# Magnetic properties of iron oxide nano-particles fabricated by the inverse micellar method.

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## Abstract

***We have fabricated various types of iron oxide nanoparticles by the inverse micellar method [1,2] and systematically studied their magnetic properties by Mössbauer spectroscopy. The inverse micellar method allows control of the oxidation state and fabrication of monodisperse nanoparticles. We present an analysis of the manufactured iron oxide particles. This investigation reveals details about particle shape, size distribution, and the variation of the Mössbauer hyperfine field as function of temperature, furthermore we are presenting results on the change of magnetic properties as function of the particle size. Particularly, for the smaller particle sizes the magnetic properties show nano-scale related effects, e.g. superparamagnetism.***

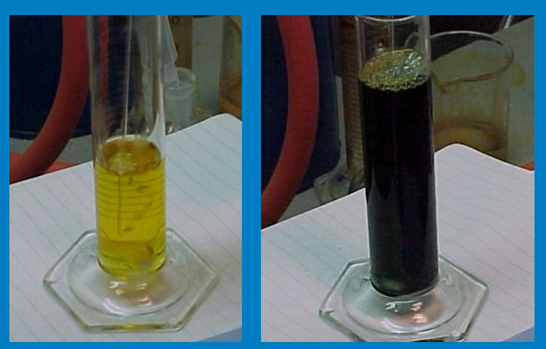

**Figure 1. Left:** Inverse micellar solution when starting addition of base.
**Right:** Inverse micellar solution when ending addition of base.

## Particle manufacture

**The particles were manufactured using the inverse micellar method [1,2].**
**The result is particles, which are homogeneous in size, shape, morphology and composition.**
**In this technique the particles are formed inside a nano-sized water droplet inside an inverse micelle (polar end facing inwards). The size of the micelles determines the size of the manufactured particles.**

## Analysis

We are presenting measurements on the samples JH032S (small particles), JH032L (large particles), JH041, JH046 which are results of similar experiments. These samples are manufactured using the same recipe.

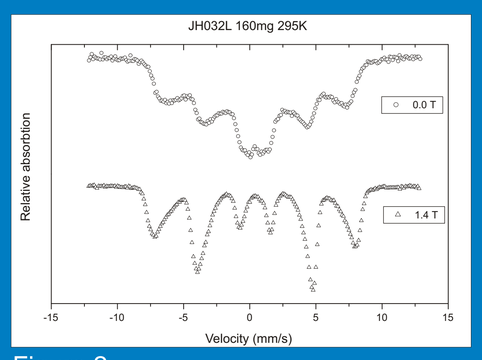


Figure 2.

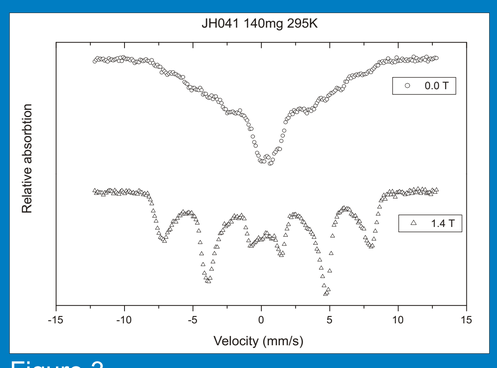


Figure 3.

Some samples show considerable particle interaction. Interaction is displayed as a very broad distribution of magnetic hyperfine fields, extending to very low values. Broadening is caused by dynamic effects. An external magnetic field can restore the magnetic splitting. This is shown in figure 2 & 3.

Mössbauer spectroscopy reveals a Quadrupole shift close to 0. This indicates that the crystal structure is cubic. Transmission Electron Microscopy (TEM) images in figure 9 support this interpretation.

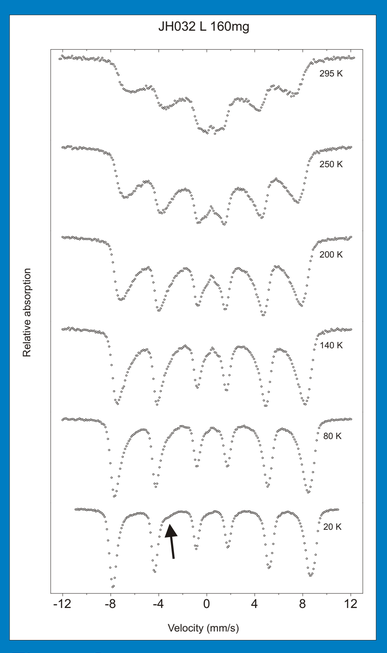


Figure 6.

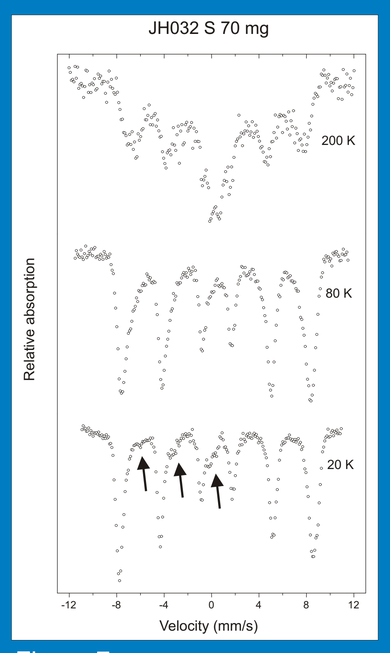


Figure 7.

Mössbauer spectra are all sextets and they change by applied magnetic fields (3:2:1:1:2:3) -> (3:4:1:1:4:3). This shows that the nanoparticles are ferrimagnetic.

