EXPERIMENT REPORT   SC 725   19/2/01

Experiment title: “SAXS by isotropic and nematic suspensions of goethite under a magnetic field.”

Beamline: ID02

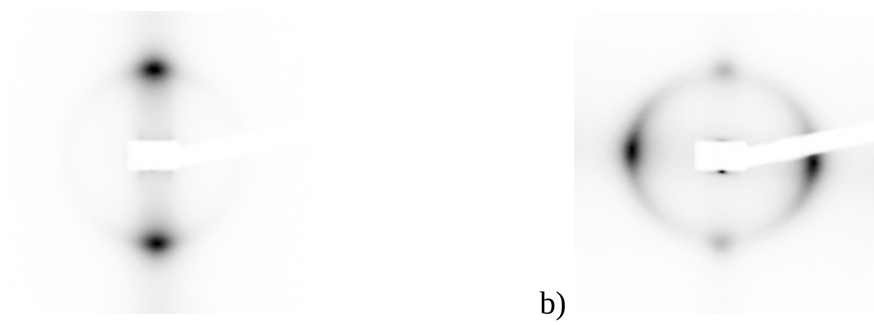
Date of experiment: 7/10/00 – 10/10/00
Shifts allocated: 8
Local Contact: Pierre Panine

Applicants: Patrick Davidson
Bruno Lemaire
Laboratoire de Physique des Solides, Bât. 510
Univ. Paris Sud, 91405 Orsay cedex

REPORT

Aqueous suspensions of goethite (α-FeOOH) crystallites form one of the very few examples of mineral liquid crystals. Transmission electron microscopy, powder X-ray diffraction and the present SAXS study on very diluted suspensions have shown that these particles are on average about 150 nm long, 20 nm wide and 5 nm thick. Polarised light microscopy has shown that these particles organise in a lyotropic nematic phase at high enough volume fraction ($\phi \approx 10$ %). The isotropic/nematic phase transition is first order, with phase coexistence, and can be described by the Onsager model.

We had previously discovered, by optical microscopy and by X-ray scattering experiments both in-house and at LURE, that these liquid crystalline suspensions had outstanding magnetic properties. The use of the magnetic-field device developed by Dr Pierre Panine, our local contact on ID02 during the SC725 experiment, has allowed us to investigate this behaviour in detail. We obtained excellent quality SAXS patterns of the different orientation states of the nematic phase at various concentrations and magnetic fields. We have thus demonstrated that the nematic phase aligns in a very low magnetic field ($\approx$ 10 mT), as compared to other lyotropic liquid crystals (several hundreds of mT). Moreover, the particles orient parallel to a small field (B < 350 mT) (Fig. 1a) but they reorient perpendicularly in higher fields (B > 350 mT) (Fig.1b). To the best of our knowledge, this magnetic behaviour is unprecedented in the field of liquid crystals.



a) b)

Figure 1: SAXS patterns of the nematic phase aligned in a horizontal magnetic field of a) 25 mT and b) 600 mT

Actually, this unusual behaviour is also observed with the isotropic phase. We could easily record the SAXS patterns of isotropic suspensions at different volume fractions as a function of the magnetic field intensity. We observed that the isotropic phase becomes highly anisotropic in a magnetic field and, at large enough volume fraction, the particles orient parallel to the field then perpendicular upon increasing field intensity. This shows that the magnetic properties of the phase simply reflect those of the individual particles but they are enhanced by collective effects in the nematic phase. The study by SAXS of the orientational order of these suspensions versus field intensity has allowed us to corroborate and better understand our previous magneto-optic observations.

The beam quality of ID02 combined with the use of the magnetic-field device made it possible to observe a very interesting and completely unexpected phenomenon. Oddly enough, we noticed that the nematic suspensions of goethite undergo a reversible colloidal crystallization at high field (B > 900 mT) (Fig.2). Moreover, single crystals of this crystalline phase formed reproducibly in-situ, which allowed us to identify unambiguously the space group of the phase: c2mm. It is a 2-dimensional space group because the particles only order perpendicularly to their main axis. Moreover, we could study this colloidal crystallization as a function of volume fraction and field intensity, thus mapping this part of the phase diagram. The colloidal crystalline phase also exists in the absence of field at very high volume fractions ($\phi \approx 20$ %) but it is clearly stabilized by the field, at lower volume fraction ($\phi \approx 10$ %), at the expense of the nematic phase. This phenomenon, discovered at ID02, is also highly original in the field of liquid crystals.

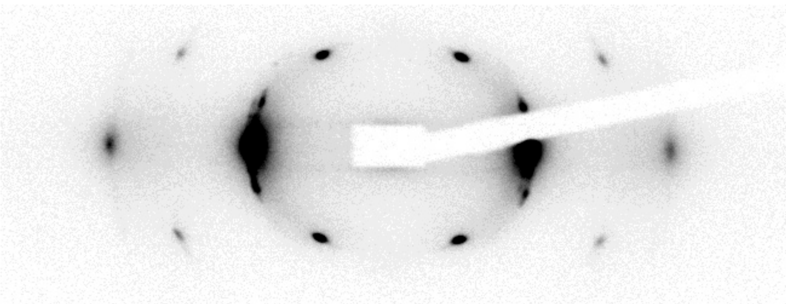


Figure 2: SAXS pattern of the colloidal crystalline phase grown under the influence of the magnetic field (horizontal).

From a more technical point of view, the experiments proved rather easy with typical exposure times of fractions of seconds with attenuators. Both configurations of the magnetic-field set-up (field parallel and perpendicular to the X-ray beam) were used to fully characterize the orientation of the suspensions and the c2mm space group.

All these experimental results are presently being carefully examined and we are writing an article describing the various orientations of these suspensions as a function of volume fraction and field intensity. Another future article should describe the colloidal crystallization under magnetic field.