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# APPLICATION OF X-RAY SCATTERING AND DIFFRACTION TECHNIQUES TO STUDIES OF HIGHLY CHARGED COLLOID SUSPENSIONS IN THE VICINITY OF THE CRYSTALLIZATION POINT

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Colloidal suspensions of monodisperse silica particles (diameter 164 nm) in dimethylformamide (DMF) have been studied by means of small angle X-ray scattering (SAXS) and X-ray photon correlation spectroscopy (XPCS) near and above the crystallization conditions. The SAXS measurements performed at low volume fractions $\phi$ and relatively high added salt (LiCl) concentrations $C_s$ revealed multiple (above twenty) minima in the form factor $P(q)$, confirming low polydispersity of the colloidal particles (below 2%).

This and similar colloidal systems have been studied by means of SAXS and XPCS in the liquid phase [1-3].

Static properties of the colloidal suspension (its structure) is usually described in terms of the structure factor $S(q)$ which can be relatively easily extracted from the SAXS results and calculated theoretically using appropriate models. Fitting the models to the experimental data allowed estimation of the particle effective charge value $Z_{eff}$, the only free parameter in the model. The colloid and added salt concentrations were adjusted such that the freezing criteria (Hansen-Verlet) could be met for relatively dilute samples ($\phi$ < 0.1) and the samples often crystallized. In such cases the scattering spectrum turned into diffraction pattern which could be best seen using the CCD detector (Pilatus).

The diffraction patterns from crystalline samples sometimes resembled those characteristic for single monocrystals, suggesting large dimensions of ordered regions even for those samples which looked like a system of coexisting liquid and crystal (Fig. 1).

The XPCS measurements performed close to the crystallization conditions revealed a mild transition from short-time to long-time diffusive behaviour with the ratio of the respective diffusion coefficient taking values between 2 and 3.

The most interesting finding in our study was the effect of colloidal crystal melting in the X-ray beam. We discovered that in some samples the diffraction pattern slowly changed into the liquid-like scattering profile. Moreover, after the beam was blocked, the diffraction patter was recreated in an identical shape (Fig. 2).

At the end of the presentation an attempt to explain the observed effects will be made.

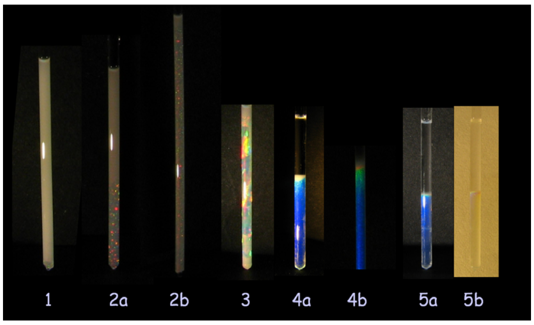


Figure 1. Colloidal samples in capillaries. For these pictures the samples were illuminated with white light directed from the left except the last picture, where only ambient light was present. 1 – liquid phase, 2 – liquid-crystal coexistence, 3 – polycrystal (or glass-crystal coex.), 4 – variable monocrystal, 5 – variable monocrystal in ambient light conditions.

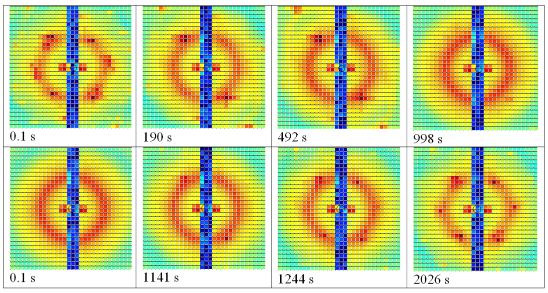


Figure 2. Structure melting (upper row) and recovery (lower row) observed using Pilatus detector.

**References**

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