

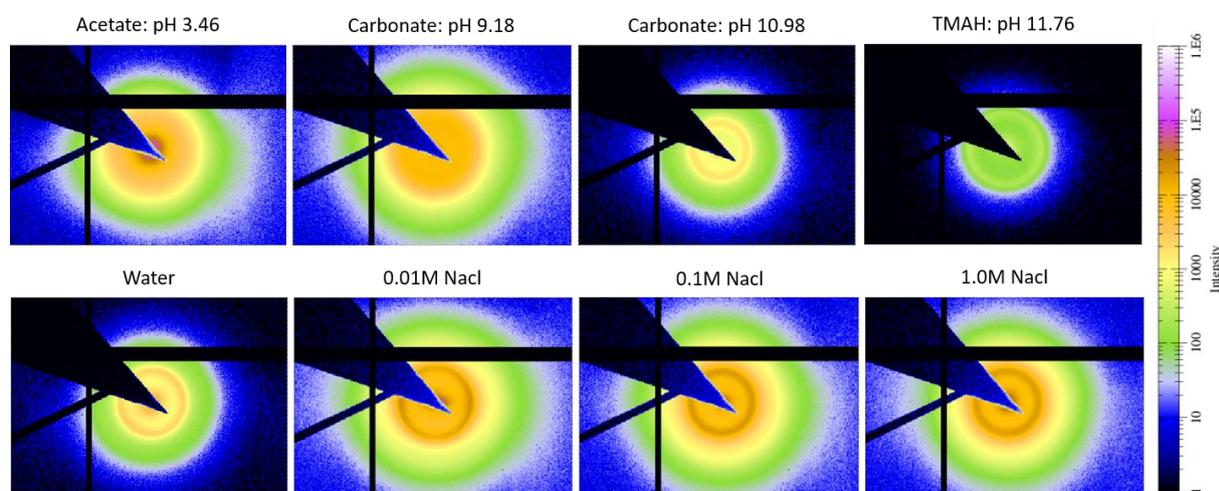
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|   | <b>Experiment Title:</b><br>Self-assembly of Colloidal Ellipsoids     | <b>Experiment number:</b><br>26-02 821 |
| <b>Beamline:</b><br>BM26B  | <b>Date(s) of experiment:</b><br>From: 06.04.2017<br>Till: 10.04.2017 | <b>Date of report:</b><br>May 2017     |
| <b>Shifts:</b><br>12   | <b>Local contact(s);</b><br>Dr. Daniel Hermida Merino                 |  |
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### Report: (max. 2 pages)

In the performed experiment we have studied the self-assembled structures formed in the sediments of magnetic ellipsoidal colloidal particles ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) [1] coated with a silica layer in an applied external magnetic field with microradian x-ray diffraction ( $\mu$ rad XRD) [2]. We have studied the self-assembly behaviour of colloidal ellipsoids at different solvent conditions.

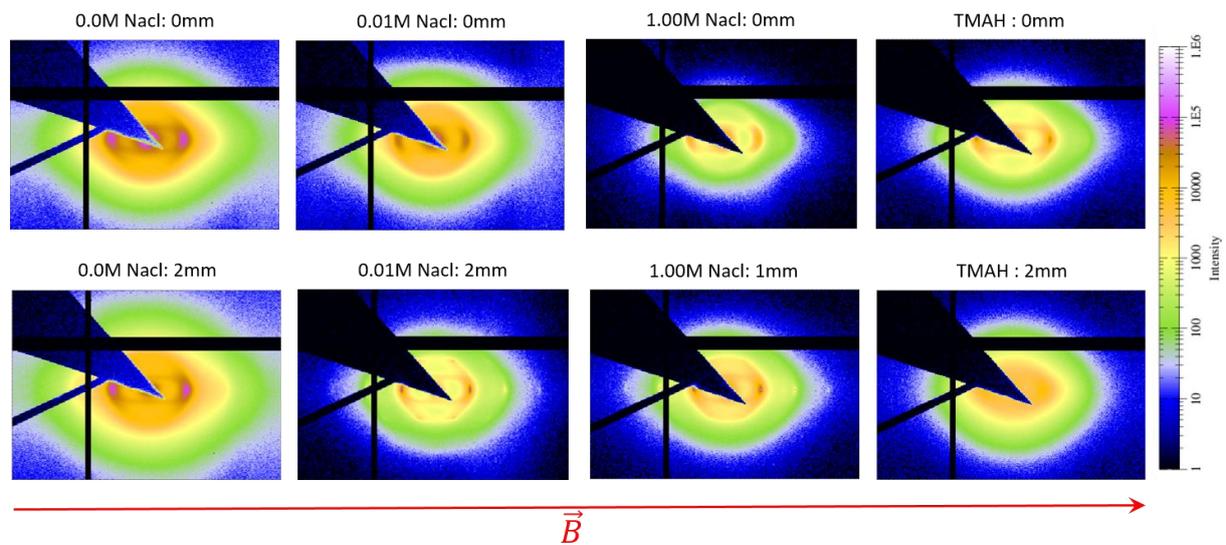
It is quite challenging to build-up the SAXS set up for this kind of experiments. Fine tuning of the CRLs and other optics has finally resulted in a high quality focusing of the beam at the detector. We were pleased with the granted 12 shifts, which allowed us to do most of the planned measurements.

In absence of the magnetic field, we found an isotropic structure factor ring for samples having pH>10 indicating the stability of the suspensions while at lower pH, we could not see any structural peak (fig. 1). However, for all salt concentrations studied (0-5M), we always found an isotropic ring in absence of the magnetic field.



**Figure 1.** 2D SAXS patterns of ellipsoids at different solvent conditions at the bottom of the sediments.

Detailed studies of the ellipsoid sediments in presence of the magnetic field revealed that at different solvent conditions, different structures were formed in the sediments. Height scans of the sediments were performed which showed that the structures are effected by an increased osmotic pressure in the sediments. Figure 2 shows the diffraction patterns of the self-assembled structures at four different solvent conditions at different heights in the sediments (0 represents the lower edge of the capillary) at a magnetic field of 550mT. For pure water, one could find only a nematic phase. However, at higher NaCl salt concentrations a higher ordered crystalline phase is found as seen by the increase in sharpness of the peaks and the increase of higher order peaks in the pattern. For a different salt tetramethyl ammonium hydroxide (TMAH), that is known to increase the surface charge of the silica particles, again we could only find a nematic phase.



**Figure 2.** 2-D  $\mu$ rad XRD patterns of height scan through sample of magnetic colloidal ellipsoids exposed to magnetic field of 550mT, at four different solvent conditions.

Rotation scans (with a total range of 70 degrees) were performed for samples at a particular height that showed hexagonal symmetry as well as nematic phase. With extensive and detailed analysis of the obtained structure we might be able to determine the exact ordering in the self-assembled phase.

To summarize, we highlight that the  $\mu$ rad XRD experiment revealed the presence of ordering in the ellipsoid sediments with different structures depending on the solvent conditions.

Finally, we would like to thank D. Hermida Merino and all other beamline staff for their excellent support.

- [1] M. Ocana *et al.*, J. Colloid Interface Sci., 212(2), 317, 1999.
- [2] J.H.J. Thijssen, et al., Advanced Materials 18, 1662 (2006).