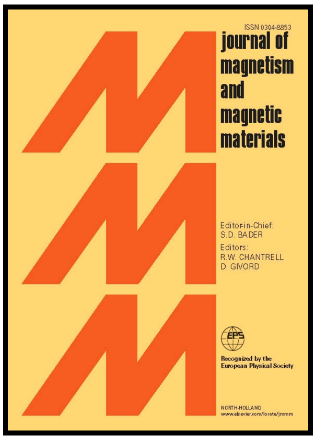

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Size analysis of single-core magnetic nanoparticles

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Abstract

Single-core iron-oxide nanoparticles with nominal core diameters of 14 nm and 19 nm were analyzed with a variety of non-magnetic and magnetic analysis techniques, including transmission electron microscopy (TEM), dynamic light scattering (DLS), static magnetization vs. magnetic field (*M-H*) measurements, ac susceptibility (ACS) and magnetorelaxometry (MRX). From the experimental data, distributions of core and hydrodynamic sizes are derived. Except for TEM where a number-weighted distribution is directly obtained, models have to be applied in order to determine size distributions from the measurand. It was found that the mean core diameters determined from TEM, *M-H*, ACS and MRX measurements agree well although they are based on different models (Langevin function, Brownian and Néel relaxation times). Especially for the sample with large cores, particle interaction effects come into play, causing agglomerates which were detected in DLS, ACS and MRX measurements. We observed that the number and size of agglomerates can be minimized by sufficiently strong diluting the suspension.

Keywords: single-core nanoparticles, size distribution, transmission electron microscopy, dynamic light scattering, magnetization, Brownian and Néel relaxation

1. Introduction

There is a wide field of applications of iron oxide magnetic nanoparticles (MNPs) with sizes from a few nm up to several micrometers in biomedical diagnosis, therapy and imaging [1,2]. Central objective of the current research is the standardization of MNP characterization methods [3]. MNPs can be classified into single- and multi-cores [4]. This contribution deals with the analysis of single-core nanoparticles. Single-core MNPs in biomedical applications consist of a single magnetic core, typically magnetite, maghemite or a mixture of both, surrounded by mostly an organic shell. In contrast, multi-core nanoparticles consist of several nanocrystals (magnetic cores) either densely or loosely packed within the multi-core structure and embedded in a matrix [3,4].

There is a variety of analysis methods which can be applied to estimate the core's magnetic moment, its size, anisotropy energy and the hydrodynamic size of the whole particle. Among them are transmission (TEM) and scanning electron microscopy (SEM), dynamic light scattering (DLS), asymmetrical flow field-flow fractionation (A4F) in combination with light scattering, X-ray and neutron scattering techniques as well as magnetic techniques such as static magnetization vs. magnetic field (*M-H*), ac susceptibility (ACS), magnetorelaxometry (MRX) and magnetic particle spectroscopy (MPS) measurements. Other analysis techniques such as Mössbauer spectroscopy provide information on the elementary composition. Most of the techniques require models to derive one or more of the listed nanoparticle parameters. In addition, since most methods measure the response of an ensemble of MNPs, the quantification of magnetic interactions between particles is an important issue.

In this paper, we summarize the analysis results of single-core iron oxide nanoparticles with nominal core diameters of 14 nm and 19 nm coated with dimercaptosuccinic acid (DMSA) applying static *M-H*, ACS vs. frequency, MRX, TEM and DLS measurements. As a consequence of the variety of analysis methods, there is some redundancy in parameters which helps one to verify and refine models.

2. Experimental

2.1 Materials and methods

Both samples, CSIC-11 and CSIC-12, are composed of maghemite/magnetite nanoparticles synthesized by thermal decomposition of iron oleate. The samples were transferred to aqueous media via ligand exchange with DMSA. Details can be found in [5]. An increase in size was obtained by reducing the oleic acid content in the reaction media.

Measurements were mostly carried out in suspensions. In some cases, samples with MNP immobilized by freeze-drying were studied. Iron contents of samples were determined by inductively coupled plasma-optical emission spectrometry (ICP-OES, PerkinElmer Optima 2100 DV ICP) after dissolving the samples in HNO_3:HCl 1:3 mixtures and diluting them with doubly distilled water.

Static magnetization measurements were performed with a Magnetic Property Measurement System (MPMS-XL, Quantum Design).

ACS vs. frequency measurements were carried out with different setups: The commercially available DynoMag system from Acreo which operates in a frequency range from 1 Hz to 500 kHz as well as custom-built systems at Acreo (up to 10 MHz) and