	Experiment title: GLASS TRANSITION VS. STRUCTURAL PHASE TRANSITION IN DILUTE SUSPENSIONS OF CHARGED VIRAL RODS	**Experiment number**: SC-4648
Beamline: ID02	**Date of experiment**: from: 27/04/2018 to: 29/04/2018	**Date of report**: 29/08/2018
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Report:

The aim of the experiment was to investigate by SAXS the self-organization in a model system of colloidal charged rods, namely filamentous *fd* viruses, for which a glass transition has been reported at very low ionic strength (*I*=0.16mM) in 2013 (https://doi.org/10.1103/PhysRevLett.110.015901). However, preliminary investigations by optical microscopy of the same system performed by our group are in contradiction with such a claim of a glass transition: instead, a phase transition (from the liquid-crystalline nematic to the crystalline smectic-B phase) has been evidenced in the same range of rod concentrations. In order to distinguish between a glass transition and a phase transition, structural investigations by SAXS are required, especially considering that virus suspensions at very low ionic strengths correspond to very dilute suspensions and therefore to large inter-rod distances.

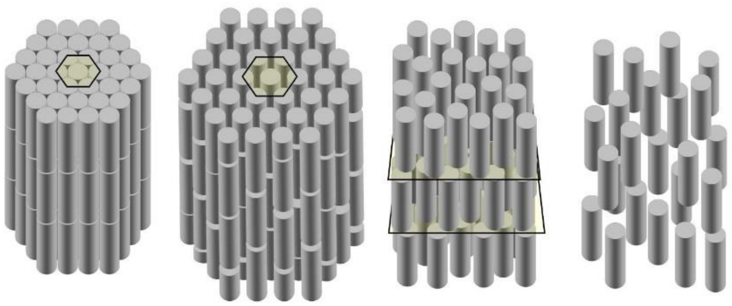

Figure 1: *Schematic representation of the different mesophases formed in aqueous suspensions of filamentous* fd *virus particles at high ionic strengths (I>5mM).*

We have studied different samples mainly probed at a sample-to-detector distance of 5m. A few samples were also studied at lower q, with a sample-to-detector distance of 31m. As detector, we used the FRELON from ESRF. The energy of the X-ray beam was initially set at 14 keV, however, due to sample damage under X-ray exposure, the energy has been increased up to 16 keV. Note that, even at this high energy, we had to perform sequential,

short acquisitions (performing therefore average afterwards to increase the count statistics) including rest times of up to a few tens of seconds between two frames in order to limit the detrimental effects of radiation damage. More precisely, two kinds of sample evolution have been seen under X-ray radiation: i) a shift of the main Bragg peak (100) to lower q. This shift is attributed to a local heating of the sample (and is therefore reversible if we let the sample relax for a very long time). However it contributes to an artificial peak broadening which is a problem for any quantitative line shape analysis of the Bragg reflections ii) If the exposure time under X-ray is increased, irreversible degradation has been observed. It is characterized by a strong decrease of intensity of the main Bragg reflections, leading ultimately to the loss of the crystalline structure of the phase.

These evolutions under radiation of our samples have clearly complicated and slowed down our data recording, as a significant rest time was mandatory between two exposures. Despite these difficulties, we succeeded in determining that the *fd* virus particle suspension at very low ionic strength do form a crystalline smectic-B phase in the dense regime, as proved by Figure 2 (red line), where a 2D hexagonal long range positional order is clearly observed.

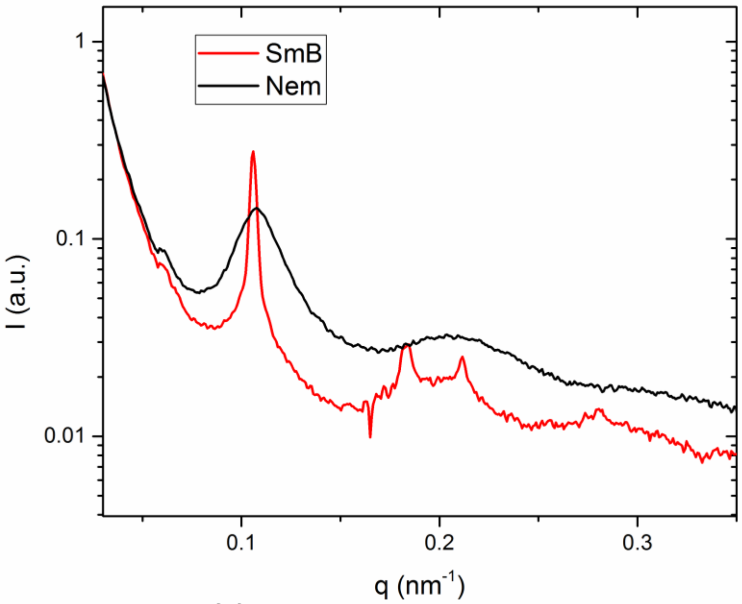

Figure 2: *Scattered intensity of* fd *virus suspension at low ionic strength. The red curve is characteristic of a long range hexagonal order normal to the viral long-rod axis, as a signature of a Smectic-B phase. For comparison, the signal obtained for a liquid-like order is also shown (black line), as measured in the nematic phase.*

Note that some unconsistencies have been observed in the capillaries prepared a few days before the synchrotron run, as many of them did exhibit an uncontrolled evolution related to, as far as we can interpret it, a change of ionic strength with time. Therefore, the allocated 6 shits were very useful to fully probe all the prepared samples, and fianly obtained a set of consistent results.

In conclusion, the main goal of the proposal has been achived with the clear identification of a structural phase transition from Nematic to Smectic-B phase (ruling therefore out any glass transition) in suspensions of charged rod-like particles at very low ionic strength. The corresponding publication has still to be written after the complete anlysis of the data (including line shape analysis of the Bragg reflections).