ESRF	**Experiment title:** **Anisotropic particles confined in lamellar phases**	**Experiment number**: SC-2393
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Report:

The purpose of this experiment was to perform SAXS measurements on a recently formulated system[1], consisting of aligned samples of lyotropic lamellar phases doped with rodlike magnetic nanoparticles. The expected results were the structure factor of the inclusions and, deriving from it, the interaction between the inclusions induced by the presence of the lamellar phase.

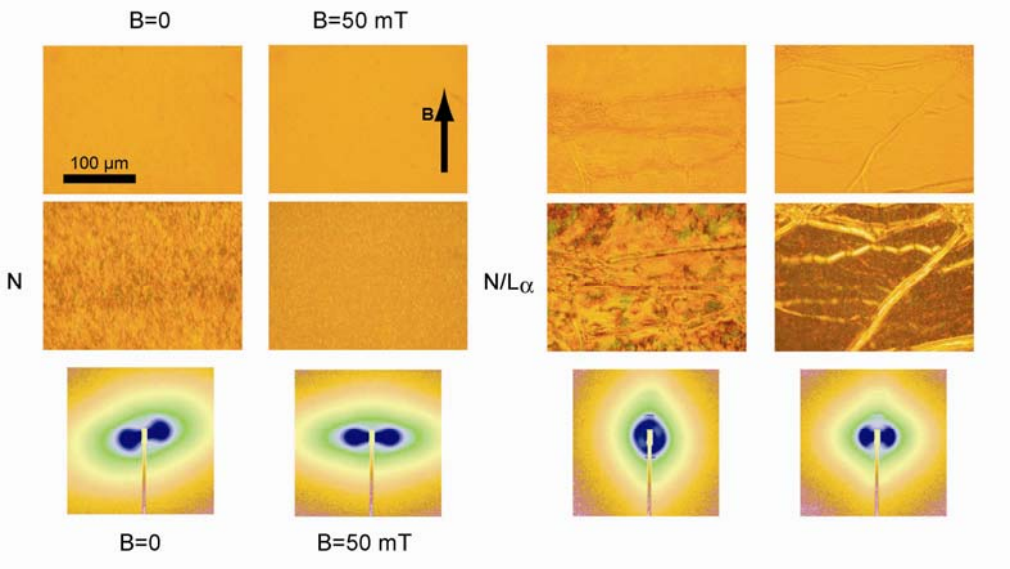

Figure 1: Optical microscopy textures (top) and SAXS signal (bottom) of the nematic phase of goethite, at a concentration $\phi_g = 8$ vol %, in water (left) and contained within the lamellar Lα phase (right). The lower microscopy images are taken between crossed polarizers. In both cases, the nematic phase is very well aligned along the magnetic field. In the panel on the right, the lamellar phase is almost completely aligned in homeotropic anchoring (the bilayers are parallel to the flat faces of the capillary.

The X-ray energy was maintained at 12.46 keV ($\lambda = 0.995$ Å) and the sample-to-detector distance was 5 m. The accessible wave vector ange was $0.02 < q < 0.9$ nm^{-1}. The typical exposure time was 0.1 − 0.5 s, chosen in order to make best use of the dynamic range of the FReLoN camera. The magnetic field (perpendicular to the X-ray beam) was applied using a motorized setup available at the beamline and based on permanent magnets. The maximum attainable field value is $B = 1.5$ T.

The lamellar phases under study all had a membrane volume fraction $\phi_m = 6.27$ %, while the volume fraction of goethite particles ϕ_g varied between 0.5 and 8 vol %. The samples were contained in flat glass capillaries (50 µm thick and 1 mm wide) and initially oriented homeotropically, i.e. with the director perpendicular to

[1] K. Béneut, D. Constantin, P. Davidson, A. Dessombz, and C. Chanéac. Magnetic nanorods confined in a lamellar lyotropic phase. *Langmuir* **24**, 8205-8209 (2008).

the walls. The incident beam was normal to the glass walls. Since at high enough field values (0.35 T, typically) the orientation of the doped phases switches from homeotropic to planar[1], i.e. with the lamellar director parallel to the walls (and this configuration is maintained after field removal), the samples could be studied with the incident beam either parallel or perpendicular to the lamellar director.

For the highest goethite concentration (8 vol %), the nanoparticles form a nematic phase inserted within the surfactant lamellar phase (Figure 1).

