ESRF	**Experiment title:** Aging dynamics in a charged colloidal system.	**Experiment number**: HD69
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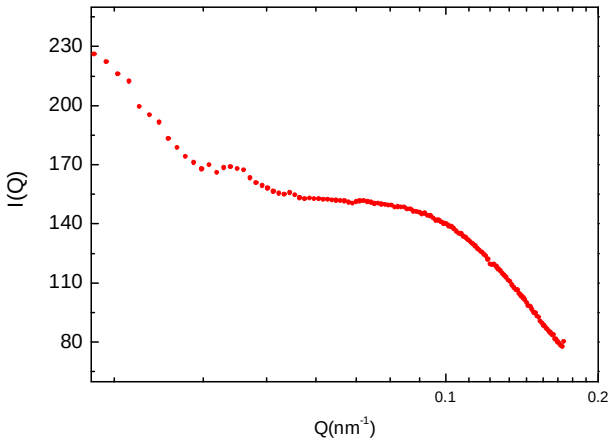
Report:

Light (PCS) and X-rays (XPCS) photoncorrelation measurements have been performed on a solution of Laponite clay in ultrapure deionized water at a fixed concentration of C_w=3.0% in weight. This system performs aging, i.e. sample properties as measured by correlation or response function change continuously with time, as the sample evolves very slowly towards equilibrium. The intriguing aging phenomenon of this system has been widely investigated varying clay concentration and ionic strength of the solution with standard PCS technique [1, 2]. The measurements have shown that the system can be found in an arrested state also at very low packing fractions. These phases are structurally arrested and the system results non ergodic on the typical experimental time window of light photoncorrelation spectroscopy. However it is interesting to explore the aging dynamics also beyond the time scale of the seconds in order to probe further the time evolution of the dynamics. Recently several measurements have shown the existence of a crossover, depending on the Q value, from an aging to a full aging behaviour, characterized by a compressed shape ($\beta>1$) of the autocorrelation curves [3].
The aim of this experiment was then to investigate the controversial aging dynamics shown by Laponite-water system on a really wide time window ranging from milliseconds up to minutes in a Q region close to the maximum of the S(Q) of Laponite. To follow the dynamics in a so large time window we have used PCS for the dynamics on shorter time scales (from millisecond up to seconds) and XPCS for the slower dynamics (from seconds up to minutes). In this context the first point to be verified was the presence of a XPCS signal due to Laponite samples.

Several samples have been prepared under nitrogen atmosphere in order to avoid contact with air and stored in quartz capillaries of 2 mm of diameter. Light scattering measurements have been performed in PCS laboratory 03.0.03 in front of ID2 beamline on a standard dynamic light scattering setup (λ=532 nm) with a scattering angle of 90°, therefore with a fixed scattering vector Q=0.022 nm^{-1}. The incident beam of XPCS setup was fixed at the energy of 13 KeV. X-rays photoncorrelation measurements have been performed trough a CCD detector in a Q range varying from 0.017994 nm^{-1} to 0.174718 nm^{-1}. Using this same experimental setup we can also probe the static scattered intensity I(Q) of the solution in this Q range as shown in Fig. 1. These measurements are in agreement with previous SAXS measurements performed on samples at several Laponite concentrations on the ID2 beamline .

Fig.1 Wave vector behaviour of the scattered X-rays intensity for sample at C_w=3.0%. This measurement results in agreement with previous measurements performed on ID2 beamline.



Both sample preparation and measurements have been performed at room temperature.
Time behaviour of the autocorrelation functions for a C_w=3.0% sample measured by PCS setup are shown in Fig. 2. At increasing waiting time t_w (defined as the time passed from sample filtration) a clear aging dynamics of the system is observed: autocorrelation curves evolve in time with characteristic relaxation times larger and larger. Two different decays can be distinguished: a diffusive one on shorter time scale (ms) and another one, due to the cage rearrangement during aging, which becomes slower approaching the arrested phase. In order to follow the aging evolution beyond the experimental limits of the conventional PCS technique, XPCS measurements have been performed on the same sample.

