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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:**

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Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
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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: Hydrodynamic Interactions in Charged Colloid Suspensions. X-Ray Photon Correlation Spectroscopy Study**	**Experiment number**: HD-7
Beamline: ID10	**Date of experiment**: from: 13 June 2006, 7:00 to: 20 June 2006, 7:00	**Date of report**:
Shifts:	**Local contact(s)**: Andrei Fluerasu	*Received at ESRF:*

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Report:

Monodisperse silica particles are prepared in two steps. First core (or seed) particles are synthesized in a microemulsion [1]. This dispersion is then grown to the final size by seeded growth [2].
The radius of the silica spheres was measured by means of dynamic light scattering and amounted to 92.5 nm. The original colloidal suspension in ethanol/DMF mixture was percipitated by centrifugation and redispersed in pure DMF at a concentration of 500 g/l. After this treatment the suspension has crystallized. From this stock solution samples of total added LiCl salt concentration amounting to 0, 10, 20, 50, 100, 150, 200 μM and 20 mM, respectively, and different colloid concentration from 50 to 280 g/l were prepared by adding a small amount of concentrated LiCl solution. For XPCS experiments the samples were filled into quartz capillaries of a diameter of 1.5 mm and sealed with glue.
The experiment was performed at the Troika III of the Troika beamline ID10C at the European Synchrotron Radiation Facility (ESRF). A schematic view of the setup and experimental details are given elsewhere [3]. The synchrotron was running in a uniform filling mode with an average current of about 200 mA. Measurements were performed at 7.990 keV, λ= 1.55 Å.
The coherent beam is provided by a 25x25 μ^2 pinhole placed at a distance of 25 cm from the sample. With this setup the primary beam intensity was about $2x10^9$ photons/s/200mA. The sample was mounted in an evacuated small-angle scattering chamber and the scattered photons were guided through a vacuum flight path to the detector stage, giving a sample-to-detector distance of 3.467 m. Photons are detected by a Bicron scintillation counter equipped with an adjustable pair of slits of 100x100 μ^2. Correlation functions are calculated in real time via an ALV-5000 digital correlator.

Results
The scattered x-ray intensity, I(q), was measured in a broad q range, Fig.1. The presence of multiple maxima in the I(q) curve indicates a very low polydispersity of the sample used, amuonting to 2%.

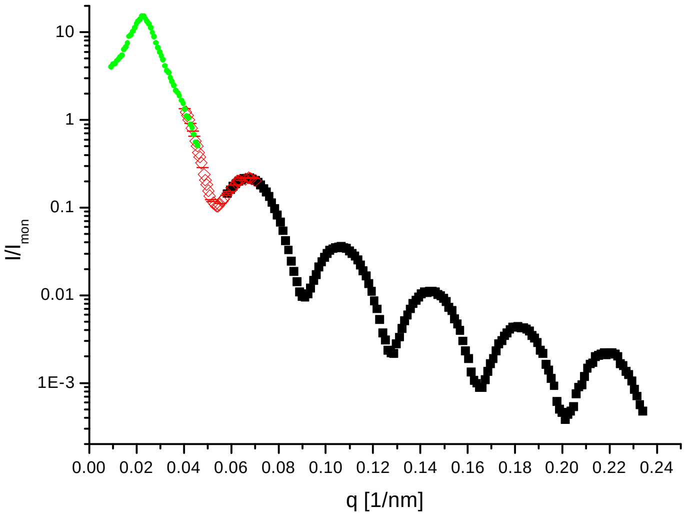

Figure 1. Static curve, I(q), measured for a suspension of silica spheres at a concentration of 150 g/l and added salt concentration of 100 μM LiCl.

