



Experiment title:
Helical colloidal assemblies in 1D-confinement

Experiment number:
SC-4175

Beamline: ID02	Date of experiment: from: 29-10-2015 to: 01-11-2015	Date of report: 15-3-2016
Shifts:	Local contact(s): Sylvain Prevost	<i>Received at ESRF:</i>

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Report:

Mixtures of β -cyclodextrin (β -CD) and sodium dodecyl sulfate (SDS) form complexes in a 2:1 molar ratio at elevated temperatures. Upon cooling to room temperature these SDS@2 β -CD complexes self-assemble into hollow ‘annular ring’ microtubes. By adding colloidal particles into the mixture colloid-in-tube assemblies are obtained after a heating/cooling cycle. Depending on the ratio of colloid-to-tube diameters various structures can be formed such as zigzag, zipper and helical sphere chains (see figure 1).

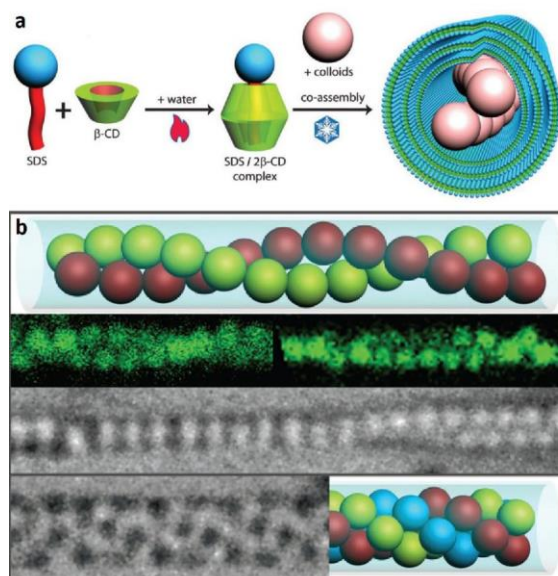


Figure 1: (a) Co-assembly of SDS@2 β -CD microtubes and colloids. (b) Helical structures are formed upon confining colloidal spheres in microtubes.

The aim of this experiment was to study the multiscale self-assembly of SDS and β -CD into microtubes and the formation of helical colloidal assemblies in 1D-confinement. The self-assembly of this complex system was characterized by SAXS at ID02, covering a total of three orders of magnitude of spatial scales.

Figure 2 shows the radial intensity profile and 2D SAXS pattern of microtubes. At small q (in blue), form factor peaks can be seen that correspond with the microtube diameter. Equidistant peaks (in green) appear at intermediate q , that are characteristic of 1D periodicity corresponding with a lamella structure. The spacing between these peaks gives the distance between the bilayers of the microtubes which is about 23 nm. At large q (in black), a saw-tooth shaped peak can be distinguished. This feature is characteristic of 2D structures. From the SAXS pattern we can conclude that the microtubes are rolled around the horizontal axis since the broadening of the peaks is only present in the horizontal and not in the vertical direction.

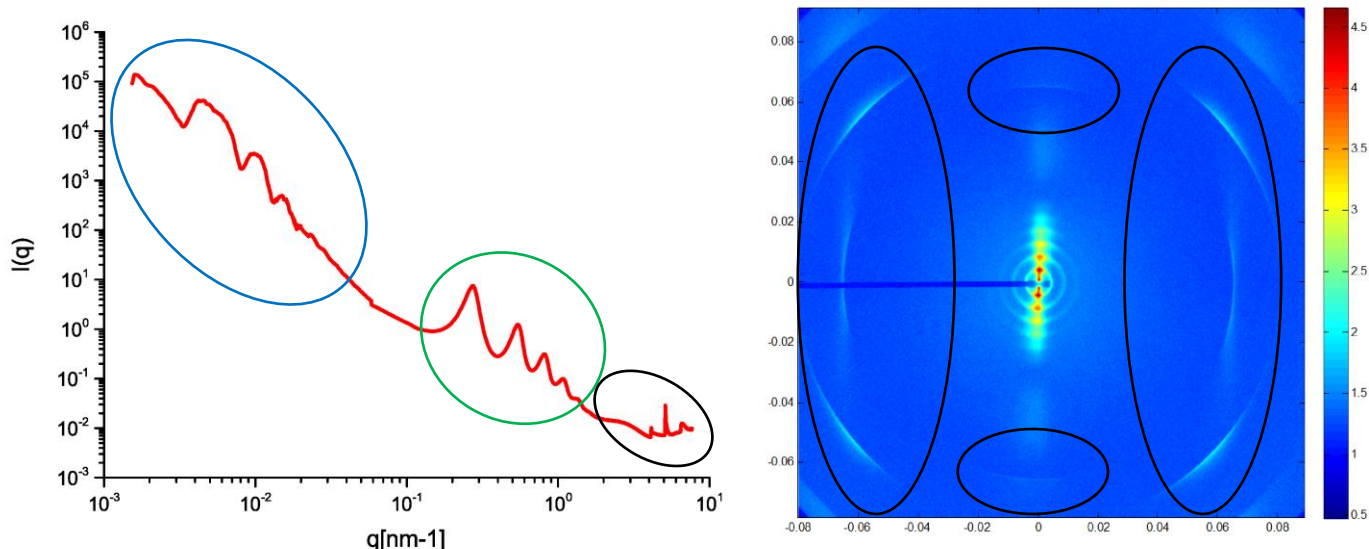
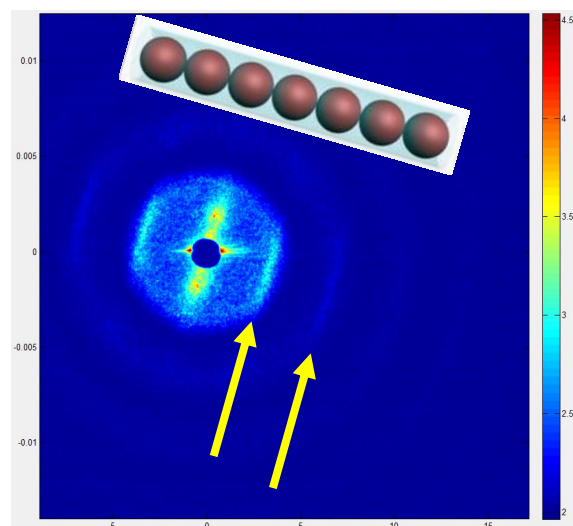


Figure 2: Radial intensity profile (left) and 2D SAXS pattern of microtubes (right).

Colloids with a diameter approximately equal to the microtube diameter can form sphere chains inside the tubes. In reciprocal space, this results in beautiful Bragg sheets as can be seen in figure 3.

Figure 3: 2D SAXS pattern of colloidal sphere chains in microtubes along with a model illustrating chain orientation. The arrows point onto Bragg sheets originating from 1D periodicity between the particles in the chains.



To summarize, with the data that we have collected we can completely characterize the multiscale assembly of SDS and CD into microtubes over three orders of magnitude of spatial scales. Furthermore, chains of colloids can be observed inside the microtubes at the right colloid-to-tube diameter. Since the formed structures depend sensitively on the colloid-to-tube diameter, samples were prepared with varying sizes of colloids. The data obtained from smaller colloids, expected to form chiral and helical structures, are being analysed extensively at the moment.

Finally, we would like to thank dr. S. Prevost for his excellent support not only during the week but also in the weekend.