DUTCH-BELGIAN BEAMLINE
AT ESRF

EUROPEAN
SYNCHROTRON
RADIATION FACILITY

Experiment Report Form

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

(next page)

DUBBLE	**Experiment title: Rheology of model clays: SAXS with microradian resolution and 2D WAXS**	**Experiment number**: **26-02-283**
Beamline: DUBBLE BM-26B	**Date(s) of experiment**: From: 25-10-2005 To: 01-11-2005	**Date of report**: - 02 -2006
Shifts: 15	**Local contact(s)**: Dr. Florian Meneau	
Names and affiliations of applicants (* indicates experimentalists): Andrei Petukhov*, Matti van Schooneveld*, Annemieke ten Brinke*, Henk Lekkerkerker, Utrecht; Hans Hoekstra*, Frank Snijkers*, Jan Vermant, Leuven		

Report: (max. 2 pages)

The rheological properties of suspensions of anisometric particles with large aspect ratio can be significantly enhanced by the use of colloidal particles of different size and shape. The flow-induced changes in the interparticle structure play a key role in this enhancement and are studied using SAXS during flow. Model systems containing lath-like hectorite clay particles and rod-like boehmite particles and mixtures thereof are studied. Both hectorite and boehmite form strong gels when dispersed in water at relatively low concentrations, while they also display a strong shear-thinning effect. An alternative manner to tailor the rheological properties is to suspend particles in a suspending medium with non-Newtonian properties. To this end, silica particles were dispersed in aqueous polymer solutions subjected to shear flows and studied using SAXS.

To improve the resolution of the SAXS setup, we have installed the Photonic Science CCD camera (x-ray imager - VHR) as an additional SAXS detector with higher spatial resolution, which was placed in front of the gas-filled detector, immediately after the evacuated fly-pass tube. The x-ray beam was carefully focused on the screen of the CCD detector. The detector effectively had 2004×1335 pixels (v×h) since 2×2 binning was used. The CCD camera was placed on computer-controlled translation stages so that the CCD detector could be brought in and out remotely, to allow the use of one detector or the other. In addition, the Photonic Science CCD detector (Photonic Science Xios II, 1270×1160 pixels) was placed close to the shear cell in order to be able to record two-dimensional WAXS patterns. Due to a too low level of the detected WAXS intensity we were not able to extract reliable information on the particle orientation in the suspension. To minimise the effects of the beam absorption in the shear cell (which can be especially significant in measurements in the tangential direction) and to further enlarge the highest q-values, we used relatively high photon energy of 15 keV ($\lambda = 0.0825$ nm).

Preliminary analysis of selected examples of the collected data, which has been performed during the experiment, has revealed a problem. The decay of the SAXS intensity observed with one detector did not match the result of the other detector so that we were not able to smoothly join the curves by adjusting only the relative scale in both data. In order to resolve the problem an additional test with both SAXS detectors was performed by recording the scattering pattern from a dilute suspension of colloidal silica spheres in cyclohexane. This colloidal system was used earlier in many of our SAXS experiments at DUBBLE in the past so that we very well knew what to expect. The results of this test measurement are presented in Fig.1. Panel (a) summarizes the data obtained with the CCD camera. Three different exposure times were used. The shortest exposure was used to reliably measure the highest SAXS intensities close to the beamstop. Longer exposure time allows us to reduce the noise of the much weaker SAXS intensity at larger scattering angles. Panel (b) displays examples of the results obtained with the gas-filled detector. The difference in the spatial resolution of two detectors is obvious: the minima of the form factor of the spherical particles are not well resolved in panel (b). Moreover, the overall decay of $I(q)$ at large q does not follow the expected Porod-law decay: $I(q) \propto 1/q^4$. At the same time, the result of the CCD detector does closely follow the Porod law. It was therefore concluded that the data collected with the gas-filled detector were not reliable.

