	Experiment title: Triglyceride crystallisation in milk and dairy emulsions : Study of liquid-crystalline structures and phase transitions by coupling of Differential Scanning Calorimetry and High Resolution Small Angle X-ray Scattering.	**Experiment number**: SC796
Beamline: ID02	**Date of experiment**: from: 7-jul-2001 to: 10- jul-2001	**Date of report**: jan 2001
Shifts: 9	**Local contact(s)**: Stéphanie FINET	*Received at ESRF:*

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Report:

SETUP

ID02 was setup with a double detection using SAXS an WAXS cameras (with a new cone for the WAXS). The sample to SAXS detector distances were selected at 1248, 2500 and 6500 mm. Two sample-holders developed at UMR8612 were installed at the focal point of the WAXS. A calorimeter (cryostat+peltier cooled) was placed on the XYZ table in a special insulation chamber allowing its use at negative temperature without any water condensation for days thanks to a dry air flow (successful). This very sensitive calorimeter allows for accurate heating or cooling temperature scans as well as isothermal recording while measuring and comparing exchanges between the sample-containing capillary (typ. 20mg) and a reference in the range –30 + 150°C. Phase transitions in an emulsion made from 10% fat are detected. A multiple sample-holder (with 2 rows of seven positions the spacing of which is 5 mm), was also used alternatively with the calorimeter. The temperature range of this multiposition sample-holder in which seven capillaries were installed, is –30 – 100°C. This device was also installed in the insulation chamber. A new design of this last device allowing the examination of frozen emulsions at negative temperature, neither destroying the texture of the material nor melting it, was succesfully used for the first time on a synchrotron bench. It allowed to obtain the first X-ray pattern of frozen mixes (fat emulsion containing 10% triglycerides) both ice and fat patterns are observed simultaneously. The main organization observed is that of the triglycerides.

EXPERIMENTS AND RESULTS

Beam time was employed the following :

Milk fat based emulsions (cream and milk) were examined at positive (20 and 4°C) and negative (-8, -10 and –20°C) temperatures after various thermal treatments (after slow cooling or quenching). The time needed to launch an experiment has not allow the recording from the very first structures obtained after quenching. The existence of such a time lag evidenced the need for the development a new device capable of introducing the sample into the precooled chamber of the calorimeter while recording SAXS and WAXS frames in order to catch the very first structures formed. The realisation of such a device is underway. The polymorphic evolution of milk fat observed at 2500 and 1250 mm at SAXS angles is illustrated on the proposal (Figure 1). The triglyceride organisation in a foam made from milk fat was observed for the first time by X-ray diffraction to our knowledge thanks to the brigthness at ID02 bench. The observation temperatures ranged from 4 to
–24°C.

A series of vegetal fat based emulsions was also examined as model systems. These emulsions made from 10% fat gave excellent diffaction patterns on which the influence of the emulsifiers onto the triglyceride crystallization within the fat droplet can be analysed. The crystallization and melting of the vegetal fat inside the droplet of such emulsions were recorded either in the range 60 to 4 or 60 to –5 °C. A series of emulsions enriched in emulsifiers were also examined as an attempt to determine the emulsifier organization in real emulsions. Polymorphic evolution in emulsion revealed to be an excellent reporter of the influence of interface molecules (proteins and emulsifiers) onto crystallization processes in dispersed system.

Cyclodextrin/lipid and phospholipid/alkylglucoside mixtures were also succesfully examined by GL et CA at positive and negative temperatures respectively as preliminary experiments in order to determine the possible formation of new structures.

About 15 000 files were recorded. Several experiment setup need to be improved (cf. above) (e.g. improvement of the temperature control in the multiple position sample-holder). Most of these results are new and needs to be confirmed before publication.