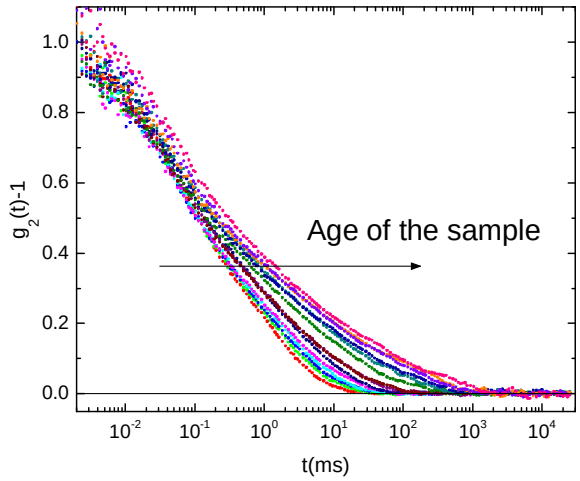


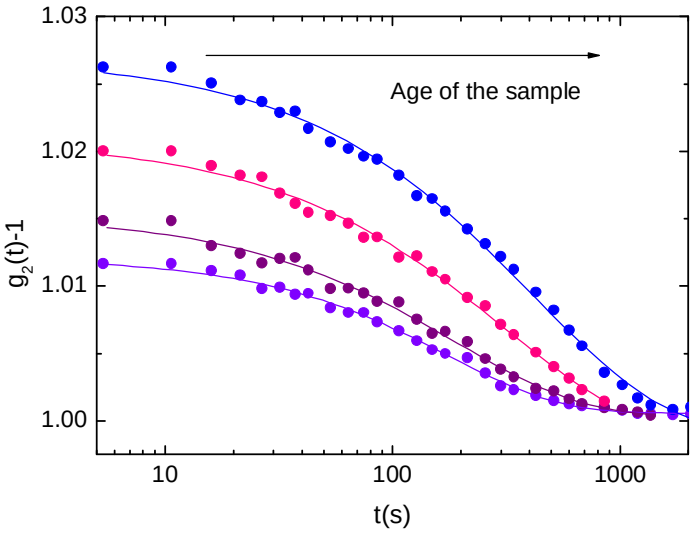
Fig.2 : Evolution of the autocorrelation function with age of the system. Characteristic relaxation times become slower and slower reflecting the approaching of an arrested state.

In Fig. 3 experimental curves obtained through XPCS technique at ID10 beamline are shown. Solid lines superimposed to data points are fits performed through equation:

$$g_2(t) = 1 + (B\exp(-(t/\tau)^{\beta}))^2 \qquad (1)$$

The fits describe very well the experimental data. Also in this case we can observe a clear aging phenomenon toward time scales longer and longer.

Fig.3 Waiting time evolution of experimental XPCS curves for a sample with C_w=3.0% concentration. Fits obtained through equation (1) are shown as solid lines.



Moreover the same sample has been filtered and stored in several capillaries and systematic XPCS measurements have been performed on each ones. In this way we can probe either the reproducibility of the sample either the eventual presence of beam damage.
From these measurements we can affirm that XPCS signal due to the sample is present, that there is not evidence of beam damage and that the samples present the same time evolution during aging. Nevertheless any evident crossover towards the full aging regime has been observed in the investigated waiting time region for the studied samples. In fact the characteristic stretched exponent β remains always less than 1 for the whole investigated dynamical evolution. This result is in disagreement with previous results already present in literature and need for more detailed investigations.

[1] B. Ruzicka *et al.* Phys. Rev. Lett. **93**, 258301 (2004).
[2] B. Ruzicka *et al.* Langmuir **22**, 1106 (2006).
[3] M. Bellour *et al.* PRE **67**, 031405 (2003); R. Bandyopadhyay *et al.* PRL **93,** 228302 (2004); F. Schosseler *et al.* Phys. Rev. E. **73**, 21401 (2006).