	Experiment Title: The Hexagonal Columnar Liquid Crystal Phase of Gibbsite in Confinement	**Experiment number:** 26-02-530
Beamline: BM26	**Date(s) of experiment:** From: 27.11.2010 Till: 29.11.2010	**Date of report:** December 2010
Shifts: 9	**Local contact(s);** Dr. Guiseppe Portale	

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Report: (max. 2 pages)

We would like to stress out that only 9 shifts of beam time were granted in stead of the 12 shifts that we asked for. Therefore we were limited in time, but we did our best to make use of the time available.

Our aim was to obtain detailed information about the structure of hexagonal columnar liquid crystals of large gibbsite platelets in confinement. Recent research has shown that large gibbsite platelets with diameter D≈500nm form distorted columnar crystals resulting in vertically elongated SAXS-patterns due to decoupling of the particle normal **n** and the column axis **N** [1]. The effect of decoupling was more pronounced near the capillary bottom.

We expect to see that confinement suppresses distortions of columnar crystals. Also, we are interested to study how confinement influences the formation of (hexagonal) columnar structures in general.

Wedge shaped cells have been developed as shown in figure 1. The wedge had an angle of either 0.05º or 0.32º. The cells were filled with highly viscous suspensions of 420nm sized gibbsite platelets in tetralin and stored either horizontally (wedge left) or vertically (wedge at the bottom). We did a careful examination of cells by the method of µXRD and data analysis is in progress. We show preliminary results obtained in one vertically and one horizontally stored cell.

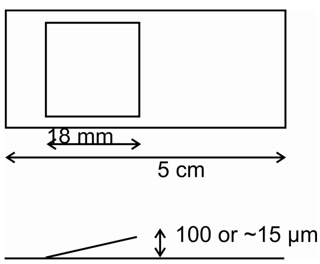

Figure 1. Designs of the wedge shaped cells. Two coverslips (18x18mm and 50x24mm) were placed on top of each other. The slides were touching on one side (the wedge), on the other side the slides were separated using a spacer of either 100 µm or 15 µm correspondingly generating wedge angles of 0.32 º and 0.05º.

Figure 2 (vertically stored cell) represents three patterns measured at different distances from the wedge, a) is highest in the sample; c) is lowest and closest to the wedge. All patterns possess a broad first order and weak second order peaks originating from positional order in the sample, which indicates either the columnar phase (C) or a columnar nematic phase (N_{col}). Rotational scans gave clues about phases, which can be identified as columnar a) and columnar nematic b), c) [2]. Also the columnar phase in a) is oriented in the vertical direction. The results are unexpected in the sense that gravity produces a density gradient in the cell where highest density is expected closest to the bottom. The $N_{(col)}$ phase is expected to be formed at lower density than the C phase. A possible explanation is the following. Due to the large size, particles get 'jammed' in the wedge while in the top part of a sample the particles do have more space for rearrangement and a C phase is formed on top of a N_{col}

phase. Vertical elongation of the pattern in a) is not present in b) and c), indicating that the wedge indeed might have suppressed the decoupling of **n** from **N**. It can also be noticed that intensity decreases with decreasing sample thickness upon approaching the tip of the wedge.

