



	Experiment title: XPCS on thermosensitive core-shell particles	Experiment number: SC 2553
Beamline: ID10A	Date of experiment: from: 24/09/2008 to: 29/09/2008	Date of report: 25/02/2009
Shifts: 18	Local contact(s): A. Fluerasu	<i>Received at ESRF:</i>
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Report:



Fig. 1: Flow capillary filled with suspension (blue): the capillary is constructed as a flow-through capillar. Thus, the same capillary can be used for each experiment. This offers the possibility to subtract the scattering of the empty capillary and the solvent after cleaning of the capillary. Another advantage is the possibility to measure during a constant tube flow. The capillary itself is a mark tube with a wall thickness of 0.01 mm. The temperature of the sample can be adjusted by the capillary holder made of copper and stainless steel tubes, which are connected with a glass capillary in the measurement window, using a Peltier-heating.

We report on the study of the dynamics of concentrated suspensions of thermosensitive PS-PNIPAA core-shell particles. Since the particles are known as weak scatterers, special care was taken to the accurate background subtraction. Thus a new flow-capillary holder was constructed for this experiment (see Fig. 1).

During the experiment suspensions of the thermosensitive core-shell particles are measured at different concentrations ranging from 8.7-18.8 wt%. It is known that some samples may undergo degradation upon longer exposure times or multiple exposure with small time increments. Therefore the stability of the samples was checked by measuring the suspensions with increments of different exposure times (0.01s-1s). Unfortunately, the static scattering intensities $I(q)$ differ in absolute scale and positions of the extrema (cf. fig. 2a). A pronounced decrease in the structure factor peak at $q \approx 0.05 \text{ nm}^{-1}$ was obtained. After discussion with our local contact, Dr. Fluerasu, we focused on the sample with the highest amount of crosslinker (10mol%, KS16). To prevent degradation effects the exposure time was set to the minimum of 0.01s per picture (for the two-dimensional detector "Princeton"). For about 10 frames no change in the scattering pattern of the sample was obtained. The corresponding correlation functions are shown in fig. 2d.

To underline the results scattering data were also taken using a constant flow (0.1 $\mu\text{l/h}$ to 30.0 $\mu\text{l/h}$). A flow rate of 2 $\mu\text{l/h}$ was enough to obtain always the same static small-angle x-ray pattern. It turned out, that the absolute scale of the maximum of the structure factor was much higher than in the case of measurements without flow ($\approx 80 \text{ cm}^{-1}$ instead of $\approx 50 \text{ cm}^{-1}$ at $q \approx 0.05 \text{ nm}^{-1}$, see fig. 2b). Thus, some effects due to beam damage or local heating of the sample can not be excluded

when measuring without flow. No dynamics were found by measuring the suspensions with flow (fig. 2d).

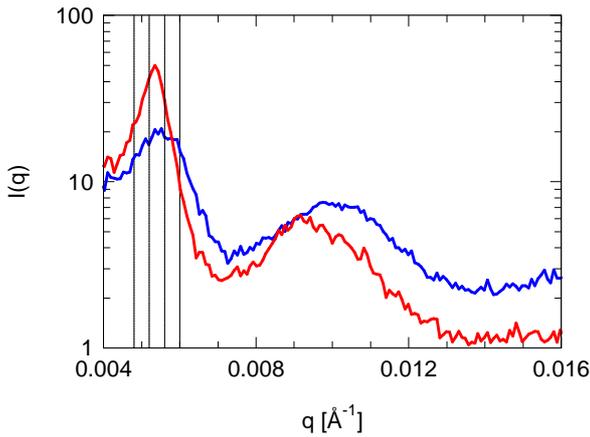


Fig. 2a: Statics without any flow of one of the first 10 pictures (red line) and one of the last 10 pictures (blue line) for a 18.8 wt% suspension of latex at 12°C. q -values used for the dynamics (cf. fig. 2c) are marked with vertical black lines. Exposure time: 0.01 s.

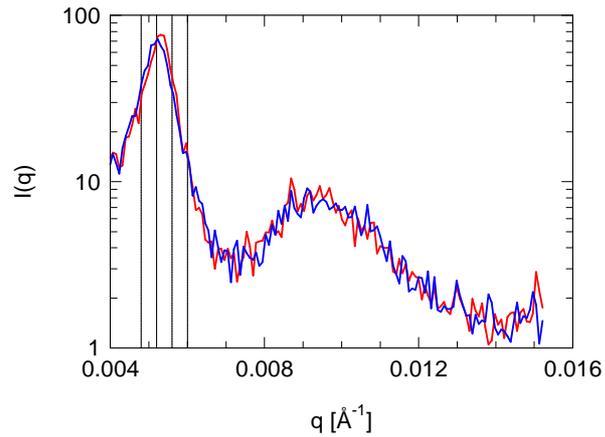


Fig. 2b: Static at a flowrate of $2\mu\text{l}$ per hour of one of the first 10 pictures (red line) and one of the last 10 pictures (blue line) for a 18.8 wt% suspension of latex at 12°C. q -values used for the dynamics (cf. fig. 2d) are marked with vertical black lines. Exposure time: 0.01 s.

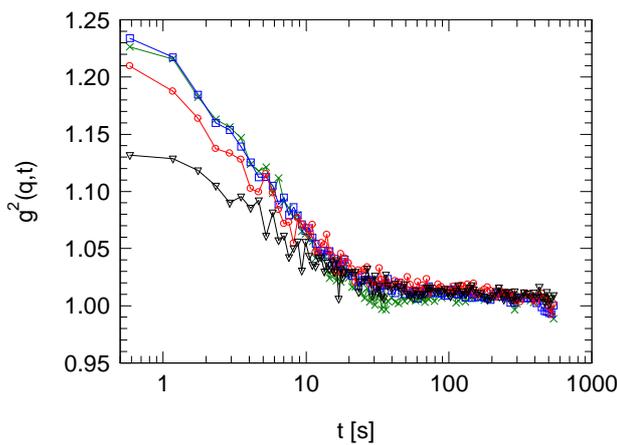


Fig. 2c: Correlation functions, determined using the q -values marked in fig 2a. q -values : 0.0048\AA^{-1} (black), 0.0052\AA^{-1} (red), 0.0056\AA^{-1} (blue), 0.0060\AA^{-1} (green). For the determination of the correlation functions all measured intensities/runs were used.

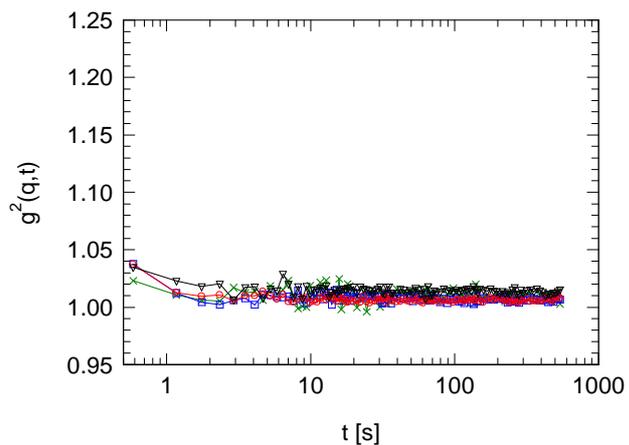


Fig. 2d: Correlation functions of q -values marked in fig 2a. q -values : 0.0048\AA^{-1} (black), 0.0052\AA^{-1} (red), 0.0056\AA^{-1} (blue), 0.0060\AA^{-1} (green). For the determination of the correlation functions all measured intensities/runs were used.