Experimental report

08/04/2024

Proposal: **5-32-943**

Council: 10/2022

Title: Dependence of the thickness of non-magnetic surface layer on the sizeof superparamagnetic iron oxide nanoparticles

Research area: Physics

This proposal is a new proposal

Main proposer: **Artem FEOKTYSTOV**

Experimental team: Artem FEOKTYSTOV
Steffen TOBER

Local contacts: Nina-Juliane STEINKE

Samples: Fe3O4/C18H34O2/C7H8/C7D8

Instrument	Requested days	Allocated days	From	To
D33	2	2	17/05/2023	19/05/2023

Abstract:

Iron oxide nanoparticles are of particular interest for medical applications as they are biocompatible and can be used for diagnostics, imaging, drug delivery and cancer treatment. For these and other applications understanding and controlling the magnetic properties is crucial.

The debate about the magnetization distribution within these nanoparticles is ongoing. There is still no satisfying model that is able to explain the observed reduction of the saturation magnetization of the particles compared to the bulk material. Possible mechanisms that were suggested include a magnetic core shell structure with a magnetically depleted surface layer in addition to spin canting around defects within the particles, a homogeneous distribution of spin canting in the particle or the formation of antiphase domain boundaries due to lattice defects.

Dependence of the thickness of non-magnetic surface layer on the size of superparamagnetic iron oxide nanoparticles

Spherical magnetic nanoparticles are known to possess a reduced saturation magnetization as compared to bulk. One of the explanations among the others is the existence of a non-magnetic layer due to spin misalignment on the nanoparticle surface.

The aim of the experiment was to determine if there is a correlation between the thickness of the non-magetic surface layer and the nanoparticle radius.

A range of toluene-based iron oxide nanoparticle with diameters of 5, 10, 12, 15 and 20 nm obtained commercially and in-house was studied by means of small-angle scattering of polarized neutrons. The particles were stabilized against agglomeration with oleic acid. In parallel, the particles were prepared in a 80%-solution of deuterated toluene to reduce nuclear contrast for nanoparticle iron oxide cores and thus enhance magnetic signal. Depending on the nanoparticle size the data was collected at two or three detector distances of 2, 5.3 and 10.3 m with corresponding collimation lengths of 5.3 (both for 2 and 5.3 m) and 10.3 m. With the wavelength of 5 A the accessible q-range constituted 0.05 – 1.9 nm^{-1}. During the measurements samples of the magnetic nanoparticles were held in a horizontal magnetic field applied orthogonally to the neutron beam. In order to keep the magnetization at the same degree of saturation for all samples, different magnetic fields for different particle sizes were applied: 0.4 T (20 nm), 0.95 T (15 nm), 1.3 T (12 nm) and 1.4 T (10 nm). For 5 nm nanoparticles the maximum available field of 3.0 T was utilized also because of an initial concern about the data analysis due to bi-modal size distribution in the sample.

Data was corrected on background scattering from the empty cell and 80%-d-toluene buffer and put in absolute scale with the direct beam measurement. GRASP software was used for data reduction. An additional offset of 0.12 m was necessary to merge scattering curves from different detector distances. This offset was a result of some maintenance works prior the cycle and was provided by the local contact later for the data analysis.

In the figure the pure nuclear scattering (left) and a nuclear-magnetic interference (right) is plotted as obtained from a 2D fit of detector images for each sample.

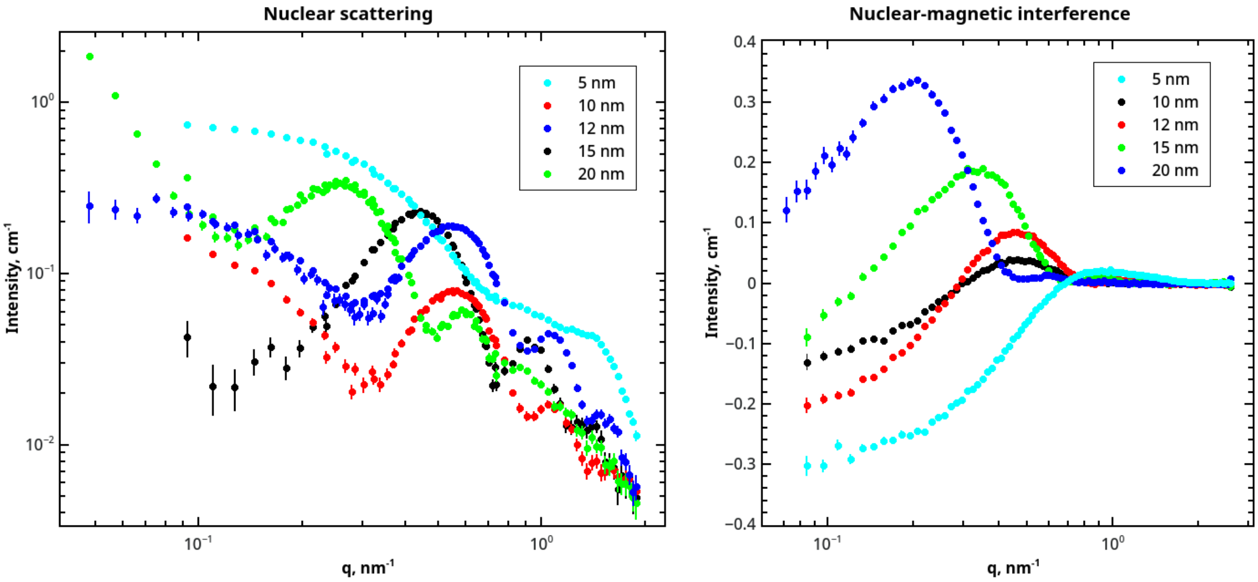

Figure. Scattering curves obtained from a 2D fit of detector images: nuclear scattering (left), nuclear-magnetic interference (right).

The above mentioned data will be simultaneously refined to obtain the thickness of the non-magnetic surface layer for each sample. For that the nuclear scattering will be modeled with a spherical core-shell, where size distribution parameters of the particle core will be fixed to the values obtained from small-angle X-ray scattering (SAXS) and only particle concentration and surfactant shell thickness will be varied. In the case of 20 nm nanoparticles, a contribution from larger objects has to be included into the fit of nuclear scattering data, which is probably a results of chain building under the action of the magnetic field. However, the objects are very large and their contribution lays in very small angles, which won't affect the fit results for the single nanoparticles. For 5 nm nanoparticles a bi-modal size distribution will be taken into account.

The magnetic scattering for all samples will be modeled by a sphere with a reduced size as compared to the SAXS results.