

# Experimental report

26/09/2023

**Proposal:** 5-32-940

**Council:** 10/2022

**Title:** Effect of the field induced rearrangement of superparamagnetic iron oxide nanoparticles on their medicalmagnetic hyperthermia applicability

**Research area:** Physics

**This proposal is a new proposal**

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**Samples:** SPIONs with a Fe<sub>3</sub>O<sub>4</sub> core and an oleic acid/oleylamine shell, embedded within the lipid matrix of cubosomes

Instrument	Requested days	Allocated days	From	To
D33	4	3	14/04/2023	17/04/2023

## Abstract:

We propose a SANS study on the rearrangement of superparamagnetic iron oxide nanoparticles (SPIONs) embedded within the lipid matrix of cubosomes in-situ during magnetic heating. Using a high-frequency AC field setup, we will characterize resulting structures as well as the dependence of agglomeration on magnetic field frequency and amplitude. Given the strong effect of structure formation on magnetic hyperthermia performance, such knowledge will be highly relevant for real life application of magnetic nanoparticles.

## Effect of the field induced rearrangement of superparamagnetic iron oxide nanoparticles on their medical magnetic hyperthermia applicability

As initially proposed, we conducted an experiment to study superparamagnetic iron oxide nanoparticles (SPIONs) using small-angle neutron scattering (SANS) at the D33 beamline. The studied nanoparticles, SPIONs

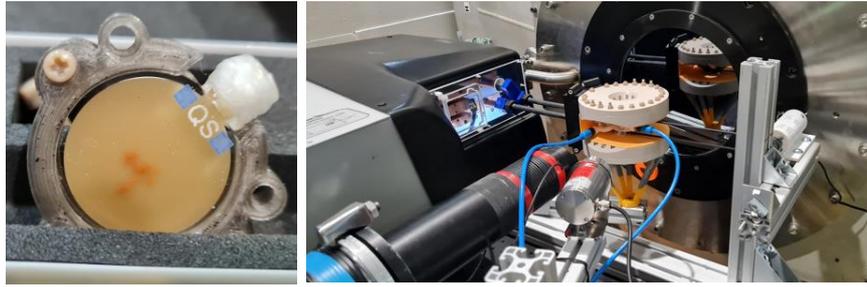


Fig. 1. Left: One of the precipitated samples after loading into the SANS cuvette. Right: Experimental setup, as used at D33@ILL. The coil set is centred at the beam; the driver can be seen on the left side of the nozzle.

with a  $\text{Fe}_3\text{O}_4$  core and an oleic acid/oleylamine shell, were described in [1,2]. The experiment aimed to gain insights into the potential structure formation of SPIONs in situ during magnetic heating using alternating current (AC) fields. The ultimate goal was to understand this process for potential application in magnetic hyperthermia. Unfortunately, after transporting the samples to ILL, the nanoparticles showed a high degree of precipitation (Fig. 1). We have exhausted multiple possibilities of recovering the samples from ultrasound baths through dilution as well as evaporation and preparing a new dispersion; however, with no success. Measurements of the precipitated samples did not give a sufficient signal to noise ratio to allow any further analysis.

To carry out the experiment, we utilised a customised setup from NBnanoscale, incorporating an open coil system. This setup allowed us to apply AC fields with varying amplitudes (2-17.2 mT) and frequencies (137-360 kHz) perpendicular to the neutron beam. The system underwent multiple upgrades to prepare it for neutron experiments. A more robust coil was designed with a larger opening angle. A rotating sample holder was employed, along with air cooling for the sample and in-situ sample temperature monitoring. The system proved to work exceptionally reliably, causing no significant downtime during the beamtime. Due to the precipitation of the original sample, we have used a previously characterised test sample for benchmarking the system performance and suitability for neutron beamlines with great success. The test samples were  $\text{Fe}_3\text{O}_4$  nanocubes dispersed in a water solution with average particle sizes of 12 nm.

[1] Caselli, L.; Mendoza, M.; Muzzi, B.; Toti, A.; Montis, C.; Mello, T.; Di Cesare Mannelli, L.; Ghelardini, C.; Sangregorio, C.; Berti, D., *Int. J. Mol. Sci.* 2021, 22, 9268.

[2] Mendoza, M.; Montis, C.; Caselli, L.; Wolf, M.; Baglioni, P.; Berti, D., *Nanoscale* 2018, 10(7), 3480–3488.

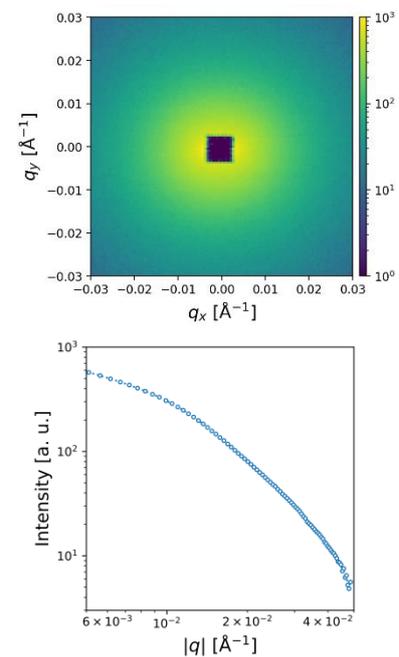


Fig. 2. Typical results of the measurement of the test samples for the AC field setup. 2D reduced data is shown at the top, with corresponding  $I(q)$  curve at the bottom.