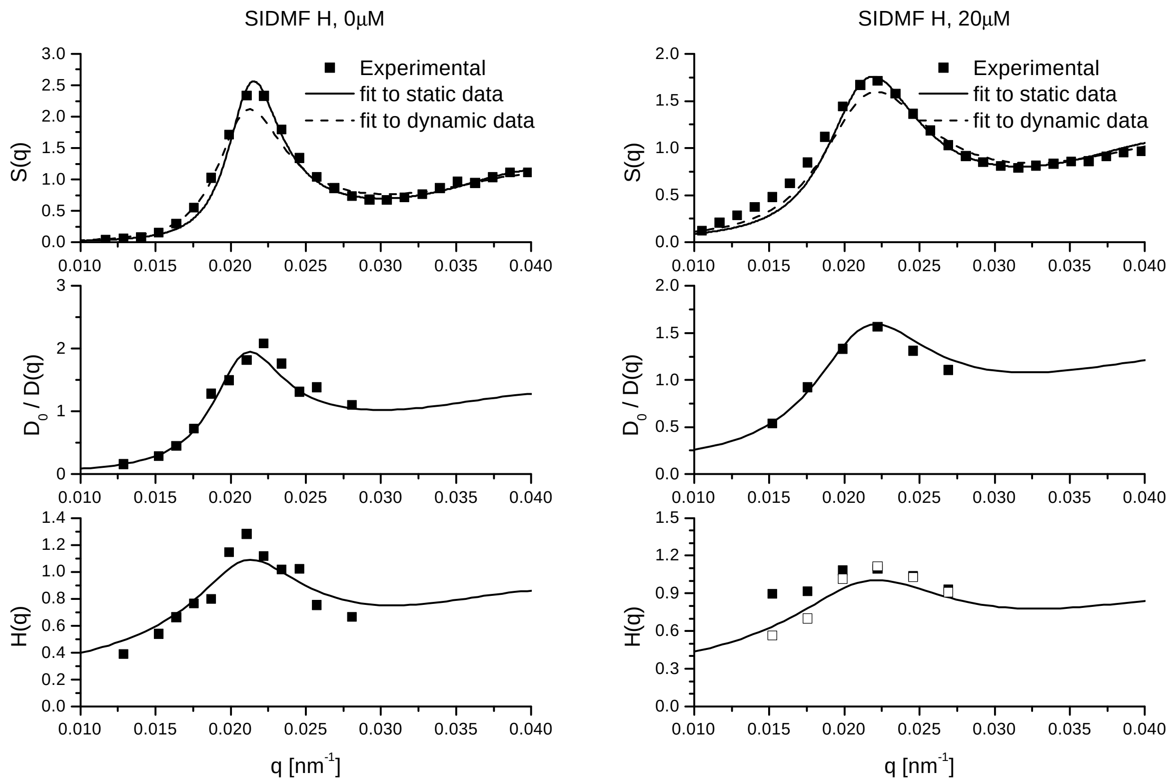

Figure 2. The static structure factor, S(q), normalized diffusion coefficient, $D(q)/D_0$ and hydrodynamic function H(q) measured (symbols) and calculated (solid lines) using theoretical models, for salt free and low salt (0 and 20 μM LiCl) suspensions of silica particles in DMF at a colloid concentration of 150 g/l.

We measured the static structure factor, S(q), normalized diffusion coefficient, $D(q)/D_0$ and hydrodynamic function H(q) over the full salt and colloid concentration range up to the crystallization point. We interpret the data in terms of a theoretical scheme based on DLVO-type continuum model of charged colloidal suspensions, which accounts, in an approximate way, for many-body hydrodynamic interactions, and was successfully used to fit experimental data of apoferritin [4] and charge stabilized fluorinated latex spheres [5]. Even at the salt free and low salt conditions, very close to the crystallization point of the colloidal system, the agreement of the experimental data with the theoretical models is good, Fig.2. This is in contrast to the conclusions of the work

of Riese et al. [6] done on a similar system. In this work screening of hydrodynamic interactions in strongly charged colloid suspensions in water/glycerol mixtures was reported [6]. We have not seen such an effect neither in our previous studies at intermediate salt concentrations [5] nor in this experiment, which covered the entire strength of long range repulsive interactions from almost crystalline samples to the hard sphere limit.
A substantial part of our samples with the strongest repulsive interactions, i.e. at the lowest salt and/or the highest colloid concentrations have crystallized. This fact was corroborated both by visual inspection and 2D I(q) measurements using the CCD camera. The studies of structure, dynamics and phase separation (crystallization) in charge stabilized colloidal suspensions was not the aim of the present experiment and was not possible because the lack of time. Our preliminary data obtained for these systems indicate that such studies are feasible, would be very interesting and should be performed in the near future.

References
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