	Experiment title: Time-resolved studies of the self-assembly of polyphiles - a novel class of amphiphiles	**Experiment number**: **CH-4293**
Beamline: ID02	**Date of experiment**: from: 21.04.2011 to: 25.04.2011	**Date of report**:
Shifts: 12	**Local contact(s)**: Jeremie Gummel	*Received at ESRF:*

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Report:

Synchrotron SAXS experiments were performed on the ID02 beam line (ESRF, Grenoble, France) using a pixel detector (1M PILATUS). The X-ray scattering images were recorded using a monochromatic incident X-ray beam ($\lambda = 0.154$ nm) covering the $0.016\ nm^{-1} < q < 4.2\ nm^{-1}$ range of momentum transfer ($q = 4\pi/\lambda \sin\theta$), with 2θ the scattering angle).

Early stages of polymeric nanoparticles formation is monitored by in-situ synchrotron SAXS experiments. Recently elaborated new strategy enables to prepare stable polymeric nanoparticles from homopolymers (not copolymers) of one type only and without any assembly-triggering additives. Poly(ethylacrylic acid) in aqueous solution is tuned to the pH where it exhibits thermosensitivity and the formation of nanoparticles is temperature-induced. The kinetics of this formation is clearly monitored by SAXS. Experiments indicate slightly elongated, not exactly spherical shape of the resulting nanoparticles confirmed also by cryo-TEM. Neutron Spin Echo spectroscopy shows that the PEA nanoparticles are relatively loose with a lot of solvent inside, in agreement with results from previous light scattering work.

In-situ synchrotron SAXS was successfully used to monitor in real time early stages of polymeric nanoparticles formation according to a new strategy elaborated recently by M. Sedlák et al., based on heating of a thermosensitive relatively highly charged polymer

(poly(ethylacrylic acid)) capable of effective and fully irreversible hydrogen bonding upon self-assembly into discrete aggregates/nanoparticles. The self-assembly process was clearly seen and evaluated in both the low q range (Guinier regime) and intermediate q range (Kratky plot). GNOM analysis indicated slightly elongated, not exactly spherical shape of the resulting nanoparticles. This conclusion was also supported by cryo-TEM pictures of the resulting nanoparticles. The best fit of the form factor was obtained with a model of sphere with attached Gaussian chains with polydispersity over size of attached Gaussian chains. Neutron Spin Echo spectroscopy revealed the Rouse behaviour, which means that the formed PEA nanoparticles are relatively loose with a lot of solvent inside, definitely not rigid and densely packed. This is in agreement with results from previously published light scattering work showing relatively low values of particles density ($\rho = 3Mw/4\pi R3$).

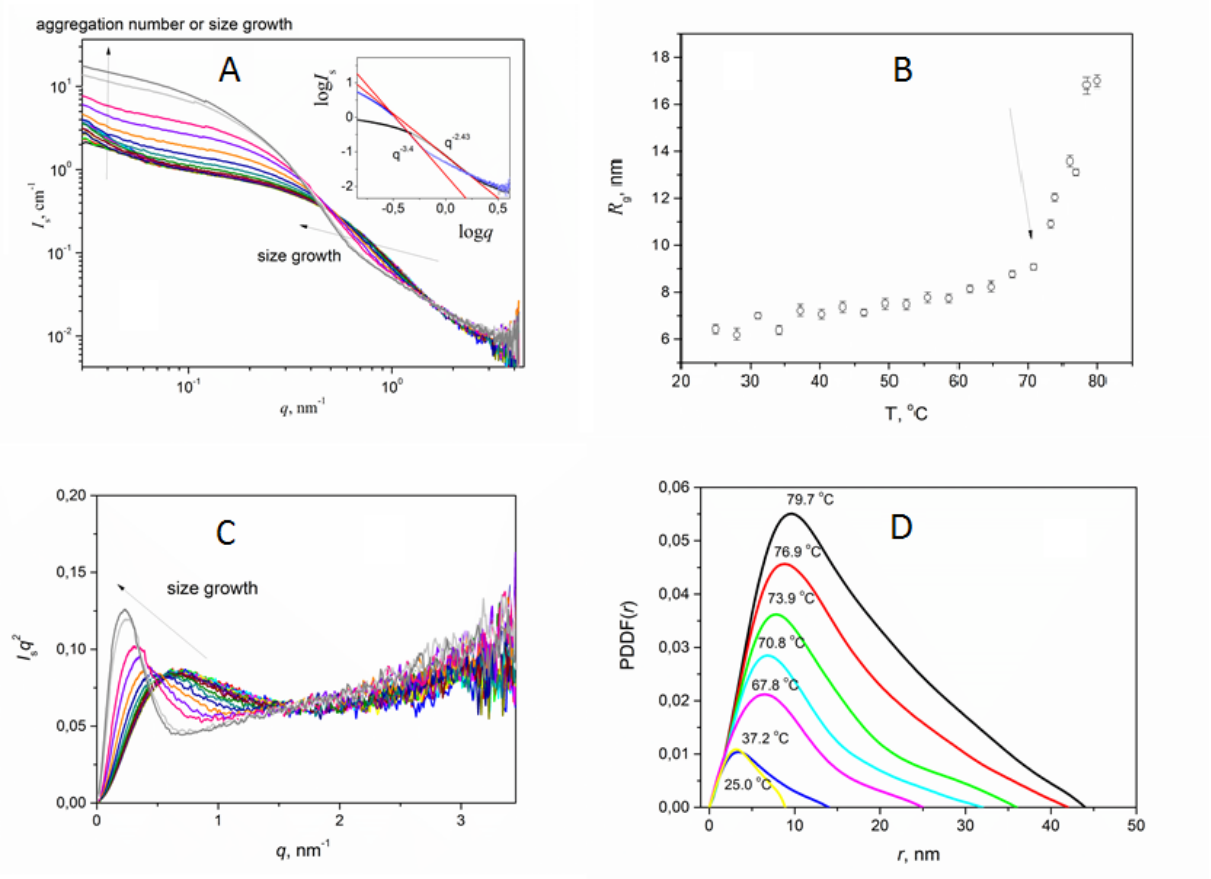

Figure 1. (A) Scattered intensity I_s as a function of the scattering vector q for different temperatures. Inset shows a log-log plot for the initial (25ºC) and final temperature (80ºC), respectively. (B) Temperature dependence of the apparent radius of gyration. (C) Kratky plot for different temperatures. (D) Pair-distance-distribution function for different temperatures.