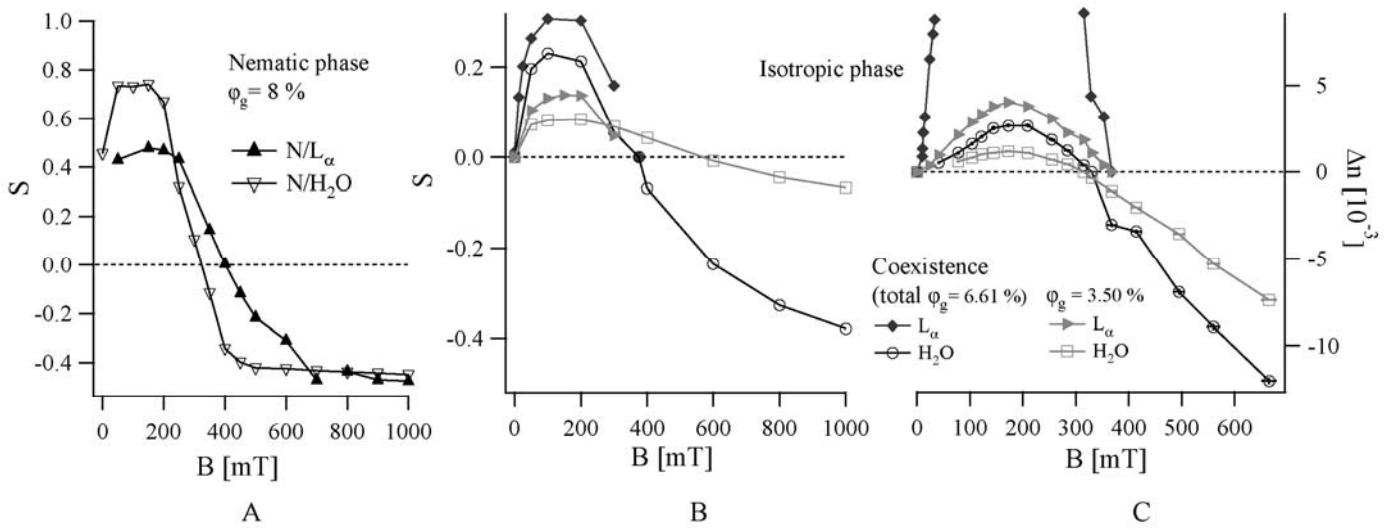

Figure 2: A − Order parameter of the nematic phase of goethite nanorods, in the lamellar phase (solid triangles) and in water (open triangles) as a function of the applied magnetic field. B − Induced order parameter and C − induced birefringence of the isotropic phase of goethite nanorods as a function of the applied magnetic field, for two concentrations ϕ_g. The symbols are the same in subfigures B and C.

The signal given off by the nanorods in the SAXS images can exhibit a certain anisotropy, directly related to the order parameter *S* (intrinsic, in the nematic phase, or induced by the magnetic field, in the isotropic phase). The order parameter is shown as a function of the applied field *B*, in the nematic and isotropic phases, both for the aqueous solutions and for the nanorods confined within the lamellar phase (Figure 2, A and B). For the isotropic system, we also show the field-induced birefringence Δn (Figure 2, C). Both the order parameter and the birefringence are higher in the confined system, showing that the lamellar host enhances the field susceptibility of the nanorod solution. On the other hand, the low-field order parameter in the N/L$_\alpha$ phase ($S \sim 0.45$) is much lower than in aqueous solution, N/H$_2$O ($S \sim 0.75$).

Both the lower order parameter in the nematic phase and the higher susceptibility in the isotropic phase are compatible with a second-order phase transition, as opposed to the first-order transition in the aqueous system.

Finally, the presence of the inclusions affects the structure of the host lamellar phase which becomes stiffer, as seen by the decreasing width of the Bragg peak with increasing goethite concentration (data not shown). We can tentatively attribute this effect to a strong interaction between the nanorods and the surfactant bilayers, leading to the formation of hydrogen bonds. The nanorods are thus adsorbed onto the bilayers and increase their stiffness.

A first paper based on these results has already been submitted for publication[2], and a second one is under way, dealing with the detailed analysis of the SAXS signal as a function of concentration and magnetic field, for the different scattering geometries mentioned above.

[2] D. Constantin, P. Davidson, and C. Chanéac. Lyotropic lamellar phase doped with a nematic phase of magnetic nanorods. *Submitted.*