EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

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: Ultra-filtration process of anisotropic colloidal particles in magnetic fields**	**Experiment number**: SC-866
Beamline: IDO2	**Date of experiment**: from: 12-09-01 to:14-09-01	**Date of report**: 25-02-2002
Shifts: 6	**Local contact(s)**: T. Narayanan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Laboratoire de Rhéologie, Domaine Universitaire, B.P. 53 F-38041 Grenoble Cedex 9:

Frédéric Pignon*, Ayse Alemdar Yilmaz*, Albert Magnin*, Jean-Michel Piau

ESRF, BP 220, F-38043 Grenoble Cedex, France:

Theyencheri Narayanan*

Report: In these experiments we studied the structuring mechanism of deposits formed during the ultrafiltration process of aqueous anisotropic nano-particles in a magnetic field. The magnetic circuit developed on ID-2, which can provide very uniform magnetic fields of up to 1.4 Tesla was installed on the beamline. An aqueous suspension of the plate like clay such as montmorillonite (natural and Fe form) and rod like clay such as sepiolite have been used here. In the first set of experiment, the orientation of clay particles under different magnetic field strength (0-1.43 T) has been checked in steady conditions (P = 0 bar). The orientation of Fe-montmorillonite particles was observed in fields above 0.4 T (Fig.1) by means of SAXS experiments.

In the second set of experiment the ultrafiltration cell placed in the magnetic field was aligned with respect to the beam, then the cell was filled with Fe-montmorillonite dispersion and a pressure difference of 0.5 bar was applied to retentate. During the deposition process under the magnetic field (1 Tesla), we probed the structure of the deposit close to the membrane, 0.2 mm up to 4 mm above it and followed the time evolutions of particles orientations (Fig. 2a). Fig. 2b shows the SAXS pattern and variation of scattering intensity along the vertical and horizontal axes for a fixed Q value ($Q = 0.04\ nm^{-1}$) over time. The scattering intensity along the vertical axes dramatically increase at the shortest distance (z = 0.2 mm) at time t = 3000 s. Fig.3 compares the filtration results between the magnetic field being applied to the dispersion during the filtration and it being applied at the end of the filtration. The filtration of Fe-montmorillonite without magnetic field give rise to a cake consists of non oriented particles (Fig. 3a). After 2h of filtration, the magnetic field was sufficiently strong compared to the particle interactions in the cake to induce orientated structures (Fig. 3b). When the magnetic field was applied during the filtration procedure (Fig. 3c), the build up of the cake has a greater orientation than the cake, which has been submitted to magnetic field at the end of filtration (Fig. 3b). Simultaneously, the filtration experiments has shown that the filtration flux is enhanced under the magnetic field. In conclusion, the development of this new device under magnetic field has allowed us to control the orientation of the build up structure at the length scales of the colloidal particles, in order to fabricate new types of dense nano-materials with a controlled alignment of the objects.

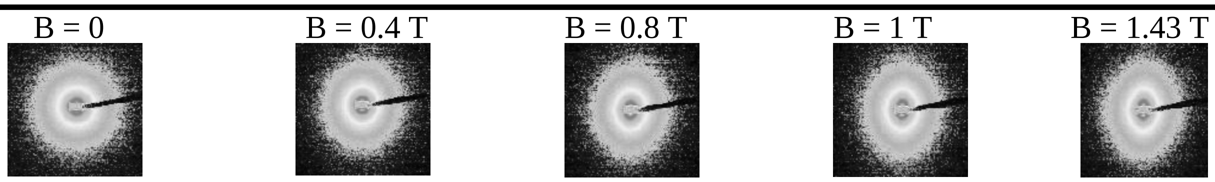

Fig.1 SAXS patterns of Fe-montmorillonite dispersion in a capillary under different magnetic field strength

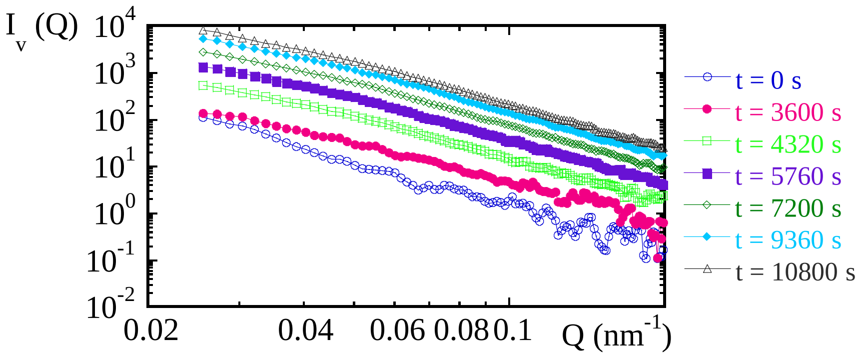

Fig 2a Variation of scattering intensity along vertical axis of Fe-Montmorillonite during time z = 0.2 mm

