ESRF	**Experiment title:** Investigation of the hexagonal columnar ordering in silica casted gibbsite platelets by microradian small angle x-ray diffraction	**Experiment number**: 26-02-353
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Microradian x-ray diffraction (μradXRD) is a very powerful technique for structure characterization of colloidal self-assembled crystals and liquid crystals [1]. In particular, the μradXRD setup at DUBBLE was extensively used to study suspensions of sterically-stabilised gibbsite platelets in apolar solvents [2]. On the other hand, colloidal platelets suspended in water play an important role in many applications. The aqueous suspensions are more complex since gibbsite particles are charged and their Coulomb interactions depend on the ionic strength of the solvent.

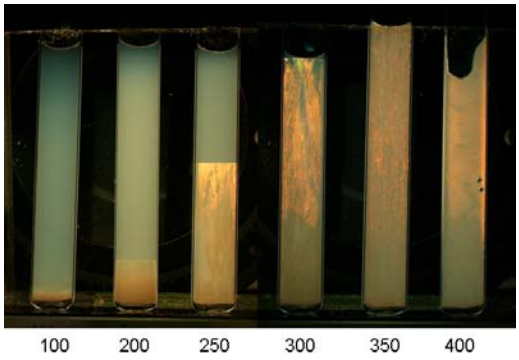

Figure 1: Example of the rich phase behaviour shown by gibbsite platelets in water and 10^{-4}M NaCl at particle concentrations 100 up to 400 g/L, which are displayed under the photos. Samples are separated into isotropic-nematic (100, 200, 250 g/L), (isotropic)-nematic-columnar(300, 350 g/L). At concentration of 400 g/l the suspension forms a nematic gel.

Recently, we have intensively studied the liquid crystalline phase transitions of suspensions of colloidal gibbsite platelets in aqueous solutions [3-5]. One of the important issues involved is the competition between self-organisation and gelation, which sensitively depends on the aspect ratio of the colloidal platelets as well as on the ionic-strength-dependent interaction potential. An example of rich phase behaviour in phase-separating aqueous gibbsite solutions as a function of particle concentration is illustrated in Figure 1. Previously, polarization microscopy and Bragg diffraction of visible light were used to discern the different phases within the samples. However, these techniques do not allow study the differences within similar phases at different ionic strengths and particle concentrations in detail.
In this experiment microradian X-ray diffraction was done in order to extend earlier optical characterizations, to quantify the typical distances within these crystals and discern similar phases at different ionic strengths. The μradXRD setup was built. The compound refractive lens was installed before the sample on a four-motor goniometer to allow for alignment of the position and the orientation of the lens. A similar goniometer was constructed for the sample. Most data are collected with the Photonic Science CCD detector (22 micron pixel size), which was installed in front of the gas-filled detector on the vertical and horizontal translation stages to position the detector and to allow the data collection with the gas-filled SAXS detector (by moving the CCD off). The main sources of vibrations in the optics hutch (cryostat and turbo vacuum pump) were switched off. The beam intensity was extremely stable during the whole experiment and its position showed very little fluctuations.

In the first series of measurements all liquid crystalline phases were examined in the range of 10^{-1}M-10^{-4}M NaCl in water at particle concentrations in between 50 and 500g/L. In Figure 2 typical diffraction patterns are shown for isotropic, nematic and columnar phases as measured in this experiment. The development of the fluid-like particle ordering in nematic and columnar as well as the intercolumnar periodic structure in the columnar phase can be clearly observed.

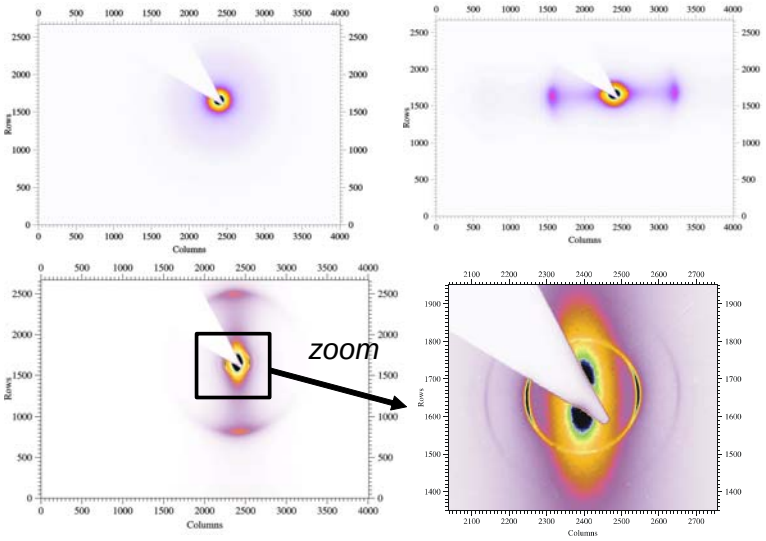

Figure 2: Small angle X-ray diffraction patterns for isotropic (left top), nematic (right top) and columnar (bottom) samples at 10^{-4}M NaCl. The bottom right pattern displays the magnified view of clear intercolumnar reflections, which are not present in the nematic phase.

Moreover, sol-gel transitions are present in aqueous gibbsite suspensions. The second series of measurements was aimed at the understanding of structure and origin of gibbsite gels and to locate them in the phase diagram. By visible observations gelation was observed at relatively low concentrations and both highest (10^{-1}M NaCl) and lowest (10^{-4}M NaCl) ionic strengths. Detailed analysis of the data is in progress.

Silica gibbsite columnar composite materials

Experiments were done in order to cast gibbsite columnar cases in a silica matrix. After having achieved columnar phases of silica coated gibbsite in water phase separation experiments were performed in Stöber mixture and fixated by initiation of the condensation reaction. Moreover composite samples obtained by controlled drying in silicon oil [6] were prepared but showed no columnar ordering. We believe that the problem is related to the sample preparation, which has destroyed columnar structure. Further efforts have to be invested in fabrication of better columnar composite materials.

Goethite nanorods

In addition to the main experiment, we have performed preliminary measurements on suspensions of goethite [α-FeOOH] nanorods with particle dimensions of about 250nm x 60nm x 20nm. Most samples were phase separated in an isotropic and a nematic phase, some were only isotropic. These test measurements were of importance for the the study of these samples in an external magnetic field, which will take place 15-20 November at DUBBLE.

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