

**Experiment title:**

Polymerisation in Bilayer Membranes

Experiment**number:**

SC-534

Beamline:

ID 01

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8

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Report:

Polymerisation reactions in lyotropic liquid crystalline phases are a research area of common interest to colloid-, polymer- and material chemists. To this end, lyotropic liquid crystalline phases are charged with monomer molecules and subsequently a polymerisation reaction is initiated with the intention to produce a polymeric material that, in an ideal case, would *preserve* and *stabilise* the structure of the employed surfactant phase.

Our own research in this field concerns free radical polymerisation reactions in surfactant bilayers. It appeared that the polymerisation of styrene within the bilayers of Dioctadecyldimethyl ammoniumbromide (DODAB) *vesicles* leads to peculiar polymer colloid morphologies by phase separation: small polymer latex beads are attached to vesicle bilayers to form vesicle-polymer hybrid particles. In order to characterise the influence of the monomer/polymer on the surfactant mesophase in more detail, we performed this SAXS/WAXS study. As adequate model system for the vesicle bilayers, we applied lamellar DODAB / water systems ranging from 20-50 %wt of DODAB.

In a second step, styrene was added to the DODAB / water systems in a molar ratio of [DODAB]:[styrene]=1:1 and 1:2.

After addition of an initiator (AIBN) and heating to 70 °C, polymerisation was induced. Scattering experiments were performed at the experimental station ID01 operating with a sample to detector distance of 70 cm or 430 cm. Samples were mounted in small brass cuvettes having kapton windows on both sides, being thermostatted in a Linkam hotstage.

Results:

DODAB/ water systems:

Up to 55 °C and independent of DODAB concentration, all systems show a gel-like lamellar phase characterised by a constant lamellar spacing of 37 Å. In this temperature interval, the lamellar phase coexists with an excess water phase. Above the phase transition temperature at about 55 °C, lamellar phases are established where the spacing linearly decreases with increasing concentration DODAB.

DODAB / styrene / water systems:

At low temperatures between 10-20°C hardly any influence of the present styrene can be detected: independent of DODAB concentration, lamellar phases ($d = 37 \text{ \AA}$) similar to those of the pure DODAB / water system are found.

At elevated temperatures, however, the ternary systems show a rather diverse behaviour. While DODAB concentrations lower than 10 %wt lead to lamellar bilayer phases, hexagonal and cubic phases are seen at concentrations higher than 20 %wt.

The low temperature phases typically show diffraction peaks from oriented micro-domains in the sample (see figure 20 %wt DODAB, 25°C, 1:2 styrene)

on-line polymerisation of styrene in DODAB lamellae:

The polymerisation of styrene in cubic DODAB phases, (e.g. 30 %wt DODAB, 1:1 styrene, 70°C), does not lead to phase changes on the time scale of polymerisation.

Cooling to room temperature after polymerisation and repeated heating seems to induce phase separation and destruct the cubic phases.