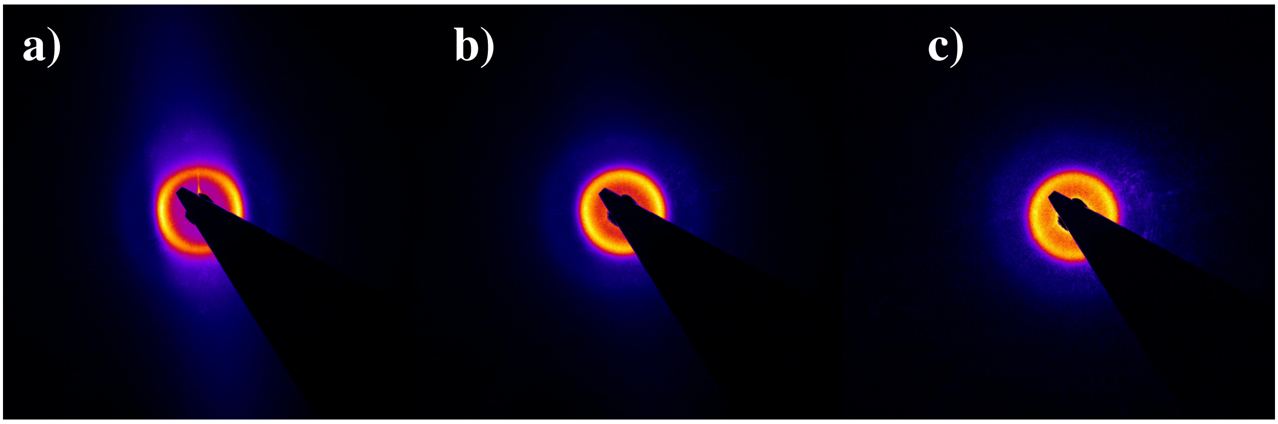

Figure 2. 2D scattering patterns of a highly viscous dispersion of sterically stabilized 420nm sized gibbsite platelets in a wedge cell (0.05°) which was stored vertically, i.e. with the wedge at the bottom taken at a distance of a) 12mm b) 2.0mm and c) 0.5mm from the wedge. The horizontal position remained constant.

Figure 3 shows patterns measured at different distances from the wedge, but in the horizontal direction. All phases were determined as being N_{col}. It was observed that the peaks resulting from side-to-side correlations of platelets get sharper and more intense closer to the tip of the wedge (a). This indicates that confinement promotes better ordering. In the vertically stored cell the opposite was observed - peaks broadened and the intensity of peaks decreased closer to the wedge. The difference is probably due to the storage method. With the wedge down, the particles do not only sediment due to gravity, but also due to wedge effects, giving the particles less time to equilibrate.

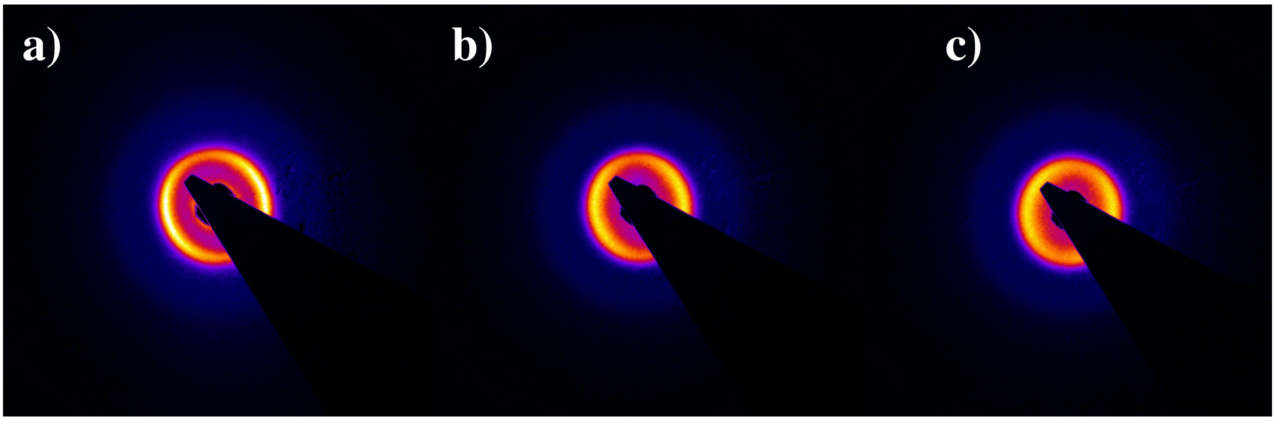

Figure 3. 2D scattering patterns of a highly viscous dispersion of sterically stabilized gibbsite platelets (D~420nm) in a wedge cell (0.05°), stored horizontally. The wedge is on the left site and pictures were taken at a distance of a) 0.5mm, b) 3.0mm and c) 6.5mm from the wedge. The vertical position remained constant.

These preliminary results demonstrate that the wedge definitely influences the formation of liquid crystalline ordering in suspensions of platelets. Now we are working on quantitative analysis of all data to understand exactly the influence of the wedge and the angle of the wedge.

[1] M.C.D. Mourad *et al*, Langmuir **26**, 14182 (2010).
[2] A.B.G.M. Leferink op Reinink *et al*, J. Phys.: Condens. Matter, accepted (2011).