ESRF	Experiment title: X-ray Photon Correlation Spectroscopy study of Hard-Sphere Colloids	Experiment number: SC-1184
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Report:

During the SC1184 experiment we measured the dynamic properties of hard-sphere colloidal suspensions made of sterically-stabilized polymethylmethacrylate (PMMA) particles in decalin. We performed an X-ray Photon Correlation Spectroscopy experiment in the standard way used at the ID10A beamline. Structural and dynamical properties have been extensively measured at three volume concentrations, Φ=0.02 (dilute case), Φ=0.40 and Φ=0.49. Fig. 1 shows the small-angle scattering (SAXS) intensity from the three solutions when illuminated by a 12 μm coherent beam.

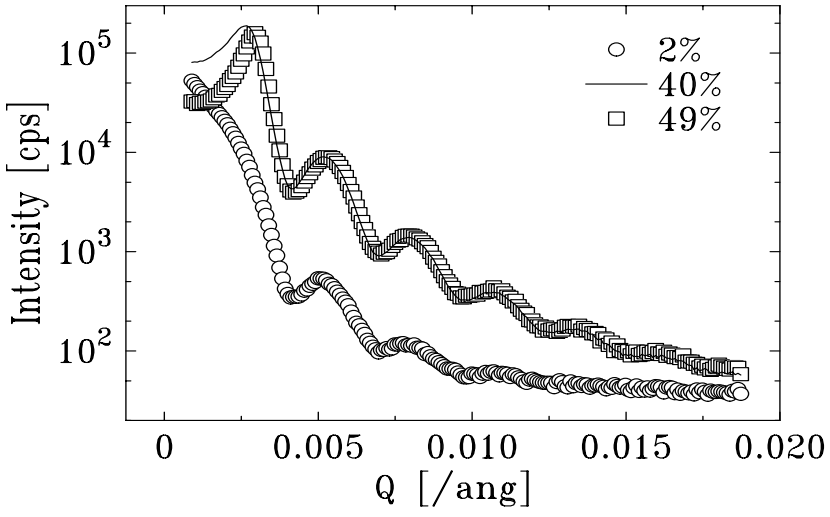

Fig.1 – Monitor-normalized SAXS intensity from the PMMA colloidal suspension at 0.02, 0.40 and 0.49 volume concentration. Raw data.

The SAXS data show the usual intensity oscillations related to the spherical shape of the particles. At the highest concentrations the behavior changes dramatically at low Q due to the direct inter-particle interactions and hence to the presence of a structure factor.
The systems proved to scatter X-rays very efficiently and to be quite resistant to radiation damage during the collection of the dynamic data. To check the stability of system a systematic monitoring of the scattered intensity profile was performed during the acquisition of the dynamic data.

Dynamic properties have been measured well beyond the first structure factor peak. Fig. 2 shows a typical intermediate scattering function at the volume concentration Φ=0.49 (fig. 2a) and the preliminary results as a function of Q*R for the normalized inverse diffusion coefficient $D_o/D(Q)$ for the short-time diffusion at the same volume concentration (fig.2b), D_o being the free particle diffusion coefficient, R the particle radius and D(Q) the diffusion coefficient obtained from the first contribution of a cumulant fit.

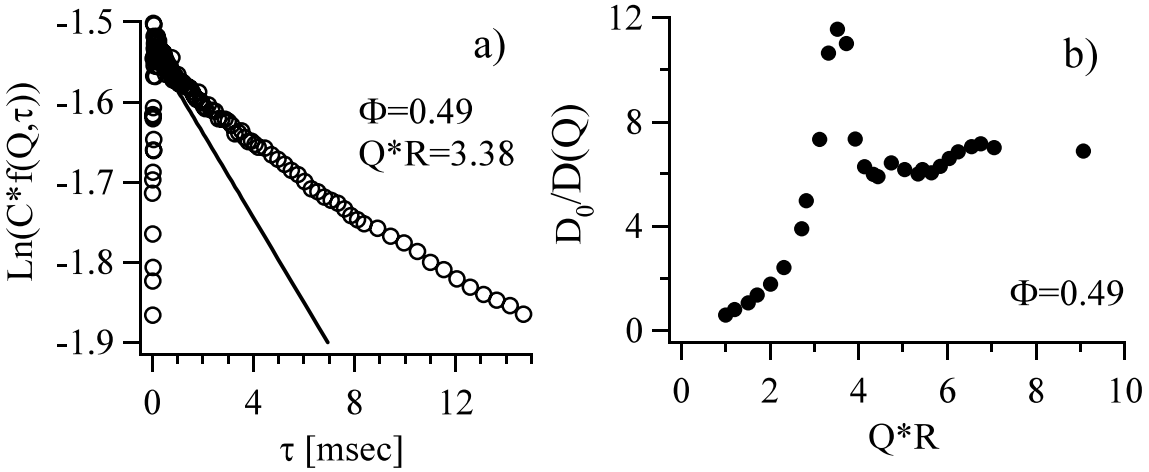

Fig. 2. a) Intermediate scattering function at Q*R=3.38 for a suspension of PMMA particles at Φ=0.49. The solid line shows the first contribution of a cumulant fit. b) Normalized, inverse diffusion coefficient $D_0/D(Q)$ as a function of QR for the same volume concentration.
Preliminary results.

The measurements span a range up to Q*R=9 and cover even the region around Q*R=7, where the second structure factor peak is expected to appear. This plot can be directly compared with Dynamic Light Scattering (DLS) data made by Pusey et al. [1] on nearly the same system at the same concentration. There is an obvious discrepancy in the first peak height where our value is greater by 30%. This contradiction is currently under investigation. However, at Φ=0.49 there are two issues that make the analysis a non-trivial task. Firstly, our correlation data have a remarkably stretched exponential behavior that makes difficult an unambiguous determination of the relaxation rates for the short-time diffusion. Secondly, our correlation data can not be exploited in the time domain below 60 μsec because of the intensity oscillations coming from the ESRF machine frequency. Both factors produce a great uncertainty in the height of $D_o/D(Q)$ and therefore we can not draw definitive conclusions so far. On the other hand, there is a noticeable difference in the size of the particles in the two experiments. The radius is typically 300nm in the case of DLS measurements compared to 114nm for the XPCS ones. Multiple scattering and sedimentation related effects could play a role for the bigger particles.

Of course, we are trying to improve our analysis. However, we think that in the future we should carefully investigate the region just below the freezing concentration (Φ=0.494) to see how the system progressively deviates from the simple exponential behavior. In addition, the ESRF machine can now routinely operate in the uniform filling mode, which offers the possibility to obtain time correlation data having a "monitor" baseline virtually free from artifacts at short times.

References

[1] – P.N. Pusey, P.N. Segrè, OP. Behrend, S.P. Meeker, W.C.K. Poon, Physica A 235(1997)1-8