Experimental report

15/09/2023

Proposal: **9-10-1741** **Council:** 10/2022

Title: Effect of electric fields on structure of ferrofluids at the solid interface

Research area: Physics

This proposal is a new proposal

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Samples: ferrofluids

Instrument	Requested days	Allocated days	From	To
SUPERADAM	6	4	26/06/2023	30/06/2023

Abstract:

The reproducible development of functional nanomaterials with pre-defined properties is an essential need in different areas. In this context, magnetic-responsive materials play an important role as they can be tuned by magnetic fields. Electric fields, similar to magnetic fields, could be also a driving force inducing the assembling of magnetic nanoparticles in the bulk and at the interface. Recently it was shown that MNPs in ferrofluids can aggregate under external electric fields and according to the proposed theoretical model, it was shown that the process depends on the particles size, dielectric constant of the solvent and MNPs, temperature, and concentration among others. By using neutron reflectometry we propose to investigate NPs assembly on planar interfaces depending on particles diameter and also dielectric constant of the solvent, when an external electric field is applied to the interface. The evolution of the interface structure under external field will be traced by analyzing changes in the scattering length density depth profiles derived from neutron reflectivity curves.

Experimental report

Proposal #9-10-1741: Electric fields effect on structure of ferrofluids by neutron reflectometry

Proposal #CRG-2957: Electric field driven changes of the structure in ferrofluids close to solid substrates

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Scientific background

Stimuli responsive materials which can self-assemble depending on the environment are of great interest. In this context nanoparticles (NPs) offer unique opportunities as they can be custom designed and it is very important to understand the assembly mechanisms to control the resulting structures, which is crucial for the synthesis of materials with controllable properties. In the case of ferrofluids (FFs), suspensions of colloidal magnetic nanoparticles (MNPs), the response of the MNPs on an external magnetic field can result in various structures and MNPs aggregates. MNPs assembly on planar interfaces from liquid dispersions has attracted particular attention since the first layer can be used as a template for further deposition. Magnetic field enhances the structure of MNPs at the interface with solid. Very recently it was shown that electric fields, similar to magnetic fields, could be the driving force inducing the assembling of MNPs at the interface. Understanding the collective interactions of dissolved magnetic NPs, their self-assembly on surfaces and the influence of external fields are of interest from a fundamental perspective and for applications as well.

Several years ago it was shown by SANS that MNPs in transformer based ferrofluids can aggregate under external electric fields. The same type of particles in transformer oil was used in the very recent neutron reflectometry (NR) investigations of impact of external electric and magnetic fields on FFs behaviour. So, the aim of the current experiment was continuation of previous work and to have full explanation about effect of electric field on particles self-assembly.

Experimental results

The aim of this proposal was to investigate the interface structural changes in FFs as well as the magnetic fluid-solid interfaces under electric fields. All planned samples and conditions (electric fields strength etc) were measured during the received beamtime at SuperADAM.

The NR experiment was based on a standard configuration used to study the solid-liquid interfaces when a plane neutron beam passes through a single-crystal silicon substrate (thickness about 10 mm) to meet and scatter from the interface between a liquid sample and an electrode film (thickness below 1000 Å) deposited on the substrate. The electrode was represented by a double metal layer on the monocrystal silicon substrate (the silicon monocrystal is used for guiding the neutrons to the interface). Using bimetal-layered electrode with Ti/Ni (50/1000 Å) the most suitable and possible (taking into account the instrument features) neutron contrast conditions for reflection could be reached. To apply the voltage on the electrodes and control the intensity we used a high voltage generator and specially modified solid/liquid cell with electrodes connections.

The investigated nanofluids were prepared by dispersing magnetic nanoparticles with different sizes into organic solvents (xylene) and transformer oils. Also fullerenes C_{60} NP were added to ferrofluids samples with TO. The samples in the experimental cell were exposed to DC of various intensities (up to 700 kV/m) at room temperature.

In Fig. 1, one can see that the electric voltage acting on the TO-based ferrofluid (0.1% wt) causes changes in scattering intensity at whole q range. Slight but resolvable evolution of the curves with increasing electric field is indicative of the reorganization of the ferrofluid at the interface, which shows that the particle adsorption is field sensitive. In the static electric field the particles are polarized and due to the high dielectric contrast between the transformer oil and magnetite, the induced dipoles are strong enough to activate the attractive interaction. After switching field off there is almost no effect of relaxation (there are no any changes at experimental reflectivity curves (Fig. 2)). Effect of electric field become more pronounced when concentration of NPs is 10 times higher (Fig. 3) or with adding fullerenes C_{60} NP (Fig. 4). For xylene-based MF we can observed no difference at experimental NR curves (no any visible effect) (Fig. 5). It should be mentioned that there were not any changes of the reflectivity curves for pure solvent (without NPs) when maximum field with 700 kV/m was applied.

