EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

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.

ESRF	**Experiment title:** Cellulose nanofibrils with surfactants as aqueous rheology modifiers	**Experiment number**: SC-2882
Beamline: ID02	**Date of experiment**: from: 14-06-2010 to: 18-06-2010	**Date of report**:
Shifts: 9	**Local contact(s)**: Dr. Jeremie Gummel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

In personal care products, there are several polymers and surfactants that are only used to give the product its desired structure. Because fossil fuels, used to make these materials, are becoming scarce, it has been suggested that instead oxidised cellulose could be used as a rheology modifier in these products. It has been found that C6-partially oxidised cellulose forms gels with salt and surfactants. In this project it is our task to find out why these gels form.

In this experiment we studied the structure of cellulose-salt and cellulose-surfactant gels. We had three days to perform this experiment, so the first day we performed static X-ray scattering experiments, on approximately 350 samples. These samples contained 2 component mixtures of cellulose (4 or 8 g L^{-1}) and salts (LiCl, NaCl, KCl, CsCl and NH_4Cl), cellulose and clays or cellulose and the surfactant sodium lauryl ether sulfate (SLES). We studied the SLES samples at two different temperatures, 25 and 50°C. The scattering of static 3 and 4 components gels (cellulose-clay-salt and cellulose-clay-salt-surfactant) were also measured during this experiment.

The second part of the experiment we used to follow the X-ray scattering of the gels under shear. For these experiments, we prepared 2 component gels with cellulose and salt or cellulose and surfactant. We used the whole shear rate range possible on the rheometer. Due to the large amount of data generated in three days, we will focus in this report on scattering from cellulose and salts, since by using different ions we tried to use contrast variation to understand gel formation by cellulose with salt in more detail.

We used five different kind of salts (LiCl, NaCl, KCl, CsCl and NH_4Cl) and studied two different concentrations: 0.2 and 0.3 M. In Figure 1, the scattering curves of 4 g L^{-1} cellulose with these different salts are shown. In this figure, one can clearly see that there is a trend as function of the atomic number of the

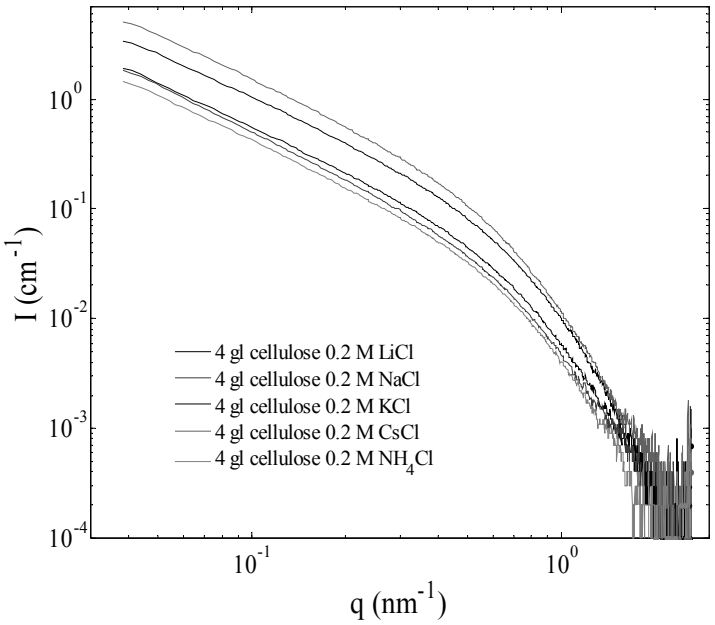

Figure 1: Static scattering curves of 4 g L^{-1} cellulose with different salts

counterions. However, this trend was less prominent for the other cellulose concentrations. This may be explained by the capillaries having different thicknesses so our next step will be to correct for the thickness of the capillaries.

After that we will fit these scattering curves with the form factor of a flexible cylinder with an ellipsoidal core. This form factor has been used to fit the scattering curves of cellulose alone and we expect that it will also fit the scattering curves of the cellulose gels with salt.

In the second part of the experiment we performed RheoSAXS measurements. We already knew that these gels are shear thinning, which can also be seen in Figure 2a, where the rheology data (viscosity as function of the shear rate) are shown. We hoped to get some insight of what is happening to the structure of the gel by measuring the small angle X-ray scattering of the gel under shear.

In figure 2b the scattering curves of as function of the shear rate are presented. The corresponding rheology data are the last 10 data points of figure 2a. In Figure 2b it can be seen that at the higher shear rates the sample shows some shear thickening; the viscosity increases. In figure 2b the scattering curves at the higher shear rates have a slightly higher intensity at low q. These scattering measurements are certainly helping us to understand the properties of the gels and we are confident that they will form the basis of at least one article, but hopefully more.

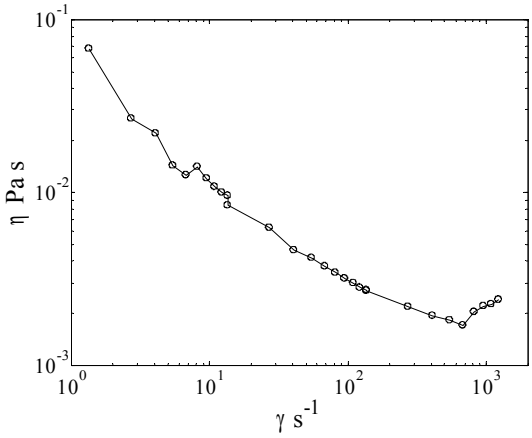

Figure 2a: viscosity as function of the shear rate

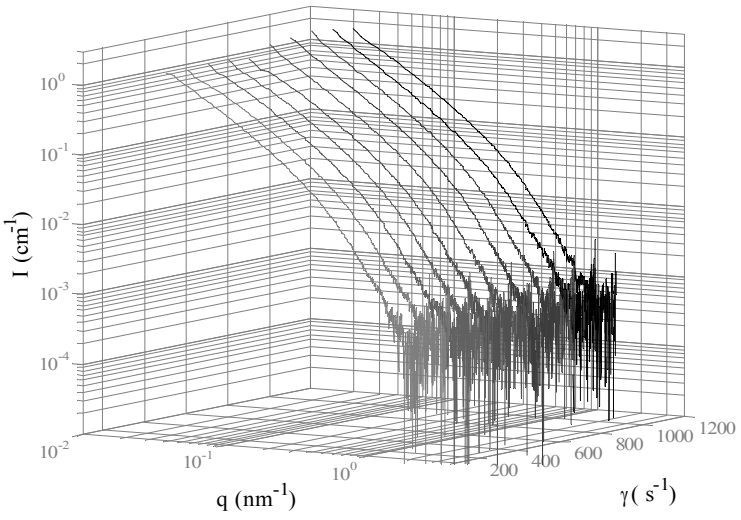

Figure 2b: scattering curves of 4 g L^{-1} dialysed cellulose with 0.2M NaCl as function of the shear rate, X-ray beam in a radial position relative to the Couette cell.