Magnetic Properties of Fe Oxide Nanoparticles Produced by Laser Pyrolysis for Biomedical Applications

M. A. García[a,b], V. Bouzas[a], R.Costo[c], S. Veintemillas[c], P. Morales[c], M. García-Hernández[c], R. Alexandrescu[d], I. Morjan[d], P. Gasco[e]

[a] *Instituto de Cerámica y Vidrio, CSIC*
[b] *Dept. Fis. Mat. Univ. Complutense Madrid*
[c] *Inst. Ciencia de Materiales de Madrid CSIC, Spain*
[d] *Natl. Inst. Lasers Plasma & Radiat. Physics Romania*
[e] *Nanovector S.r.l., Torino, Italy*

Abstract. We report on the magnetic characterization of Fe oxide nanoparticles by laser pyrolysis and the relationship between the preparation conditions and the magnetic response. It is shown that controlling the preparation conditions during the pyrolisis allows tuning the nanoparticles morphology and structure and consequently the magnetic properties of the nanoparticles. The nanoparticles are loaded into solid lipid nanoparticles without degradation nor significant modification of the magnetic properties.

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INTRODUCTION

Iron oxide nanoparticles are commonly used in biomedical applications as contrast agent for Magnetic Resonance Imaging. The magnetic properties of the nanoparticles strongly depend on their features so nanoparticle production methods with a narrow size distribution are required for these purposes. Among the different methods that can provide this kind of nanoparticles, laser pyrolysis has the advantage to produce homogeneous material in quantities fairly larger than other chemical methods that requires a long time to produce few milligrams and results expensive. A key step for the preparation of nanoparticles is the functionalization to ensure biocompatibility and avoid degradation. However, any manipulation of the nanoparticle may induce modifications on the nanoparticles or promote aggregation that alter the magnetic properties and compromise their use for biomedical purposes. The Iron oxide nanoparticles can be loaded into solid lipid nanoparticles (SLNs) by a warm microemulssion method that improves substantially the colloidal stability without alteration of the particle size distribution, being remarkable that the process SLNs loading disaggregate the colloid with much smaller final hydrodynamic sizes. We

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report here a study of the magnetic properties of Iron Oxide nanoparticles prepared by laser pyrolysis and their relationship with the synthesis parameters. The effect of different procedures including the development of solid lipid nanoparticles on the magnetic properties is also discussed.

EXPERIMENTAL

The Nanoparticles were synthesised by laser pyrolysis as described in [1]. Two set of samples were prepared using different devices and conditions. BONFEX samples we prepared at filiation *c* while SF samples were synthesised using a different set-up at filiation *d*. A previous study demonstrated the reproducibility of the method, showing that the morphological features of the nanoparticles scarcely depend on the experimental set-up but on the synthesis parameters [2]. Table 1 summarises the main parameters of the synthesis for the BONFEX samples. The synthesis conditions for SF samples are detailed in [1]. Main difference between both sets was the laser power and gas mixture used in the synthesis. The nanoparticles were loaded into a SLNP following the procedure described in [3]. Magnetic properties were measured with a Magnetic characterization has been carried out with a Superconducting Quantum Interference Device (SQUID) magnetometer. A structural characterization of the samples is also reported on [1,2].

Table 1: Experimental parameters employed in the laser pyrolysis synthesis of magnetic iron oxide nanoparticles. Ethylene and sulphur hexafluoride are employed are sensitizers. The pressure is 300 mbar (400 mbar in Bonfex4) and the nozzle diameter was 2 mm.

SAMPLE	GAS FLUX			EVAPORATION T(C°) $Fe(CO)_5$
	CARRIER	COAXIAL	WINDOWS	
BONFEX02	$9C_2H_4$+9Air	22 He	726 N_2	26
BONFEX03				10
BONFEX04	9 C_2H_4		726 N_2+40Air	10
BONFEX05				22
BONFEX06	18 C_2H_4	190 He	1517He+40Air	22
BONFEX08	$12C_2H_4$+6Air		1517He	10

RESULTS AND DISCUSSION

Figure1 show the magnetic measurements (magnetization curves and thermal dependence of the magnetization) . Increasing the concentration of C_2H_4 in gas carrier (BONFEX6 and BONFEX8) led to smaller saturation magnetization (M_S) and larger coercive fields (H_C) at low T. The samples exhibited superparamagnetic behaviour at room temperature with Ms vales below 10 emu/g. The blocking temperatures are in the range of 40-80 K. SF samples exhibit much larger M_S values of the order of 40 emu/g.