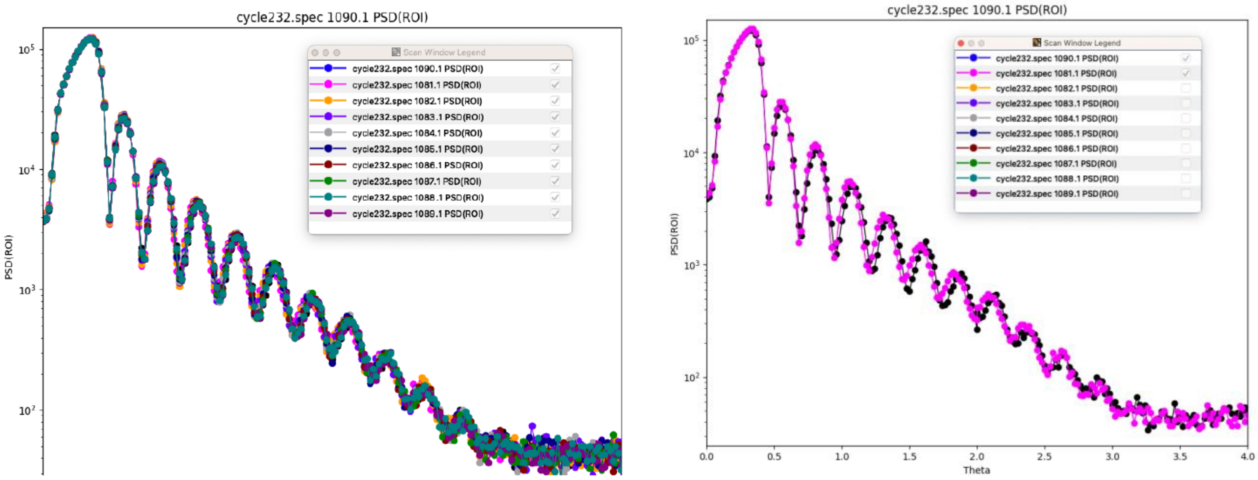

Fig.1. Specular reflectivity curves obtained in the NR experiment with TOFF sample with 0.1% wt of NPs when applying an electric field from 0 to 700 kV/m (on the left). There are some small changes in the oscilations intensity and shift of the peaks positions. Experimental NR data for initial interface and then with maximum field, 700 kV/m, applied (on the right). Change of reflectometry curves depending on electric fields strength.

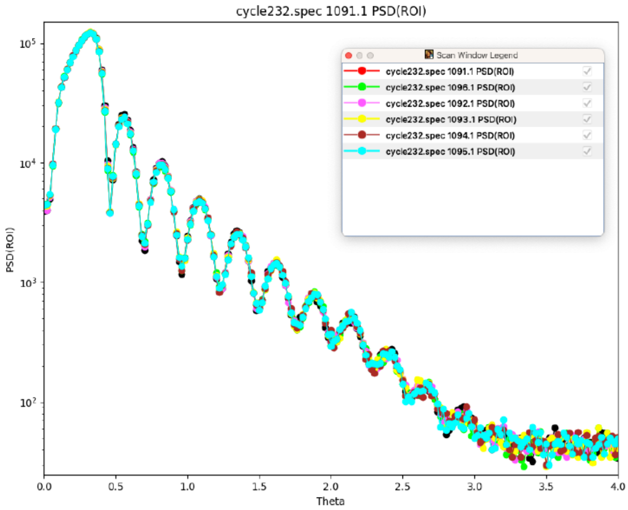

Fig.2. Specular reflectivity curves obtained in the NR experiment with TOFF sample with 0.1% wt of NPs at different times after the external electrical field of 700 kV/m was switched off. There are almost no any changes in the NR curves.

