Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **In-situ polymerization of PNIPA laponite nanocomposite gel**	**Experiment number**: **01-02-921**
Beamline: Dubble, SAXS	**Date of experiment**: from: **Sep 13 2010** to: **Sep 15 2010**	**Date of report**: February 10, 2011 *Received at ESRF:*
Shifts: 9	**Local contact(s)**: Giuseppe Portale	

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Purpose:

In this work we wanted to study a new nanocomposite gel, composed of the polymer poly(N-isopropylacrylamide) (PNIPA) with embedded laponite-clay nanoplatelets acting as cross links. In addition we also introduced magnetite nanoparticles into the system. The gels can be stretched to a high degree, by mechanical force, resulting in alignment of polymer chains. The 10 nm big magnetite particles are superparamagnetic and will align into chains and columns in magnetic fields, altering the mechanical properties of the gels.These two alignment procedures were expected to be highly recognizable in the scattering patterns. In this project we investigate the possibility of obtaining permanently aligned clay- and magnetite nanoparticles when the gels finish polymerizing, and in this experiment we wanted in addition to look at the evolution of polymer chains during polymerization.

Results:

We studied different PNIPA-Laponite gels with changing contents of magnetite particles and varying magnetic fields, both in-situ and ex-situ. The reaction solutions were filled into 2 mm capillaries shortly after the initiator had been added to start the polymerization reactions. The capillaries were mounted in either a custom made holder between two permanent magnets with the magnetic field perpendicular to the beam direction, or in a sample holder capable of holding several capillaries at once.

During the in-situ data collection on NC-gels without magnetite particles, there was no change in the curve shape with time, only a decrease in scattering intensity. During long exposure times the beam heated up the samples, and some of the water was expelled from the gel itself. When water was later reabsorbed the intensity was recovered. The preservation of the curve shape with time indicate that there are no inhomogeneities appearing in the material during polymerization. Therefore, the cross-linking with laponite platelets is likely to be homogeneous throughout the samples, which is an important result.

Anisotropic scattering patterns (Figure 1) were recorded for the NC-gels with embedded magnetite particles (FNC-gels) when they were situated in magnetic fields. The anisotropy was maintained in the samples after they were removed from the magnetic field, as we hoped for. Cake integration (Figure 2), in the parallel and perpendicular directions to the magnetic field, revealed structural changes at length scales bigger than 30 nm which corresponds well to chains consisting of 10 nm magnetite particles.

A detailed analysis of these data is in progress.

