	Experiment title: Biaxial nematic phase in colloidal goethite dispersions	**Experiment number**: 26-02-466
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Report: (max. 2 pages)

The following actions were taken in preparation for this experiment. The cryotiger (cryogenic cooler of the second crystal of the monochromator) was switched off to minimize the vibrations. After the monochromator had warmed up, the turbo pump mounted on the monochromator was also switched off for the same purpose. The second crystal and the mirror were made flat as much as possible to avoid beam focusing before the experimental hutch. Beryllium lenses were installed on top of a column including 4 rotation and translation remotely-controlled stages for their alignment. Two Photonic Science CCD detectors were used to measure scattering intensities. The high resolution Photonic Science detector with the pixel size of 9 micron square, which is purchased by our group, was used to get highest angular resolution. Another Photonic Science detector (pixel size of 22 micron square) was used to access wider q-range. The sample capillaries were mounted on the sample holder of a 1.5 Tesla permanent magnet, which was borrowed from the beamline ID-02. The magnet was installed on top of two translation axes to allow scanning through the samples. The magnet has two sets of poles, which can generate a field either orthogonal or parallel to the x-ray beam. In addition, we have brought our home-made 0.7 Tesla permanent magnet in order to pre-align some of the samples in the magnetic field before the measurements.

Goethite suspensions show very rich phase behaviour in the presence of an external magnetic field [1,2]. At relatively low field strength the particles align along the magnetic field due to their (relatively weak) permanent magnetic moment appearing in nanoparticles due to incomplete compensation of the magnetic moments of the two anti-parallel magnetic sublattices. At stronger fields (> ~250 mT) the induced moment becomes more important and the particles turn perpendicular to orient their easy axis along the field.

During this experiment we have studied in detail the suspensions of the goethite particles with their $L/W \approx W/T$, where L, W and T are, respectively, the length, the width and the thickness of the board-shaped goethite particles. At this relation between the aspect ratios one can expect the formation of biaxial nematic phase. Indeed, the combination of the very high resolution of the microradian setup at DUBBLE and a convenient magnet with easy switchable direction of the magnetic field allowed us to collect convincing evidence of the formation of the biaxial nematic as well as biaxial smectic phase.

We would also like to highlight here some of the results on the field-induced reorientation of the smectic phase. In a thermotropic smectic phase a rather complex reorientation process was observed [3]. To illustrate our results for the lyotropic smectic phase, in Figure 1 we present patterns measured in a capillary of sterically stabilized particles in toluene with a volume fraction of 3.5 % (due to a sedimentation profile the volume fraction in the smectic phase is actually much higher). It is exposed to a 120 mT magnetic field in the horizontal direction perpendicular to the x-ray beam. In the vicinity of the beamstop one can clearly see two sets of very bright and sharp reflections. These reflections originate from the interlayer periodicity in the smectic phase of goethite nanorods. Note the reflections up to fifth order are visible. This indicates that the modulation of the electron density has sharp edges, i.e. the smectic layers are very well defined. The initial orientation (before applying a field) is with the sharp reflections on the vertical line. Orthogonal to the peaks of the initial orientation new peaks are observed, originating from smectic domains aligned parallel to the field. This suggests that the reorientation process does not occur gradually, via intermediate orientations, but nucleation of domains with an orthogonal orientation (compared to the initial orientation) occurs. These domains grow in time until at some point all particles will probably orient in the field. Higher in the sample (Figure 1A/C, at a lower volume fraction) the reorientation process is faster.

