ESRF	**Experiment title:** **An XPCS investigation of doped lamellar phases**	**Experiment number**: SC-2085
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Report:

The purpose of this experiment was to study the dynamics of nanoparticles confined between the lamellae of liquid crystalline lamellar phases. We had chosen three different systems of doped lamellar phases: a. Goethite nanoparticles in a swollen $C_{12}EO_5$ lamellar phase, b. Silica nanoparticles in a swollen $C_{12}EO_5$ lamellar phase, c. Polyoxometalate anions in a $C_{12}EO_4$ lamellar phase.

The lamellar phases were sucked into flat optical capillaries of 50μm thickness (with 50μm glass walls). The phase was aligned macroscopically with the lamellae parallel to the face of the capillary. We expected to study both the diffusion of particles parallel to the lamellae with the beam perpendicular to the capillary or the dynamics of the lamellae in reflectivity.

In order to avoid beam damage to the samples we chose to work at 12 keV, hoping that the beam would interact less with the lamellar phase. After the first day of measurements we were still facing beam damage problems so for the rest of the experiment we were working at 8 keV which has the benefit of increased flux and coherence. However, in lamellar phases the signal was disturbed by the texture fluctuations under the beam and could not be reasonably analysed. Thus, we switched to a system that proved to be more robust, namely free suspensions of goethite nanoparticles in water.

Goethite suspensions

The goethite nanoparticles[1] are rodlike and polydisperse, with an average size of $150\times25\times10$ nm^3. They also have interesting magnetic properties: when subjected to a magnetic field, they align themselves either parallel to the field (for B<250 mT) or perpendicular to it at higher field values. At concentrations above 8.5 vol%, the suspensions go through a phase transition to a nematic phase.

The samples that we used were two isotropic goethite suspensions at 4% and 7.3% volume fractions and a nematic goethite supension (at 8.5 vol%). The measurements were done either in the absence or the presence of a magnetic field that was applied perpendicularly to the beam. The strong magnetic field aligned the particles perpendicularly to the field, so their X-ray signal was concentrated along the direction of **B**. The measurements were also performed along this direction.

[1] B. J. Lemaire et al., *Eur. Phys. J. E* **13**, 291-308 (2004)

For each sample, a series of intensity autocorrelation functions g(t) was obtained at different q values.

After normalization by the autocorrelation signal of the monitor, $g(t)$ was fitted with a stretched exponential function: $g(t) = 1 + A[\exp(-\Omega t)^{\beta}]^2$, where the relaxation rate Ω yields the diffusion coefficient D via: $\Omega = Dq^2$. A representative fit obtained this way is shown in Figure 1.

From plots of Ω against q^2, we obtained the diffusion coefficient for different samples; the corresponding values are given in Table 1.

The dispersion relations obtained for the first two samples are shown in Figure 2 (left). The solid triangles are the results obtained in the absence of the magnetic field, while the open dots were measured on the same suspension in the presence of a strong magnetic field. Clearly, the presence of the field slows down the dynamics of the particles. This effect is still in need of an explanation.

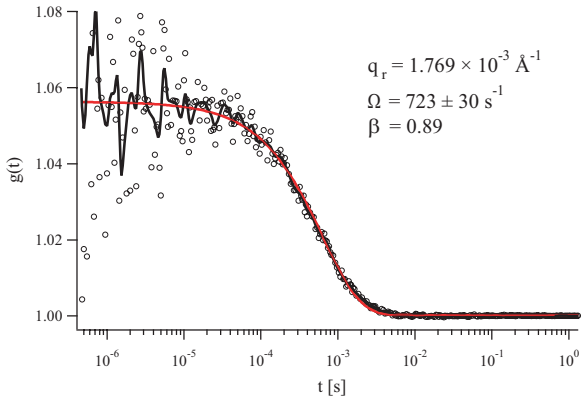

Figure 1: Raw correlation function (symbols) and smoothed curve (solid line) as well as the fit by a stretched exponential.

Sample	D [10^{-12} m^2s^{-1}]
4% B=0	3.06 ± 0.08
4% B=0.875 T	2.17 ± 0.04
7.3% B=0	2.5 ± 0.8
7.3% B=0.875 T	1.29 ± 0.02
8.5% (nematic)	1.01 ± 0.02

Table 1: Values of the diffusion coefficient for all samples.

In the nematic phase, the strong interparticle interactions give rise to a correlation peak (Fig.2, right). We were able to measure the diffusion constant (in the direction perpendicular to the director) at the position of the peak, for q values substantially higher than those accessible to DLS measurements. However, we were reaching the limits of the experimental resolution, so that the variation of $D(q)$ around its average value cannot be interpreted.

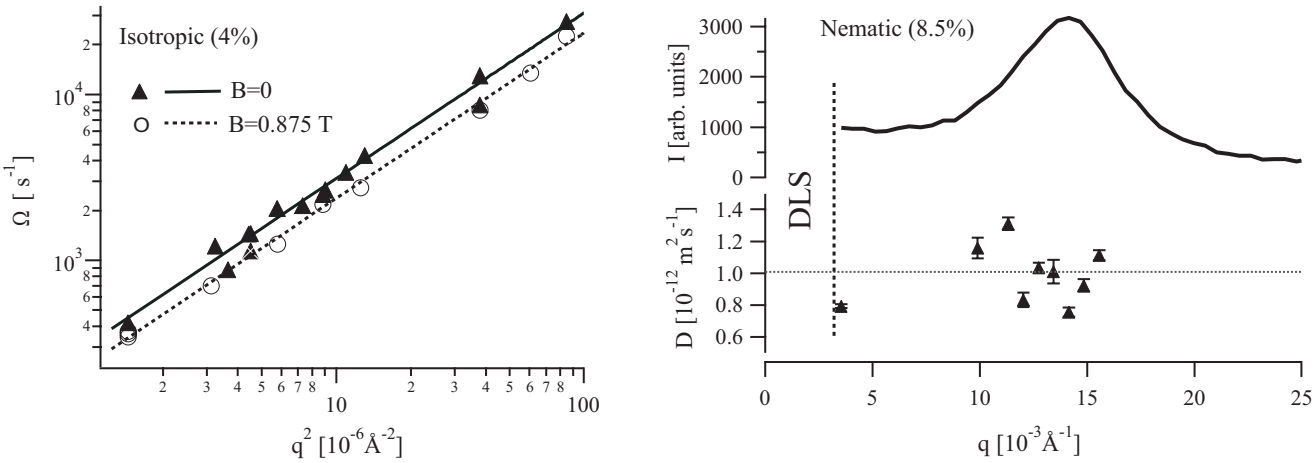

Figure 2: (Left) Dispersion relations for a 4% goethite suspension, with and without magnetic field. The lines are linear fits through the points. (Right) Diffusion coefficient of nematic goethite at different q values. The upper curve shows the static scattering intensity over the same q range. The peak due to the structure factor is clearly visible. Only the q range to the left of the dotted line is accessible to dynamic light scattering.

In conclusion, goethite nanorods have proven to be a very promising system for studying the dynamics of colloidal suspensions of anisotropic particles. In the isotropic phase, the diffusion coefficient could be measured, with and without an applied magnetic field. The magnetic field slows down the diffusion noticeably. This effect deserves a more systematic investigation.

In the nematic phase, we were able to measure the diffusion coefficient up to q values corresponding to the correlation peak due to interparticle interactions. Although the values of $D(q)$ presented here are quite scattered, the precision can be greatly improved by slowing down the dynamics by adding glycerol to the suspensions and/or cooling down the samples. Thus, the "mesoscopic" collective dynamics of the nematic phase can be probed.