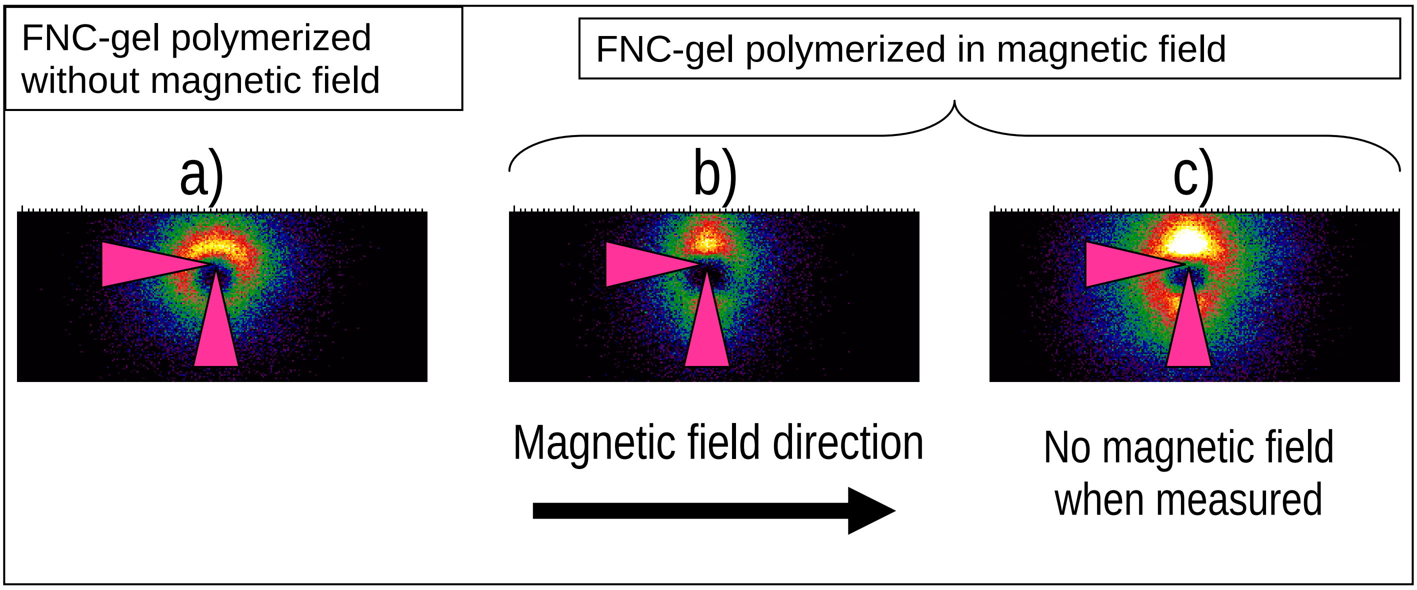

Figure 1: Scattering patterns from two FNC-gels: a) a gel polymerized without magnetic field and measured in absence of a magnetic field, b) a gel polymerized and measured inside a magnetic field and c) the same sample measured in absence of a magnetic field.

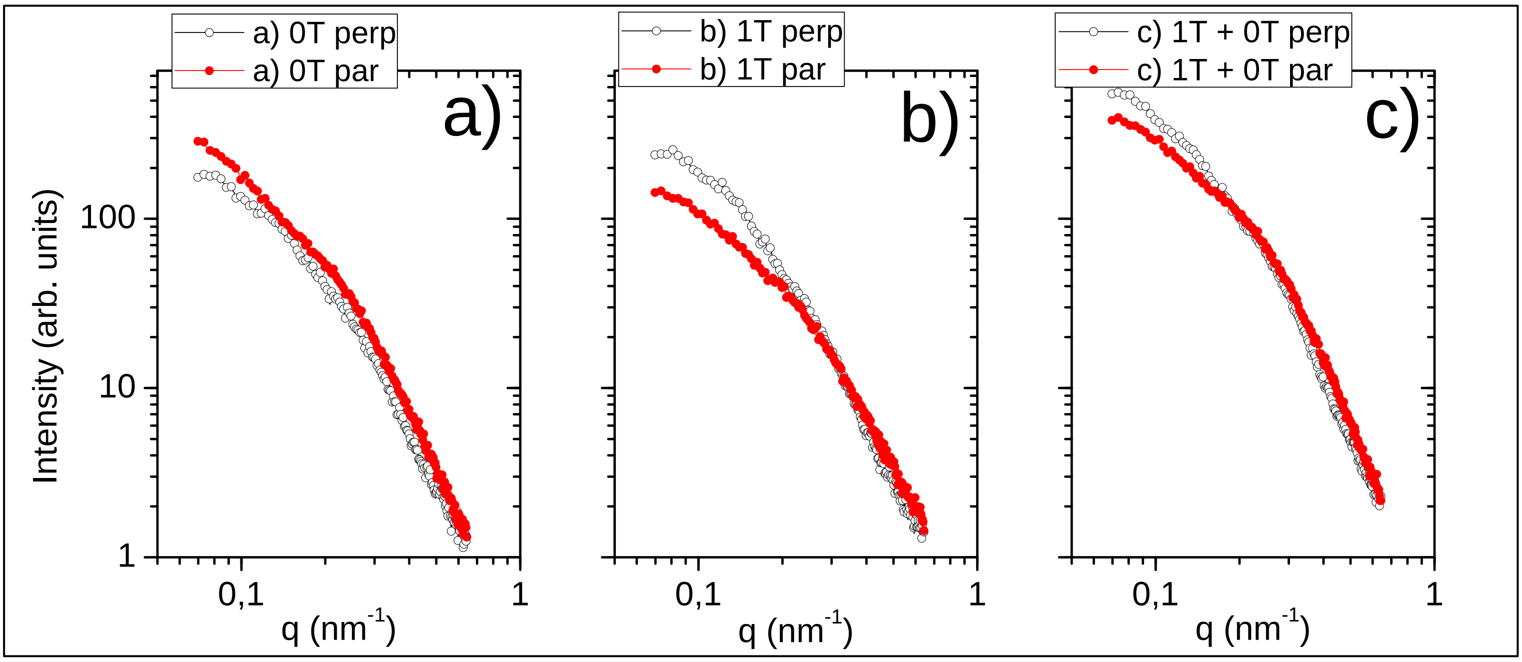

Figure 2: The resulting cake-integrations from the scattering patterns in *Figure 1*.