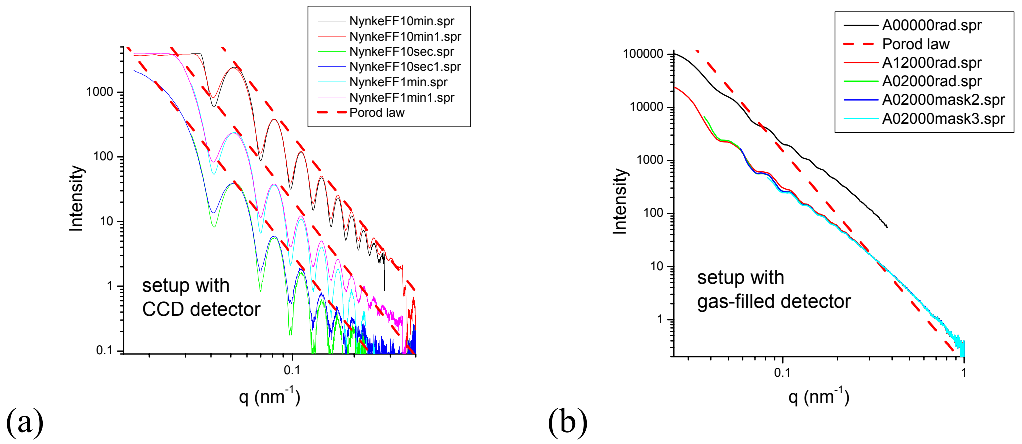

Figure 1. Radial profiles of the SAXS intensity measured in a dilute suspension of colloidal silica spheres (diameter 230 nm) with the CCD detector (a) and the gas-filled detector (b). Exposure time of 10 min, 1 min and 10 s was used for the CCD detector to overcome the problem of detector saturation at about 4000 counts. In panel (b) the exposure time was 10 min. To make sure that the problem is not related with an overload of the detector, the data was re-measured with smaller opening of the primary slits (0.2×0.2 mm^2 in files A02 and A12 instead of 0.4×0.4 mm^2 in file A00). We have also used different masks, which did not affect the result in (b) but did affect the minima in (a).

As examples of measured data, figure 2 shows the SAXS pattern for a boehmite suspension of 2.5 wt.% in water. Panels (a) and (c) are measured at rest, in the radial (flow direction) and tangential (gradient) direction respectively, while panels (b) and (d) show the effect of a shear of 100 rpm. Panels (a) and (c) show an (almost) isotropic pattern, which indicates random orientation of the boehmite rods, whereas in (b) and (d) an anisotropic pattern can be seen, a sign of alignment of the rods. The effect of shear rate and mechanical history was carefully measured.

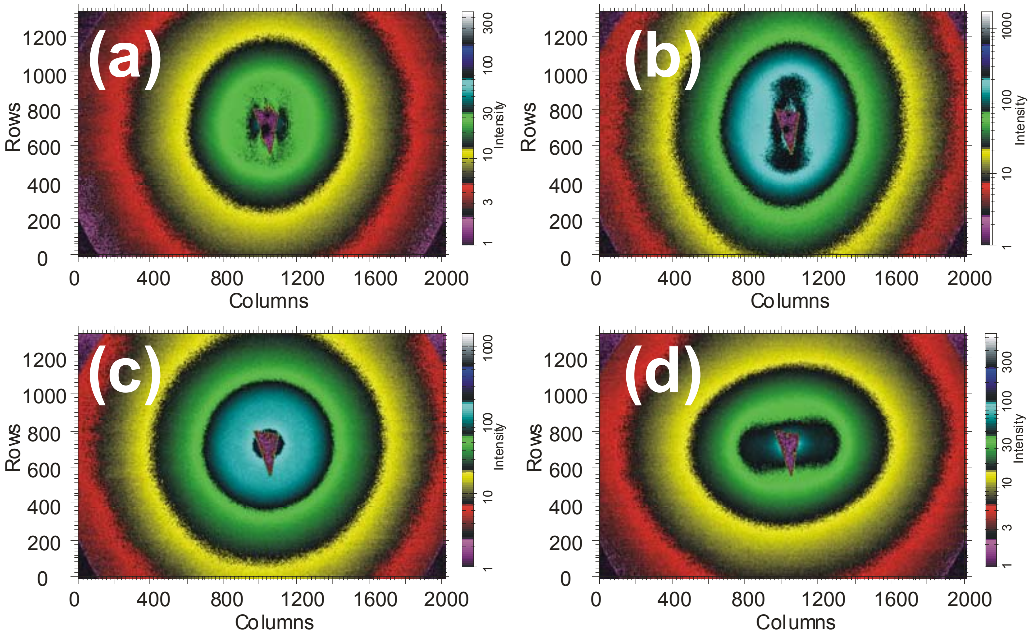

Figure 2. Scattering patterns for a 2.5 wt.% boehmite suspension at rest, (a and c) and under a shear of 100 rpm, (b and d) both in radial, (a and b) and tangential (c and d) direction.

Furthermore, suspensions of hectorite, gibbsite and hydrotalcite were investigated, as well as mixtures of hectorite with gibbsite, boehmite or hydrotalcite. Measurements on the suspension of silica in the viscoelastic polymer solution revealed the formation of strings of particles, aligned in the flow direction. At sufficiently high shear rates and volume fractions The structural analysis is being carried out in more detail using an expansion of the relative structure factor in spherical harmonics to quantify the flow-induced anisotropy.