Electronic Supplementary Material (ESI) for Soft Matter.

In-Situ Observation of Self-Assembly of Sugars and Surfactants from Nanometres to Microns

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Supplementary Information

Experimental Section

Materials

Chemicals

Sodium dodecyl sulfate (SDS, >97%), β-cyclodextrin (β-CD) and Nile Red (bioreagent for HPLC, >98 %) were purchased from Sigma-Aldrich. β-CD was dried before use to remove excess water. All water used in the experiments was purified by a Milli-Q system.

Sample preparation

Desired amounts of SDS, β-CD and water were weighed to obtain a molar ratio of β-CD:SDS of 2:1 and a final concentration of SDS and β-CD of 10, 15 or 20 wt% in water. The mixture was heated in a oil bath to 60 °C while stirring with a magnetic stirrer until a transparent and isotropic solution was obtained. Upon cooling to room temperature the microtubes were formed and a viscous and turbid suspension is observed. The microtubes were incubated for at least 48 hours before use.

Methods

Small-Angle X-ray Scattering (SAXS)

For SAXS measurements, microtubes suspensions were transferred to round quartz capillaries with a length of 80 mm, a diameter of 1 or 2 mm and wall thickness of 0.01 mm (Hilgenberg GmbH or Hampton research). Capillaries were sealed with epoxy glue (Bison kombi, two component glue). Samples were measured after 48 hours of incubation but within 5 days of sample preparation. SAXS measurements were performed at the high brilliance beamline ID02 at the ESRF (Grenoble, France).[1] X-rays with a wavelength of 0.0995 nm were used. Three different sample-detector distances LSD were used. For the smallest q-range the scattering data were recorded by FReLoN 16M detector with LSD = 30.7 metre. For mid-q

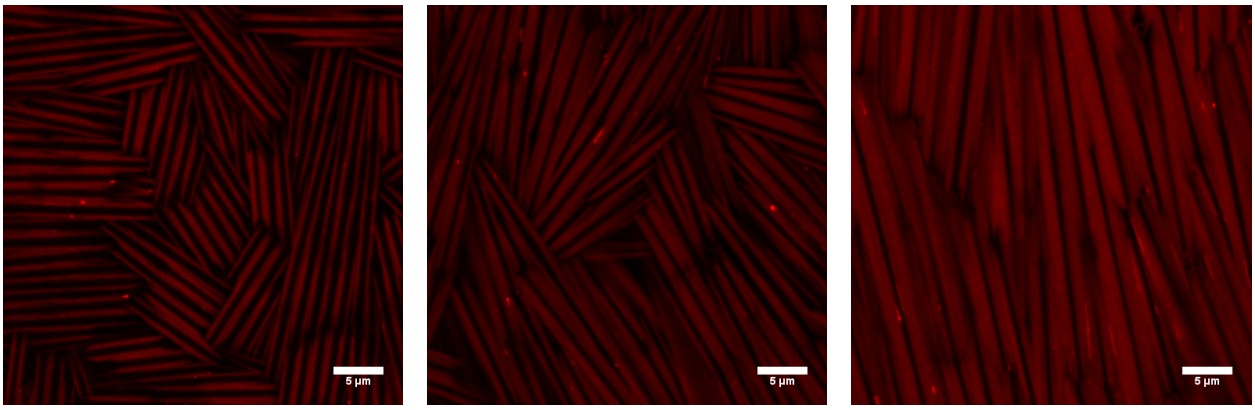
and large-q range the Rayonix MX-170HS was used with LSD = 12 and 1 metre respectively. This set-up made it possible to record scattering data from q = 0.0015 nm^{-1} to 8 nm^{-1}, where $q = (4\pi)/\lambda \sin(\theta)$ with 2θ the angle between the incident and scattered waves.

Background corrections were performed on all radial intensity profiles. Background was obtained from scattering recorded from capillaries filled with pure water. Scattered intensity was averaged over at least ten measurements.

Confocal Laser Scanning Microscopy (CLSM)

Samples were dyed with Nile Red. A stock solution of 1 mg/mL Nile Red in acetone was made. 10 μL of the stock solution was added to 1 mL of microtubes heated to 60 °C. The sample was sonicated for 1 minute while cooling down and was allowed to equilibrate overnight. Microscope samples were made by sealing a drop of microtubes suspension between two glass slides (Menzel-Gläser, 0.17 mm thick). A Nikon TE2000U inverted microscope with a C1 confocal scan head and a 100× Nikon oil objective was used to perform CLSM experiments. A HeNe laser with a wavelength of 543.5 nm (Melles Griot) was used to excite the Nile Red dye.

Supporting Figures



(a) 10 wt% SDS@2β-CD **(b)** 15 wt% SDS@2β-CD **(c)** 20 wt% SDS@2β-CD

Figure 1: Confocal microscope images of (a) 10, (b) 15 and (c) 20 wt% SDS@2β-CD complexes in aqueous solution. As the concentration is increased the overall diameter of the microtubes increases. The walls of the microtubes become thicker as the pore diameter decreases in size.

References

(1) Van Vaerenbergh, P.; Leonardon, J.; Sztucki, M.; Boesecke, P.; Gorini, J.; Claustre, L.; Sever, F.; Morse, J.; Narayanan, T. *Proceedings of the 12th International Conference on Synchrotron Radiation Instrumentation (SRI2015)* **2016**, *1741*.