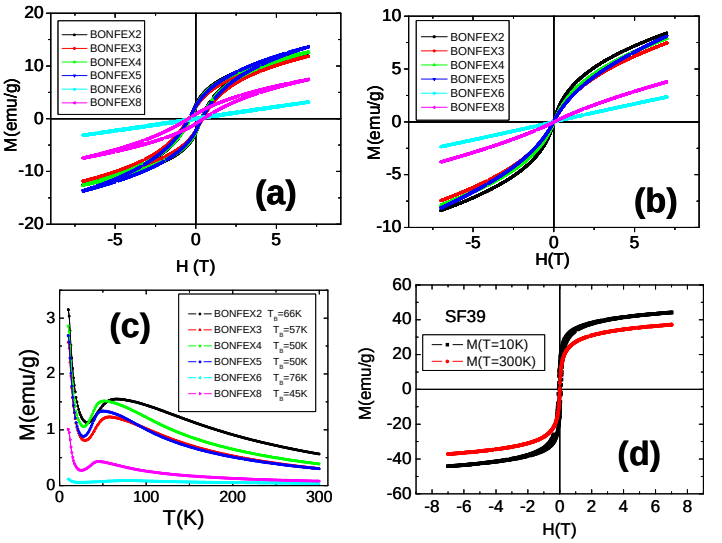

Figure 1. Magnetization curves of BONFEX samples at (a) 10 K and (b) 300 K. (c) Thermal dependence of the magnetization with an applied field of 1000 Oe after Zero-Field Cooling ($H_{cooling}$=5T). (d) Magnetization curves of SF39 sample at 10 and 300 K.

Differences in the magnetic properties can be explained in terms of crystallinity and particle size that are determined by the synthesis parameters. For the BONFEX samples the magnetization increase with particle size. This is a general behaviour of iron oxide nanoparticles. Magnetic interactions are quite sensitive to anystructural change and the oxygen vacancies and reduced coordination of Iron ctions at the particle surface lead to a shell of reduced magnetization [4]. As the particle size is reduced, this shell represents a larger fraction of the total volume yielding lower M_S values. These surface effects arise a core-shell ordered-disordered structure. The disordered shell exhibits reduced magnetization and increases the coercive force due to the formation of a spin glass phase. For particles with similar size, we found larger MS values for samples with higher crystallinity in these samples evidenced by XRD and TEM studies. Differences can be a so be explained as due to the reduced magnetization of amorphized regions.

The comparison between the magnetic properties of the as prepared NPs and the SLNs with the NPs embedded is presented in figure 2. As expected the magnetization values of the SLNs is resulted smaller than the pure iron powder. However, when the curves are normalized and we remove the diamagnetic contribution of the lipid matrix, magnetization curves matches quite well discarding a severe degradation or modification of the iron oxide nanoparticle during the load process. However, a detail of the low field region demonstrated the existence of some coercivity at 300 K in the SLNPs not present in the initial samples. This coercivity is of the order of 100 Oe and led to remanence values at about 12% of the saturation magnetization. The origin of this coercivity can be the agglomeration of iron oxide nanoparticles in the SLNPs that become magnetically coupled and therefore increase the magnetic coherent volume leading to a fraction of NPs to exhibit a blocking temperature over 300 K

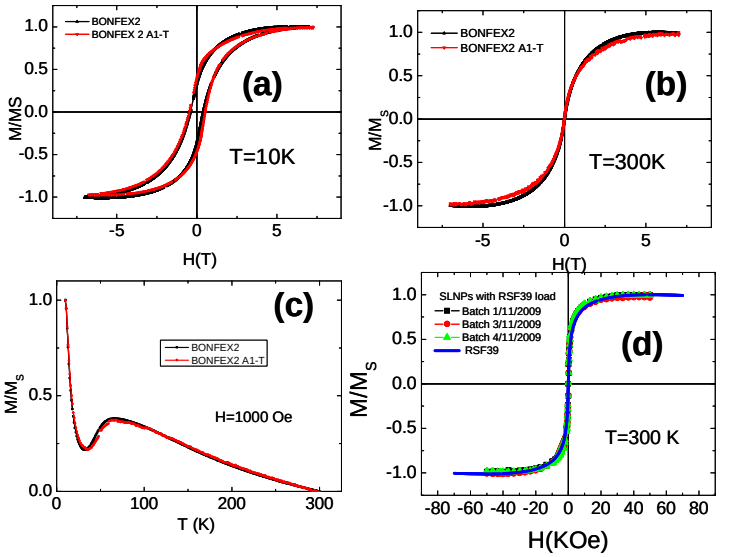

Figure 2 (a,b) Normalized magnetization curves for the initial BOFEX2 sample and the SLNs loaded wit these NPs at 300 K and 10 K. (c) Thermal dependence of the magnetization with an applied field of 1000 Oe after Zero-Field Cooling (Hcooling=5T). (d) Comparison for the SF39 sample.

The normalization factor to make curves of the NPs and the SLNs match provides the value of the real NP load in the complex. The measured load was o f the order of 5% in all the samples and matches perfectly the nominal value confirming the effectiveness of the method to load NPs preserving their magnetic properties.

CONCLUSIONS

Superparamagnetic Fe oxide nanoparticles were prepared by laser pyrolysis. The synthesis parameters allow tuning the magnetic properties of the nanoparticles (Magnetization values, blocking temperature and coercive force) by controlling the morphological and the structural features of the particles. The nanoparticles can be loaded into SLNs with good control of the load volume without degradation nor significant modifications of the nanoparticles. Just the appearance of some coercivity at room temperature upon loading is observed

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