

## Report beam experiment 26-02-286

In this experiment the influence of ionic strength and soap concentration was studied on the liquid crystalline structure of distilled saturated monoglycerides. Above the melting temperature of the hydrocarbon chains, a lamellar phase or dispersion appears. In this phase, the amphiphilic molecules form double layers separated by layers of water. The lamellar phase or dispersion was characterized (SAXS) by sharp and strong reflections, the spacing ratio of which is 1:2:3:4. Up to three orders were found using the experimental set-up at DUBBLE ( $E = 10$  keV; Sample to detector distance = 2,5 m). The lamellar phase or dispersion transforms into a cubic phase (+ water) upon heating. The cubic phase shows reflections with a spacing ratio of  $\sqrt{3} : \sqrt{4}$ . The analysis of the X-ray diagrams of the cubic phase is generally hindered by a heavily spotted appearance, a consequence of the easy growth of fairly large crystals. This was the case in the experiments. In liquid crystalline structures the arrangement of the hydrocarbon chains corresponds to that in a liquid hydrocarbon. This is indicated by a weak and relatively diffuse X-ray diffraction band at wide angles corresponding to a Bragg spacing of about 4,5 Å.

By cooling the lamellar phase transforms to the much more viscous gel phase, which is the hydrated form of the  $\alpha$  polymorph. This phase consists of monoglyceride bilayers, whose alkyl chains are crystalline, separated by water layers. The gel phase in turn may transform to the coagel, on a time scale depending on composition, previous conditions of treatment and storage conditions. It was shown that the molecular arrangements within the bulk  $\beta$ -crystal of monoglyceride and the coagel phase are identical. The  $\alpha$ -gel is characterized by the occurrence of a single x-ray short spacing at 4,15Å. This indicates that the lateral packing of the chains can be described by a hexagonal subcell and that the chains possess rotational or oscillational freedom.

In the first experimental set-up distilled saturated monoglyceride water systems were prepared with following varying parameters:

- ✓ Ionic strength: 0; 0,005 mol/l and 0,010 mol/l;
- ✓ Soap concentration: 0,5; 1,0; 1,5 and 2,0% on content of emulsifier.

This mixtures were heated from 50°C to 90°C at a heating rate of 2°C/min. The changes in short and long spacings were followed to study the crystals + water to lamellar phase or dispersion transition and lamellar phase or dispersion to cubic phase (+ water) transition. The ionic strength and soap concentration doesn't have a significant influence on the transition temperature of crystals (25%) + water to lamellar phase. In contrast, it influences the transition lamellar phase or dispersion to cubic phase (+ water). Soap stabilizes the lamellar phase or dispersion in function of temperature as a higher ionic strength in the water phase destabilizes the lamellar phase or dispersion.

In the second set-up the lamellar phase of the same systems as mentioned were cooled to 20°C as fast as possible. The changes in crystal lattice types (WAXS) – associated with the different phases – and long spacing (SAXS) were followed as a function of time. During cooling a transition of the lamellar phase or dispersion to the gel phase was observed. During the time frame of the experiment (6 hours), no transformation of the  $\alpha$ - to  $\beta$ -polymorphic form was observed.