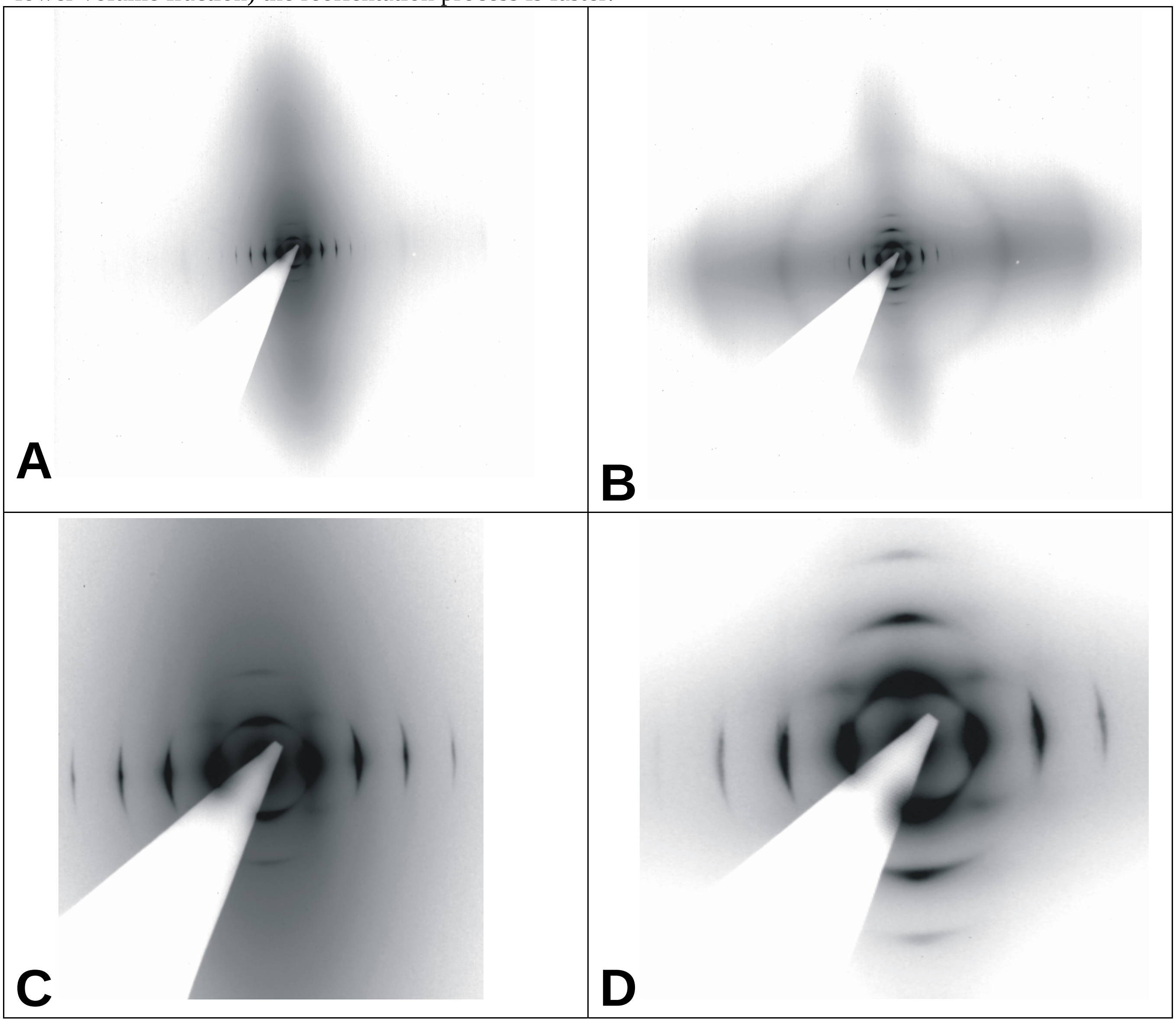

Figure 1. Panels A and B present examples of the microradian diffraction patterns measured at slightly different hights in a goethite suspension (A/C is 0.5 mm higher). An external magnetic field of 120 mT is applied in the horizontal direction normal to the x-ray beam. Corresponding zooms into the area of the smectic reflections are given in C and D, respectively.

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[2] G.J.Vroege et al., Advanced Materials **18**, 2565 (2006)
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