ESRF	**Experiment title:** Dynamics and structure of electrorheological chain formation of synthetic nano-layered silicates	**Experiment number:** 01-02-634
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Shifts: 12	**Local contact(s):** Dr. Jon Are Beukes	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Jon Otto Fossum* (NTNU – Department of Physics, Norway
Kenneth Dahl Knudsen* (IFE – Institute for Energy Technology, Norway)
Knut Jørgen Måløy* (UiO – Department of Physics, Norway)
Yves Méheust* (NTNU – Department of Physics, Norway)
Davi de Miranda Fonseca* (NTNU – Department of Physics, Norway)
Kanak Parmar* (NTNU – Department of Physics, Norway)

Scientific background – motivation for the study :

Electrorheological fluids have the property of displaying drastic changes in their rheology when exposed to a sufficiently intense electric field. The simplest form of electrorheological fluid is a suspension of polarizable particules immersed in a non-polar fluid [1]; dipolar interaction between polarized particles is responsible for their aggregation along chains parallel to the electric field [1]. Nano-layered silicate suspensions, or clay suspensions, on the other hand, are colloidal suspensions of platelets that display a complex phase behavior depending among other things on the concentration in colloids, ionic strength of the solvent, and particle sizes. These properties have been investigated theoretically and experimentally using many techniques [2], among which synchrotron X-ray scattering methods [3, 4]. However, until now, no study of their behavior when exposed to an electric field had been reported.

Optical microscopy studies carried out last year in our laboratory at NTNU in Trondheim (Norway) on suspensions of fluorohectorite clays showed evidence of an electrorheological behavior for an electric field of a few kV/mm [5]. Fluorohectorites are synthetic swelling clays with a formula X_x-$Mg_{3-x}Li_xSi_4O_{10}F_2$. The colloidal particles consist of a stacking of $\sim$ 100 silicate platelets that are bonded together by an interlayer cation X, the nature of which influences the strength of the bonding and the interaction between particles in the solvent. Water can be intercalated mono-layer by mono-layer between the platelets, which we believe is responsible for the polarizability of the particles, since suspensions obtained from dried powder exhibit no electrorheology.

The WAXS experiments reported here were carried out in order to investigate a possible orientation of the fluorohectorite particles under the influence of the electric field, so as to determine the way they are polarized. The sensitivity to the nature of the intercalation cation on the electrorheology of the fluorohectorite samples was investigated, as well as the behavior of a natural marine clay.

Experimental method:

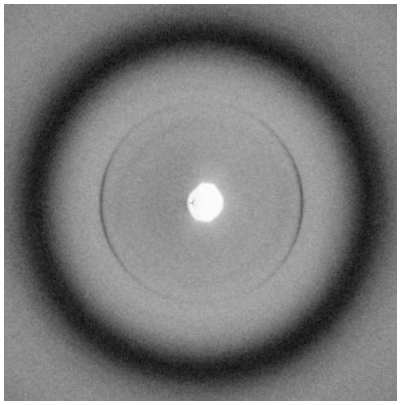
The four types of samples consist of fluorohectorite (X=Na, Ni and Fe) or marine clay particles immersed in a silicone oil (viscosity 100 cSt). They were investigated by wide angle X-Ray scattering

using a wavelength $\lambda = 0.6998$ Å. A cell that we specially designed allows to apply a high voltage E between 2 copper electrodes, between which the sample is contained. For E null, the figure obtained through diffraction by the Brownian particles clearly displays an isotropic (001) ring. For $E \sim 1$ kV, this rings becomes anisotropic (see Fig. 1(a)), showing evidence of a preferential orientation of the particles under the influence of the electric field, for the 4 types of samples. Diffraction patterns were recorded for $E = 0$ and as a function of time after applying $E \sim 1$ kV. The 12 shifts of beam time awarded were fully utilized.

Results obtained:

Analysis of the 2D diffractograms showed that the polarization vector induced inside the particles lies in the plane of their silicate sheets, which confirms our assumption concerning the role of intercalate water in the polarization process. A shift is the position of the (001) peak, i.e, in the spacing between the platelets inside a particle, when applying the electric field, further confirms that the polarization occurs between the platelets. The change in the intensity of the 2D diffractogram along the (001) ring was extracted as a function of azimuth for each sample (Fig. 1.b), from which the width of the orientational distribution for the particles submitted to the electric field was inferred. A publication relative to these findings will soon be submitted.

(a)



(b)

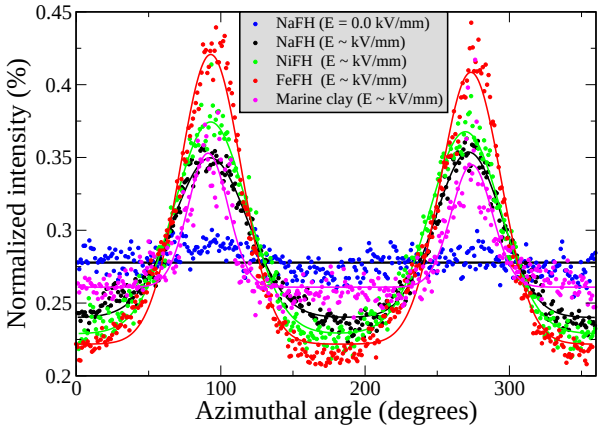


Fig. 1 – (a) Center region of a diffraction pattern obtained on a Na-fluorohectorite under an electric field of 2.0 kV/mm. The wide outside ring is the diffuse diffraction pattern due to the oil; the anisotropic ring inside corresponds to the (001) diffraction peak for the partially oriented clay platelets. (b) Intensity curves along the anisotropic (001) rings similar to that shown in (a), for the three types types of fluorohectorite and the marine clay. The width of the Gaussian fits corresponds to the dispersion in the orientations of the diffracting platelets.

References

[1] Tian Hao. Electrorheological fluids. *Adv. Mater.*, 13:1847, 2001.

[2] J. O. Fossum. Physical phenomena in clays. *Physica A*, 270(1-2), 1999.

[3] E. DiMasi, J. O. Fossum, T. Gog, and C. Venkataraman. Orientational order in gravity dispersed clay colloids: A synchrotron X-ray scattering study of Na fluorohectorite suspensions. *Phys. Rev. E*, 64:061704, 2001.

[4] G.J. da Silva, J. O. Fossum, and E DiMasi et al. Synchrotron X-ray scattering studies of water intercalation in a layered synthetic silicate. *Phys. Rev. E*, 66(1):011303, 2002.

[5] J. O. Fossum, R. K. Sangar, K. P. S. Parmar, G. J. da Silva, and Y. Méheust. Observations of electric field induced ordering and chain formation in oil suspensions of a synthetic nanolayered silicate. Submitted to ENERGY, the International Journal.