DUBBLE	Experiment title: Liquid crystalline and magnetic behaviour of Goethite colloids	Experiment number: **26-02-239**
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Report: (max. 2 pages)

The experiment was devoted to the identification and the detailed characterization of various liquid-crystalline phases formed by colloidal goethite (FeOOH) nanorods [1,2] with and without external magnetic field. For the experiment we have made use of the developing microradian-resolution scheme [3]. To increase the transverse coherence length of the x-ray wave, the second crystal of the monochromator and the focusing mirror were made as flat as possible to avoid beam focusing. Instead, the beam (photon energy 16 keV, wavelength 0.77 A) was focused at the position of the detector by a compound refractive lens (CRL), which was installed in the experimental hutch. The CRL was mounted on a lens goniometer to allow for a remote computer-controlled adjustment of its position and orientation. One of the limitations of the CRL is its limited aperture, which could significantly reduce the maximum scattering angle if the lens was mounted *after* the sample as it was in our recent experiment 26-02-253. The mineral liquid crystals formed by colloidal nanorods display a diverse range of spatial scales ranging from about 500 nm in the direction along the rod to several tens of nanometres in the two orthogonal directions. Thus, both the high angular resolution as well as the access to relatively large scattering angles was needed. To avoid problems related to the lens aperture, we have positioned the lens just *before* the sample.

The samples (contained in flat glass capillaries) were placed in between the poles of an electromagnet prepared in Utrecht for this experiment. The magnet is sufficiently light and compact to allow for its installation on a sample goniometer. At the same time, it allows to generate magnetic fields up to 0.6 Tesla in the sample, which was sufficient to observe particle re-orientation phenomena.

Most of the scattering patterns were recorded with the new Photonics Science CCD detector of DUBBLE with the pixel size corresponding to 22 microns at the phosphor screen. This detector also has a large field of view (4000×2670 pixels of about 88×37 mm^2), which allowed for data collection in a sufficiently large range of angles. This was particularly important for the samples studied here, which exhibit coexistence of large- and short-period structures. Several patterns are also recorded with a high-resolution CCD detector (effective pixel size at the phosphor screen of 1.1 micron), which was kindly provided by the ESRF detector pool. The two detectors were mounted on a computer-controlled translation stage to allow for a rapid switch from one detector to the other.

The goethite nanorods display very peculiar behaviour in an external magnetic field, which is governed by the interplay of the permanent and the induced magnetic moments [1]. During the experiment we have collected a large amount of high-quality data in samples of particles from several synthesis batches (with slightly different sizes), at various concentration and for a number of magnetic field strengths. As an example of the collected data, we show in Figure 1 the SAXS patterns of a colloidal goethite sample of volume fraction $\varphi = 11\%$ submitted to an increasing magnetic field (field direction is shown in panel b). Before application of the magnetic field the pattern is typical for a uniaxial nematic phase (panel a). In a weak field

(panel b) the structure partly transforms into a smectic A phase with the longest axis of the particles (corresponding to the direction of the permanent magnetic moment) oriented along the field. Features marked with “1” and “2” originate respectively from the interplanar periodic structure and the fluid-like intraplanar structure of the smectic phase. Partly, the system also transforms into a biaxial nematic phase with the shortest particle axis (corresponding to the direction of the magnetic easy axis) along the field, leading to the features marked with “3” in the pattern. In figure 1d, one can clearly see many features (marked with “4”), which form a distorted hexagonal net and originate from the columnar phase. Yet, a certain amount of the smectic A phase (features “1” and “2”) is still present. The transient state between 1b and 1d and the development of the biaxial nematic domains towards the columnar phase is illustrated in panel (c).

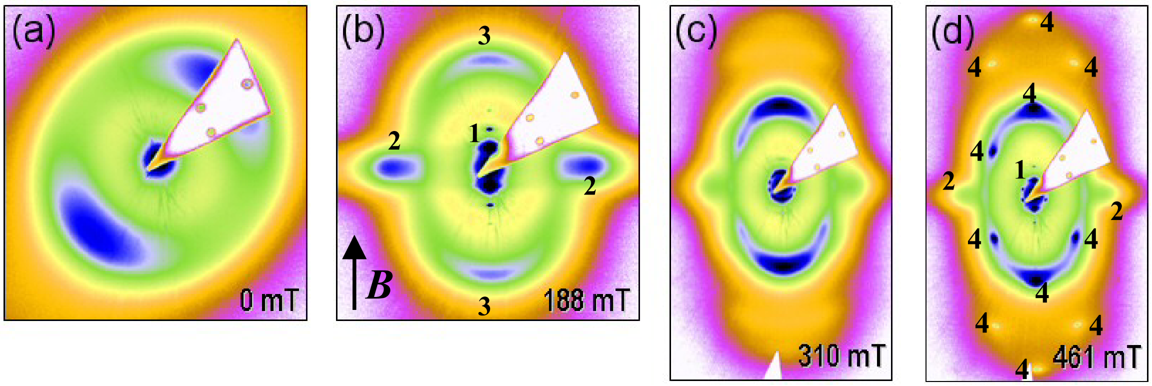

Figure 1: SAXS patterns of a colloidal goethite sample of volume fraction φ = 11 % submitted to a vertical magnetic field of intensity (a) B = 0 mT, (b) B = 188 mT, (c) B = 310 mT, (d) B = 461 mT. The arrow in (b) displays the orientation of the external magnetic field.

The importance of the used microradian resolution is further illustrated in figure 2, which displays zooms into the smallest-angle regions of figures 1b-1d. The lowest-order diffraction peaks appear at a 2θ angle as small as about 180 μrad and are very well resolved. Figure 2a clearly shows the sharp peaks corresponding to the smectic A phase with the interplanar spacing of about 430 nm. As many as three diffraction orders are clearly visible. In figure 2b, the smectic domains start to rotate towards a perpendicular orientation due to the induced magnetic moment. In figure 2c, it is visible that some of the smectic domains still hold their orientation with the particle long axis parallel to the field. We have also observed two smaller (presumably, smectic) domains with a turned orientation (features “5” in Figs. 2b and 2c), the origin of which is still a subject of investigation.

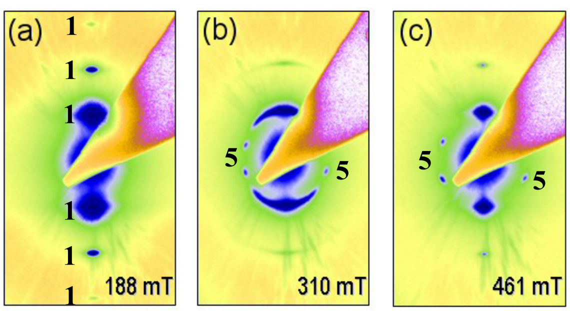

Figure 2: Enlarged portions of the SAXS patterns in Fig. 1(b)-(d).

At the same time, we have faced several problems of the microradian-resolution setup. A significant level of scattering originating from the focusing lens has been observed. It is clearly seen in Fig. 2 as ‘strikes’, which especially disturbed the measurements of weakly-scattering samples. The mechanical instabilities of the elements in the optics hutch still remain an important issue. The direct beam is observed to significantly “jump” on the detector, mostly in the vertical direction. This partly smears out the diffraction features. These problems have to be addressed in our future experiments in collaboration with the DUBBLE staff.

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References

[1] B.J. Lemaire, et al., Phys. Rev. Lett., 88, 125507 (2002).
[2] report of the experiment 26-02-213.
[3] reports of the experiments 26-02-253 and SC-1418.