a surfactant [6,7] Characterization of the powders has been carried out using X-Ray diffraction and transmission electron microscopy. On the other hand, particle surface modification has been carried out with tetramethylamonium hydroxide in order to obtain a hydrophilic solid, suitable for aqueous dispersion [8]. The colloidal properties of the samples as well as the surface transformation from hydrophobic to hydrophilic have been followed by infrared spectroscopy. Finally magnetic measurements, hysteresis loops at room temperature and 5 K and zero field cooling-field cooling curves (ZFC/FC) have been measured in a vibrating sample magnetometer.

As it is clear from TEM images, magnetite particles synthesized in this work are highly uniform in size and shape (Fig. 1). X-ray diffraction patterns showed that all the samples have a spinel structure similar to magnetite or maghemite. Crystal sizes calculated from the broadening of the (311) reflection are 4 nm for the sample with smallest particles and 15 nm for the sample with the biggest particles, in good agreement with the mean particle size observed by TEM. This result suggests that the particles present a high crystallinity.

Magnetic properties of the samples showed superparamagnetic behavior at room temperature with negligible coercivity and remanence magnetization. Saturation magnetization values of -60 emu/g of sample have been obtained. As the temperature is reduced to 5 K, the coercivity increases as a function of the particle size. ZFC/FC curves are included in Fig. 2. A shift to higher temperature of the blocking peak can be observed as the particle size increases from 25 K to nearly 300 K.

The uniformity and the crystallinity of these nanoparticles, the possibility of synthesizing great amount of product by this technique and our preliminary results on the dispersion of these nanoparticles in aqueous media show that this material is very promising for the preparation of biomedical magnetic liquids.

References:

[1] S.Sun et al, Science, **287** (2000) 1989.
[2] L.L. Beecroft et al , Chemistry Materials **9** (1997) 1302.
[3] Q.A. Pankhurst et al, Journal of Physics D, **36** (2003) 167.
[4] M.P. Morales et al, Journal of Magnetism and Magnetic Materials, **266** (2003) 102.
[5] P. Tartaj et al, Journal of Physics D, **36** (2003) 182.
[6] S. Sun et al, Journal American Chemical Society, **126** (2004) 273.
[7] J. Park et al, Nature Materials, **3** (2004) 891.
[8] V. Salgueiriño-Maceira et al, Langmuir, **20** (2004) 6946.

Figures:

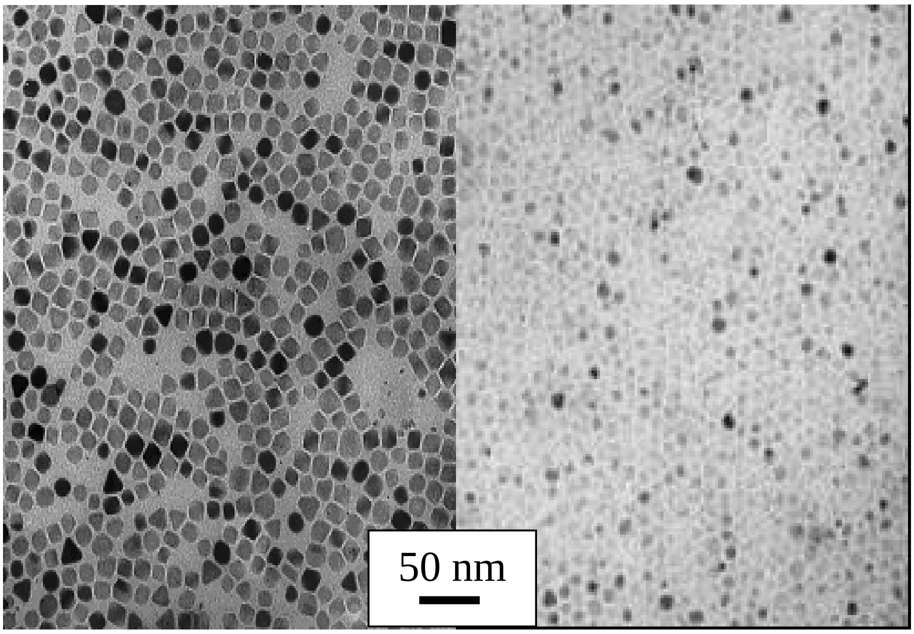

Fig.1 TEM images of magnetic nanoparticles prepared by decomposition at high temperature in the presence of surfactants of different iron precursors: a) Fe- oleate complex b) Fe-Acetyacetonate

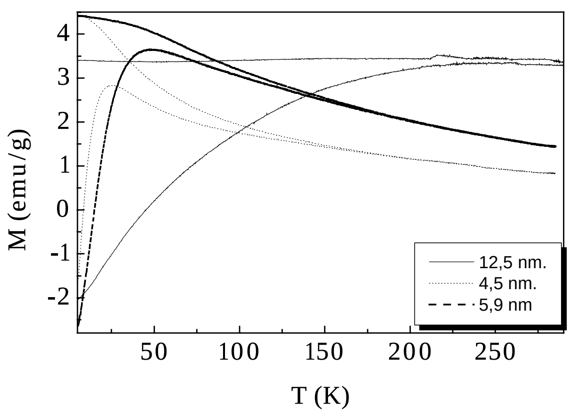

Fig.2. ZFC/FC curves in a magnetic fied of 100 Oe for powdered magnetite samples with different sizes