Experimental report

19/09/2022

Proposal:	**5-53-301**		**Council:**	10/2020

Title: Linking magnetic and structural properties of lithiated iron oxide nanocubes

Research area: Chemistry

This proposal is a new proposal

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Samples: iron oxide nanoparticles
lithiated iron oxide nanoparticles

Instrument	Requested days	Allocated days	From	To
D33	4	4	10/06/2021	14/06/2021

Abstract:

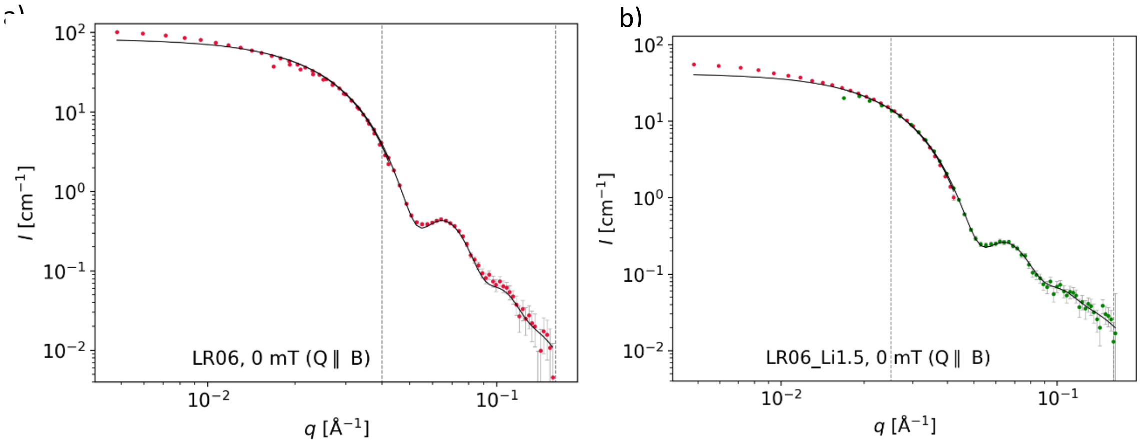
In response to concerns about the environmental impact and use of non-abundant/renewable components in lithium ion batteries, iron oxides are emerging as a viable alternative to current electrode materials. However, a clear analysis of the lithiation process on the nanoscale, including intermediate structures and the influence of lithium on the magnetic properties, is still missing.
The objective of this proposal is to investigate the chemical and magnetic morphology in iron oxide nanoparticles with varying degree of chemical lithiation. Polarized SANS will be applied to reveal the progress of lithiation and the occurring intermediate phases from the nanoparticle surface into the particle core and to correlate lithiation state and magnetization distribution. This will ultimately allow to use relatively simple magnetometric methods to determine the level of lithiation of the particles even under operando conditions.

Experimental Report: Linking magnetic and structural properties of lithiated iron oxide nanocubes

The goal of this experiment was to investigate the structural and magnetic morphology of iron oxide nanoparticles after chemical lithiation using polarized SANS.

We measured oleic acid stabilized dispersions of iron oxide nanoparticles in *n*-hexane which were subjected to chemical lithiation previous to the beamtime. Measurements were performed at zero field and under applied fields of up to 820 mT with a field direction perpendicular to the neutron beam. Using two detector distances and corresponding collimations of 8 m as well as 2.8 m at the D33 instrument gave access to a wide scattering range between $5 \cdot 10^{-3}$ and $1.7 \cdot 10^{-1}$ Å^{-1}.

The obtained nuclear SANS data of pristine, non-lithiated nanocuboctahedra (LR06) revealed two form factor minima and was evaluated using a sphere model (Fig. 1a). In contrast to this, the SANS data of particles with the highest degree of lithiation (LR06_Li1.5) were accurately approximated with a spherical core-shell model of magnetite core and lithiated iron oxide shell, considering in addition spherical oleic acid micelles (Fig. 1b).



Fg. 1: SANS measurements of a) pristine iron oxide nanoparticles and b) chemically lithiated nanoparticles..

The magnetic scattering data shows a clear splitting of *I*+ and *I*- (Fig. 2) which appears promising to determine the magnetic scattering length density profile at different applied fields. A smaller magnetic than nuclear radius is suggested for the pristine nanoparticles (Fig. 2).

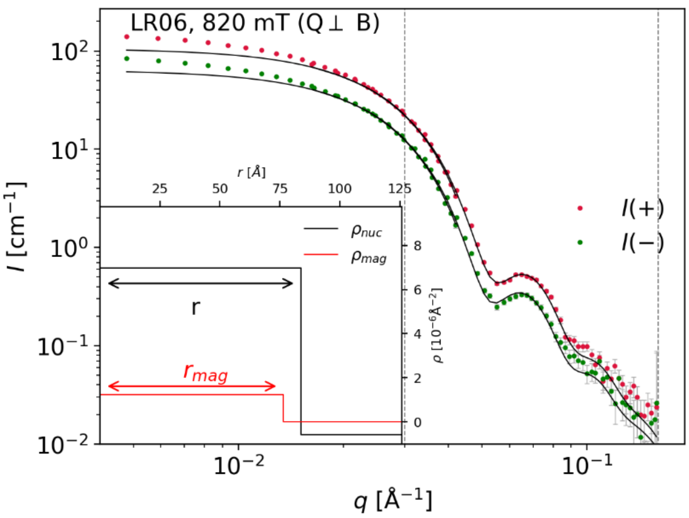


Fig. 2: SANSPOL of the pristine nanoparticles at 820 mT evaluated using a sphere model with the nuclear (black) and magnetic (red) SLD profile (inset).

Further analysis of the magnetic SANS data of lithiated particles is ongoing.