The magnetic hyperfine field is approx. 50 T for most of the samples.

The hyperfine field as a function of temperature is shown in figure 4.

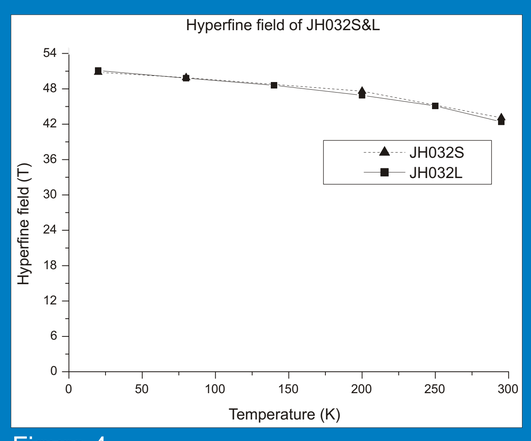


Figure 4.

## Mössbauer spectroscopy

The particles have been studied using Mössbauer spectroscopy. By Mössbauer spectroscopy it is possible to determine with high accuracy the chemical composition of the particles. An example of a fitted spectrum is shown in figure 5.

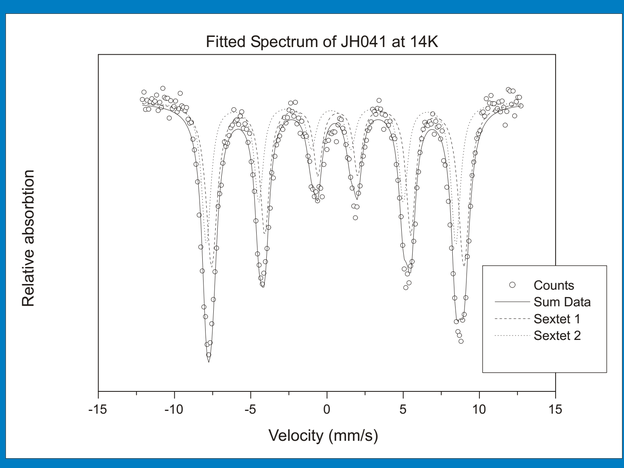


Figure 5.

The hyperfine field and the quadrupole shift indicate either magnetite or maghemite. The spectra in figure 6 and 7 show several characteristics of both maghemite and magnetite:

At high temperatures line 6 is sharper than line 1.
At low temperatures line 1 is sharper than line 6.

In stoichiometric magnetite the iron atoms are positioned at sites A and B. This is seen as two distinct sextets. Only trace of this signature is present in our spectra (see arrows) and therefore we have non-stoichiometric magnetite $Fe_{3-x}O_4$ very close to maghemite (x~1/3). Comparison with Ramdani et al. [3] supports this interpretation.

## Transmission Electron Microscopy

The TEM images of JH046 show cubic or trapezoidal particles. The particles are very homogeneous in shape. We interpret the images as showing cubically shaped particles seen from different angles. The edge length distribution presented in figure 8 is produced assuming this shape. Measuring the size of 158 particles, we found a mean edge length of 28.5nm and a standard deviation of 10.3nm.

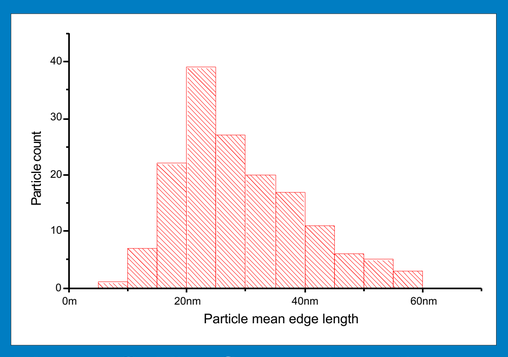


**Figure 8.** The figure show the distribution of the of the mean edge length of the particles.

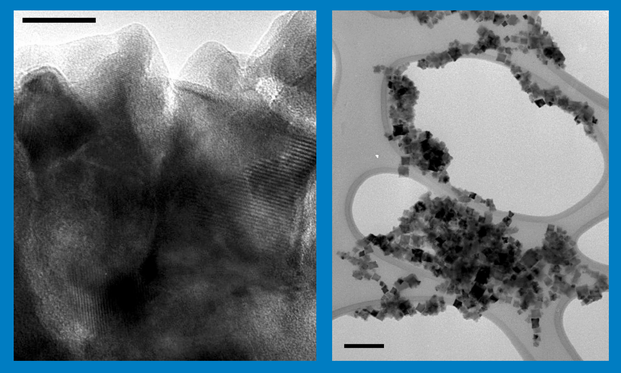

**Figure 9. Left:** TEM image of micellar particles at 390k magnification. Note the interference patterns caused by overlapping crystal planes. These can only occur if the structure is regular. The bar is 10nm.
**Right:** TEM image of micellar particles at 27.5k magnification. Note the homogeneous size distribution and particle shapes. The bar is 100nm.

**References:**
**[1] C. T. Seip et al. IEEE Transactions on Magnetics 34, (1998), 1111**
**[2] E. E. Carpenter. Synthesis of Magnetic Nanoparticles Using Reverse Micelles. Ph.D. Thesis 1999. University of New Orleans.**
**[3] A. Ramdani et al. Pertubation De L'Echange Electronique Rapide Par Les Lacunes Cationiques Dans Fe3-xO4 (x<=0.09). J. Phys. Chem. Solids Vol. 48, No 3, pp 217-228, 1987.**
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