ESRF	**Experiment title:** Structural insight into the formation of metal-organic gels	**Experiment number**: CH-6000
Beamline: ID02	**Date of experiment**: from: 07/20/2021 to: 07/22/2021	**Date of report**: 08/20/2021
Shifts: 6	**Local contact(s)**: Peter Boesecke	*Received at ESRF:*

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Report:

Experiments were carried out at the ID02 beamline at the European Synchrotron Radiation Facility (ESRF). A photon energy of 12.46 keV was selected, corresponding to a wavelength of $\lambda = 1$ Å. Scattering intensities were measured with the EIGER2 2D CCD detector at 4 and 30 m detector position. This setup covers a q-range of $0.05 < q < 5.5$ nm^{-1} and $2\times10^{-3} < q < 0.02$ nm^{-1}, respectively. Wide angle scattering was measured during the SAXS experiments at ID02 beamline by operating the Rayonix LX170-detector simultaneously. This option covers a q-range between $5 < q < 30$ nm^{-1}. The samples were filled in a glass capillary (Ø = 1 mm) at 25 °C. The sealed capillary was inserted into Mettler-Toledo heating stage to perform in-situ study from 30°C to 120°C. The obtained 2D scattering patterns were normalized to an absolute intensity scale and azimuthally averaged following the standard procedure to obtain the one-dimensional scattering profiles which were further averaged among the 3 measurements. The obtained scattering intensities were averaged during the data reduction process.

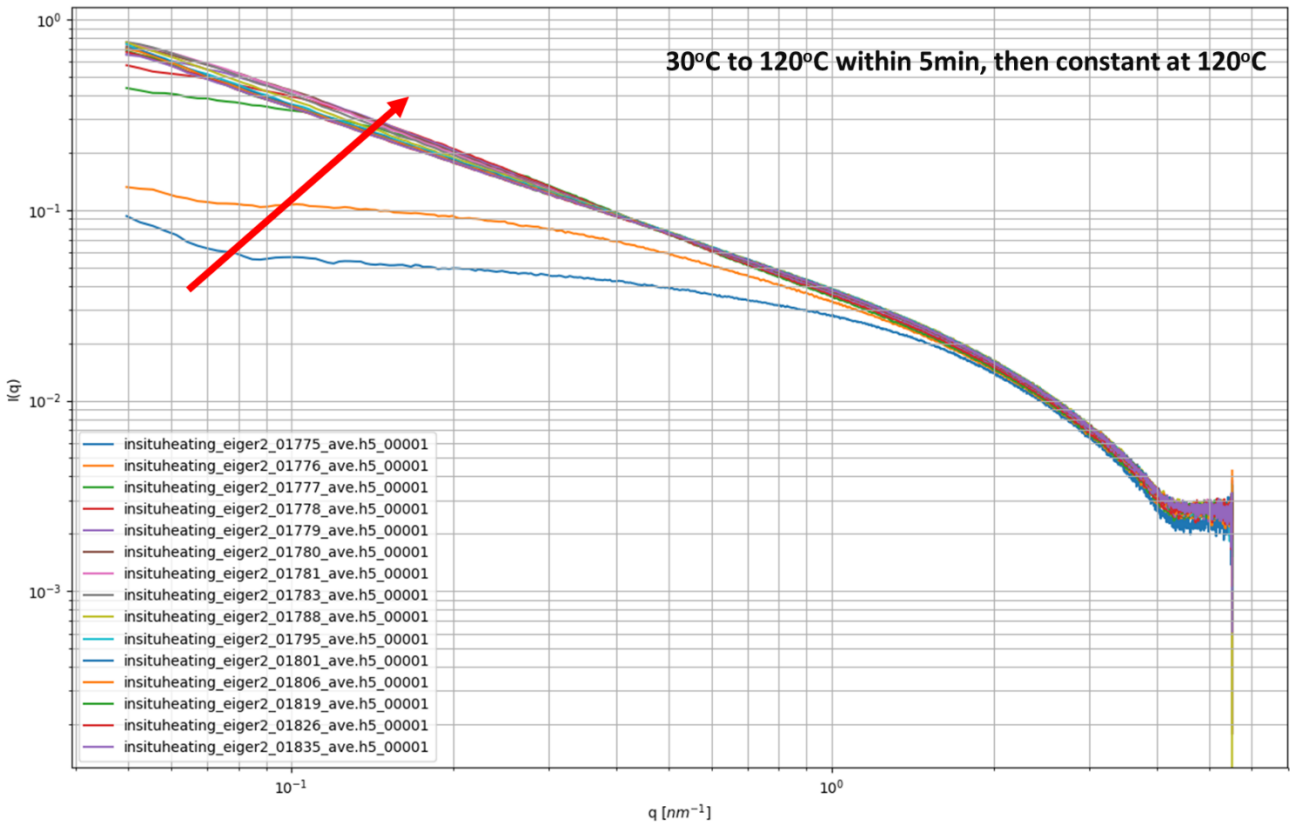

Figure 1. In situ SAXS characterization of the formation of Zr-gel, covering a q-range of 0.05 < q < 5.5 nm^{-1}. Patterns recorded every 5 min from 0 to 120 min.

