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Beamline: BM26B	**Date of experiment:** from: 07/05/2003 at 07:00 to: 09/05/2003 at 07:00	**Date of report:** 01/09/2003
Shifts: 6	**Local contact(s):** Dr. Igor DOLBNYA	*Received at ESRF:*

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Report:

Clay suspensions are examples of complex liquids, showing incredibly rich phase behaviours, and as such the study of these complex fluid systems are of great interest from the point of view of basic science. In addition, clay suspensions are technologically important, and the understanding of the microscopic organisation behind the non-linear and very diverse macroscopic behaviour may aid in controlling the relevant parameters for certain applications. In particular, understanding the role of salt in stabilising dense clay phases may have practical significance, for example in the context of clay formations originally sedimented in salt water, which can be progressively destabilized by subsequent freshwater rinsing.

We investigate the internal structure of clay/water systems in order to elucidate the organisation of clay building blocks on different length scales, with the aim of understanding the relationship between structure and macroscopic behaviour.

In the present experiment colloidal suspensions of clay particles in aqueous salt solutions have been studied; namely fluorohectorite with nominal chemical formula $Na\text{-}(Mg_2Li)Si_4O_{10}F_2$. After being gravity dispersed, these suspensions present separation in up to four phases with numerous gel and sol structures possible (see Figure 1). Amongst them a lyotropic nematic phase has been identified.

Therefore the goals of the experiment are:

a) To obtain quantitative information on this nematic phase;
b) To study the phase transition from isotropic gel to nematic gel;
c) To map out the phase diagram.

We prepared several aqueous samples 1% and 3% weight in of fluorohectorite with varying ionic strength (from $1x10^{-5}$ to $1x10^{-1}$M NaCl) in different sample tubes/capillaries, totalizing 53 different samples (plus backgrounds). And we sent them to ESRF site one month before the beginning of the experiments in order to allow them to settle owing to gravitational forces.

During experiment we collected both two-dimensional small angle x-rays scattering and one-dimensional wide angle x-rays scattering patterns from fluorohectorite using 1.55 Å x-rays at the different sample layers (strata). The samples were kept at ambient temperature and pressure.

In order to accomplish the goals we performed measurements at 45 different heights in steps of 0.5mm for each sample. Using a counting time of 60 seconds since this was the longest that we could count on the sediments while avoiding overflow. Then the transmission scans were performed for horizontally at certain different heights in the capillaries in steps of *c*. 0.19mm.

The data analysis is ongoing and it is in agreement with other experiments, since the nematic phase observed in the past is also present, as well as, the isotropic to nematic transition (see Figure 2). The current analysis is quantifying the anisotropy level and the analysis so far has been successful in providing quantitative information about the observed nematic phase.

The investigation of the phase transition must be preceded by the above analysis of anisotropy level as function of height and therefore nothing can be stated right now.

Up to this time nothing has been done about the mapping of the phase diagram, however the observed phases are in agreement with what can be found in the literature[†].

Hence the current data analysis indicates that the experiment was a successful one and we expect to publish our results soon.

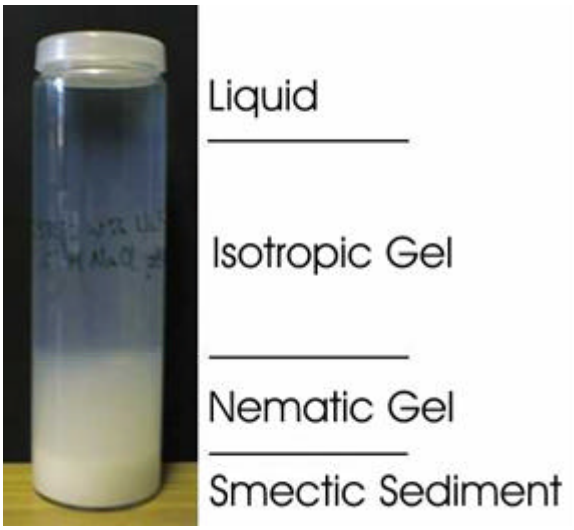

Figure 1. One of our samples showing four strata due to gravitational forces, that effectively sorted the particles by size.

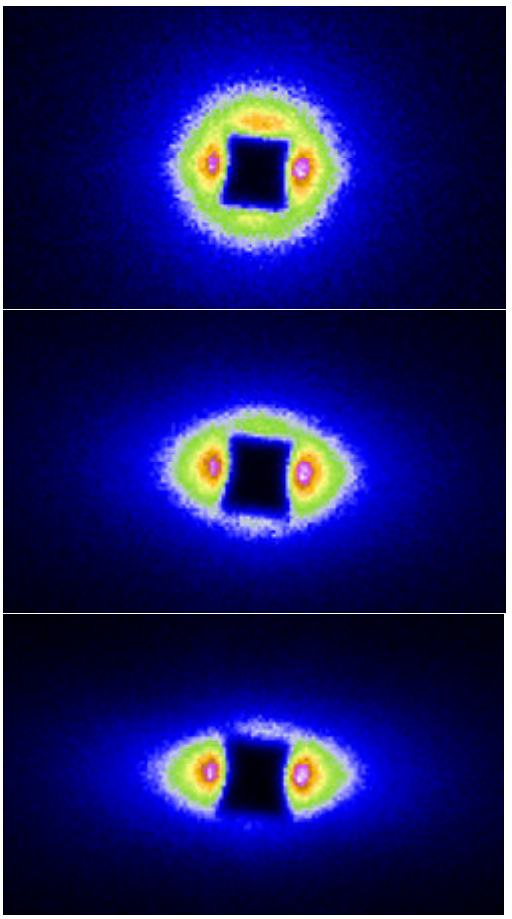

Figure 2. Small Angle X-Ray Scattering 2D images of the Isotropic Gel (upper), Nematic Gel (lower) and the transition zone between these two phases (middle).

[†] E. DiMasi *et al*., Phys. Rev. E **64**, 061704 (2001).