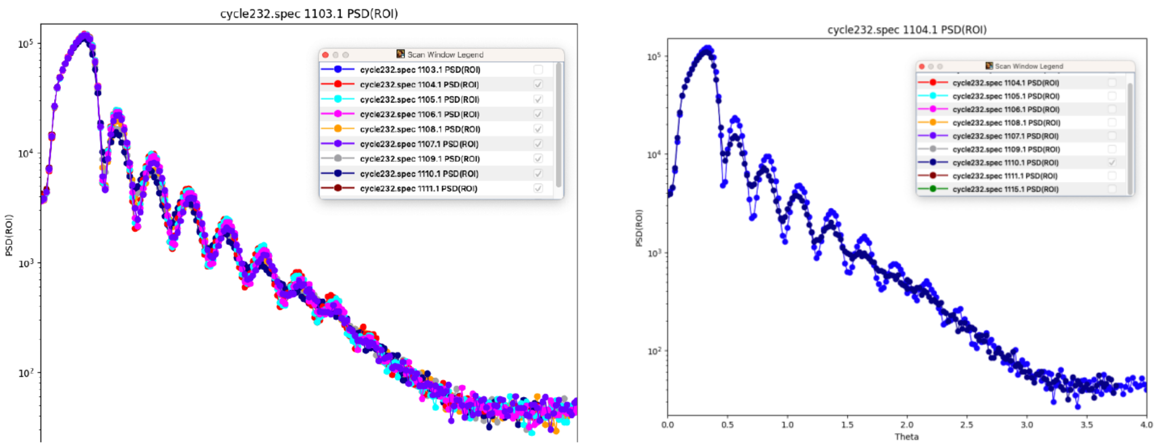

Fig.3. Specular reflectivity curves obtained in the NR experiment with TOFF sample with 1% wt of NPs when applying an electric field from 0 to 700 kV/m (on the left). There are visible smearing of the oscillations as well as shifts of the peaks positions. Experimental NR data for initial interface and then with maximum field, 700 kV/m, applied (on the right). Huge changes of reflectivity curves with electric field, namely smearing of peaks in high q range is well observed.

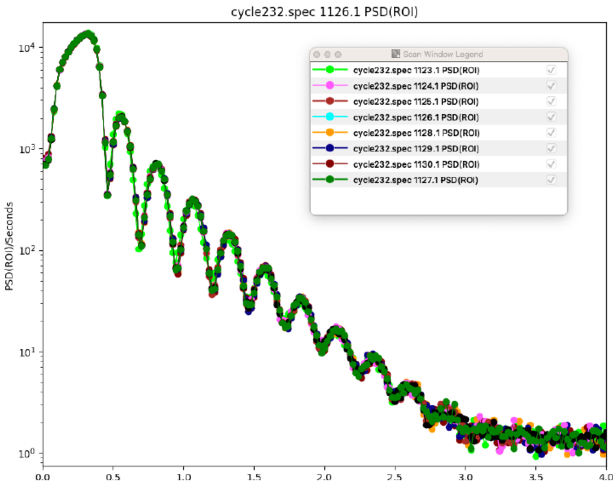

Fig.4. Specular reflectivity curves obtained in the NR experiment with TOFF sample with 0.1% wt of magnetite and fullerenes C_{60} NPs when applying an electric field from 0 to 700 kV/m. There are no any detectable changes in NR curves at different electric fields applied.

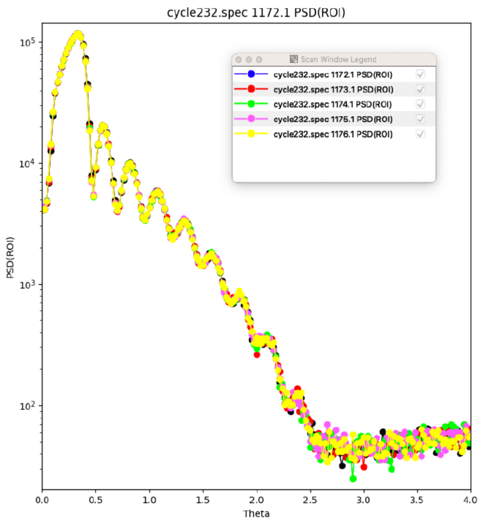

Fig.5. Specular reflectivity curves obtained in the NR kinetic experiment with xylene-based MF without field (1.5 hours per curve). There are no big difference between NR curves.

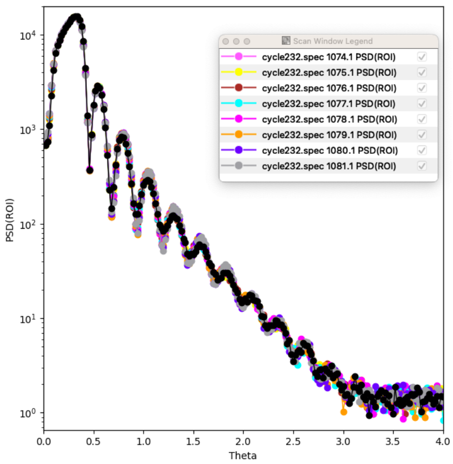

Fig.6. Specular NR curves for time-dependent adsorption. During approx. first 4.5 hours we can see changing of reflectivity curves and than there are no changes with time.