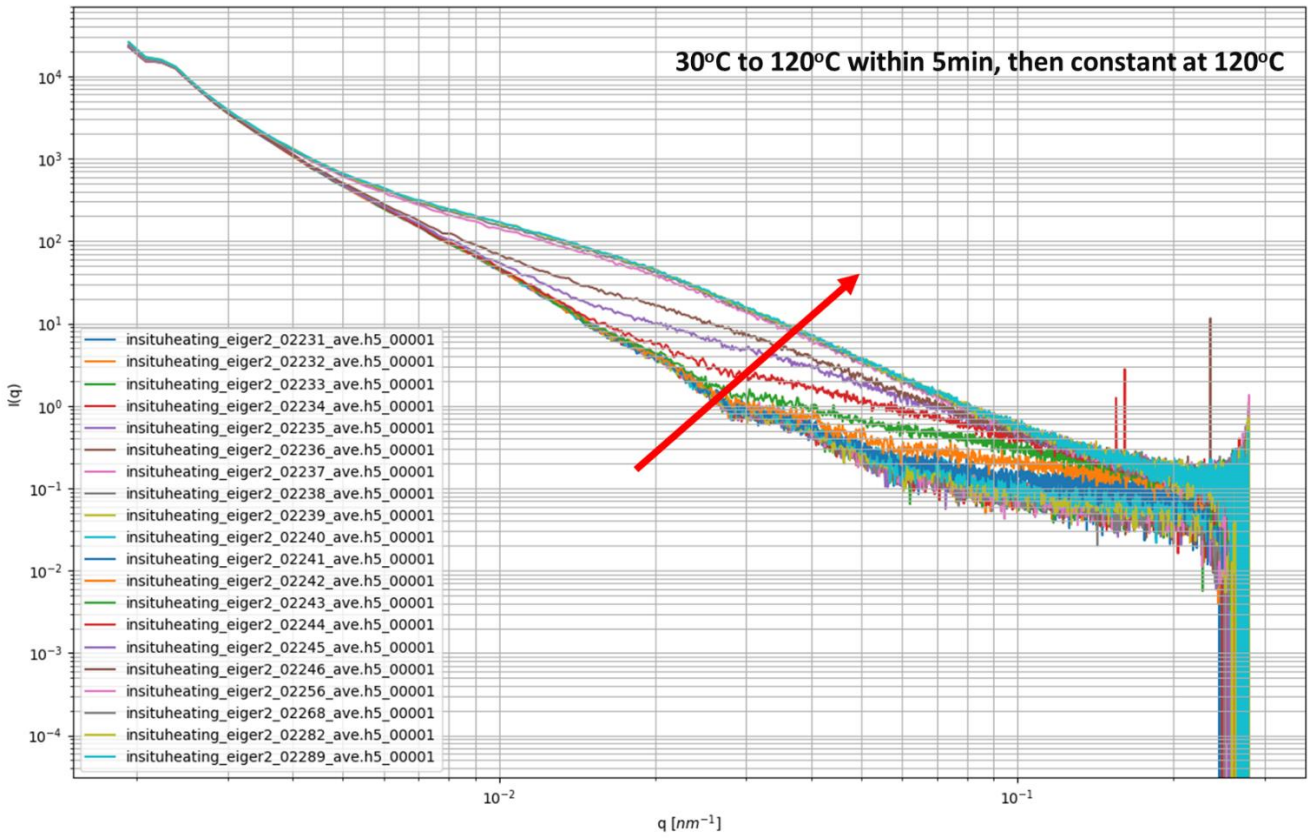

Figure 2. In situ USAXS characterization of the formation of Zr-gel, covering a q-range of 2×10^{-3} < q < 0.02 nm^{-1}. Patterns recorded every 5 min from 0 to 120 min.

In the proposal we planned to study in-situ Zr-gel formation for a better understanding of the structural evolution and texture of colloidal. We proposed 48 samples to be collected, each to be carried out every 2 min over 2 hours. Indeed, we collected 15 samples covered by a q-range of $0.05 < q < 5.5$ nm^{-1}, 5 samples covered by a q-range of $2\times10^{-3} < q < 0.02$ nm^{-1}. This is because of the longer time (8h) than expected for initial setup, beamline alignment, and intensity correction prior to data acquisition. At the end we collected USAXS region for Zr-gel as we found there were some features for bigger particle assembly.

Here we demonstrate one of the many examples studied in ID02. In Figure 1, the log–log plot of a typical time-resolved SAXS profile from an experiment with 3wt% Zr-gel precursor is shown. Note that in this experiment SAXS patterns were collected every 5 min over a time length of 120 min but for clarity only a few patterns are shown. The two dominant features in these plots were (i) the change in scattering intensity, $I(q)$ with time and (ii) the increase in the slope at low-q (i.e., Guinier region defined by $qR_g < 1$). The increase in $I(q)$ at low q is related to a change in electron density contrast between the matrix and the newly formed particles and a change in the total scattering volume (i.e., increase in particle volume or number) whereas the increase in the slope indicates an increase in the particle size with time.

In Figure 2, a clear trend of larger objects growth could be observed from USAXS region. A progressive increase in object size was anticipated due to smaller particle self-assembly. Data were collected until no further change in the scattering pattern was observed, which was taken to indicate the end of the reaction.

Data treatment and fitting is on the way through a collaboration of Brian Pauw and Glen Smales from Federal Institute for Materials Research and Testing (BAM), Germany.