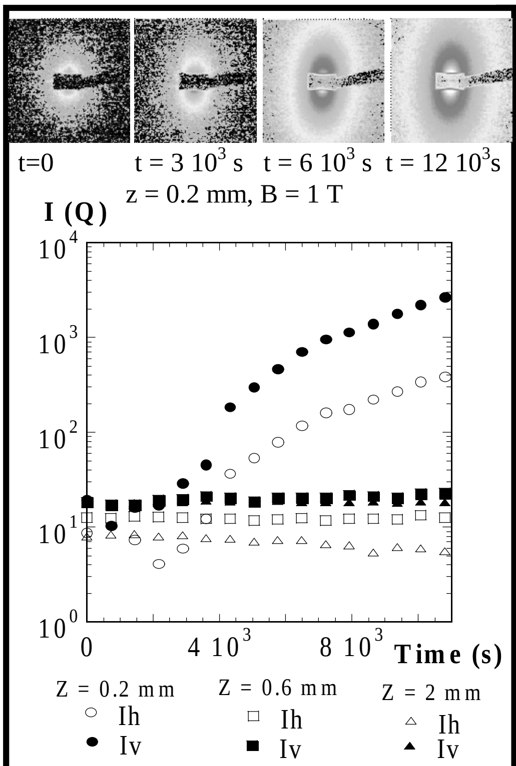

Fig.2b: Structure of build up cake under 0.5 bar in ultrafiltration process under magnetic field (1 Tesla). Variation of scattering intensity along the vertical and horizontal axes at Q=0.04 nm^{-1}

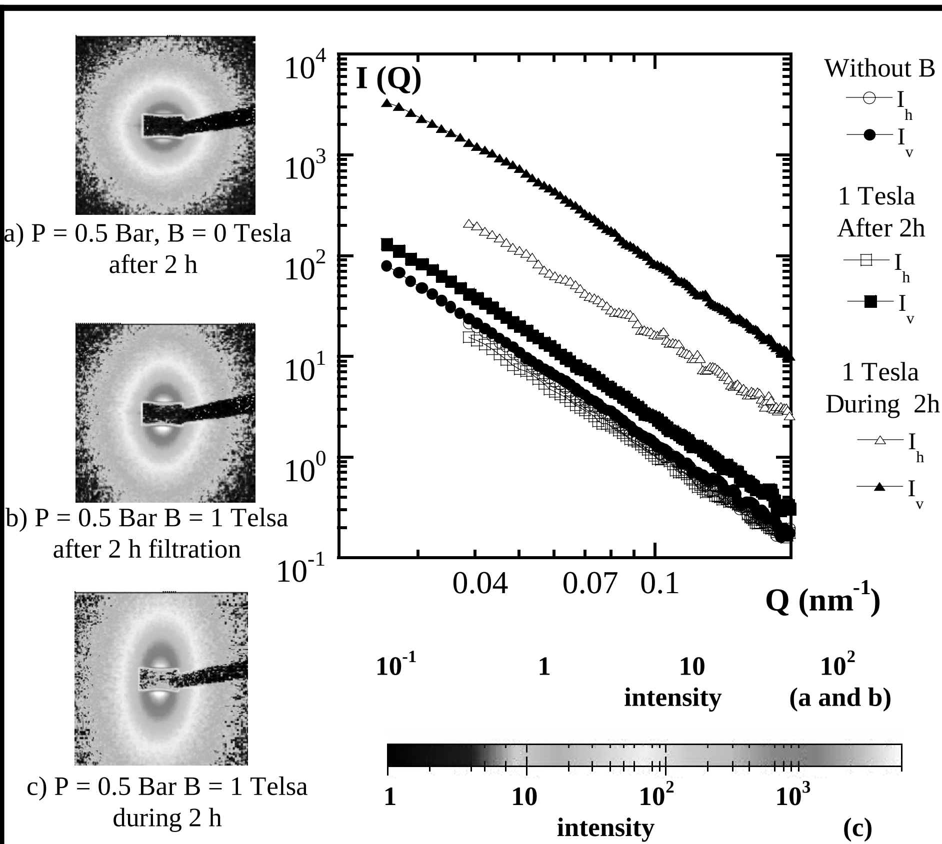

Fig.3: Effect of magnetic field applied during filtration and after filtration process of Fe-montmorillonite dispersions