

**Project name:** Encapsulation barrier properties of starch based emulsion systems

## Beamtime Report

**20.03.2014- dd.mm.yyyy** (Date of the report to be added)

### General information

Name of the rapporteur	Name of the rapporteur's organisation
Malin Sjöo	Speximo AB
Type of research (nanotechnology/health care/chemistry etc.)	Name of the research facility
Encapsulations for cosmetics/foods/pharmaceuticals	MAX IV Laboratory
Date of the measurement, duration	Location of the event
2013-11-20	Lund
Facility personnel participating in the measurement	
Tomás Plivelic, Axel Steuwer	

### Description of the project

Research description (short summary as written in the application)
<p>Speximo has a unique encapsulation technology based on starch as barrier in oil-in-water emulsions. Speximo's technology is primarily used for encapsulation and release of sensitive substances for applications such as cosmetics and skin care products, but can also be used in food and pharmaceuticals. The properties of the encapsulation barrier initially depend of the surface characteristics of the starch particles used, but can be further adjusted at the oil drop interface. Time and temperature treatments are known to alter the crystalline properties of starch and thereby the stability and permeability of the barrier layer. Since the starch in Speximo's systems is situated at the interface between oil and water, the result of time/temperature treatments will differ from aqueous systems that have been previously reported. The aim of this project was to investigate the starch barrier, mainly in terms of crystallinity and structure, during and after specific treatments in order to obtain increased knowledge to better control the effect of encapsulation, stability, and release properties.</p>
Summary of activities (experiments performed, beamtime used, preliminary overview of results, next steps and other relevant information)
<p>Speximo prepared samples of varying composition and pre-treatments. The samples were then analysed at MAX IV beamline I911-4. Both starch stabilized emulsions and starch dispersions were analysed and controlled against liquid reference samples. Temperature control was used for some experiments to further investigate the crystalline properties of the starch barrier. The effectively used beamtime was 11 h.</p> <p>The measurements provided important information regarding the structural properties of the starch and changes in crystallinity upon different treatment both for the starch material and for the encapsulating barrier in starch based emulsions. Crystallinity was</p>

lost upon heating, L values didn't change with temperature, only intensity. Emulsions and dispersions behaved similarly when analysed during heating, which is important in order to relate results to literature and results from different model systems. Measurements of stored samples after temperature treatment behaved similar to directly temperature treated samples indicating slow crystallization which may have implications on further encapsulation and release properties for specific purposes, this would mainly be confirmed using DSC or XRD. Comparison of non heated vs heated and cooled samples at room temperature after melting showed a peak at high q is still present in the emulsion, this could be related to the emulsion oil component, i.e control of additional excipients in formulations are important.

The information obtained will be related to *in vitro* nutritional data and DSC-measurements (under evaluation). When complementary experiments have been completed by Speximo the results will be related to the beamline results for publication. Furthermore, these results will add new knowledge for further interpretation of previously obtained data. The conclusions will help Speximo in further product development to design products and encapsulations for specific purposes within both oral and topical use.

**How would you describe cooperation and assistance from industrial liaison officers and national contact points while preparing and carrying out the research at large scale facilities?**

The contact and meetings and cooperation before and during experiments have been very important for the planning, performance and results of the research. The assistance have been highly professional and appreciated.

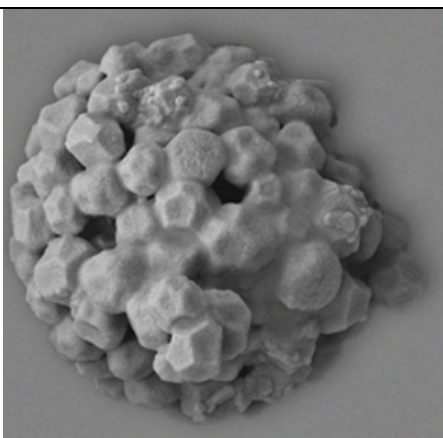
**Other personal remarks**

**Annexes**

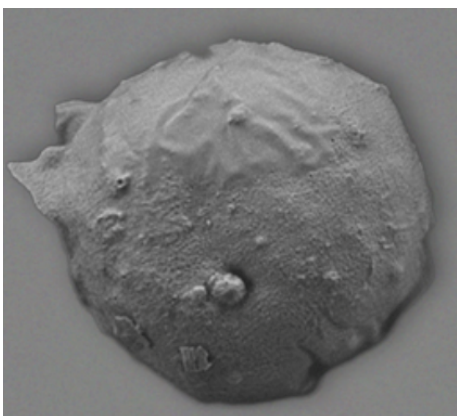
**Annexes**

(list of annexes; meeting minutes, graphical illustrations, tables and other supplementary data)

Graphical illustrations of samples and results obtained:

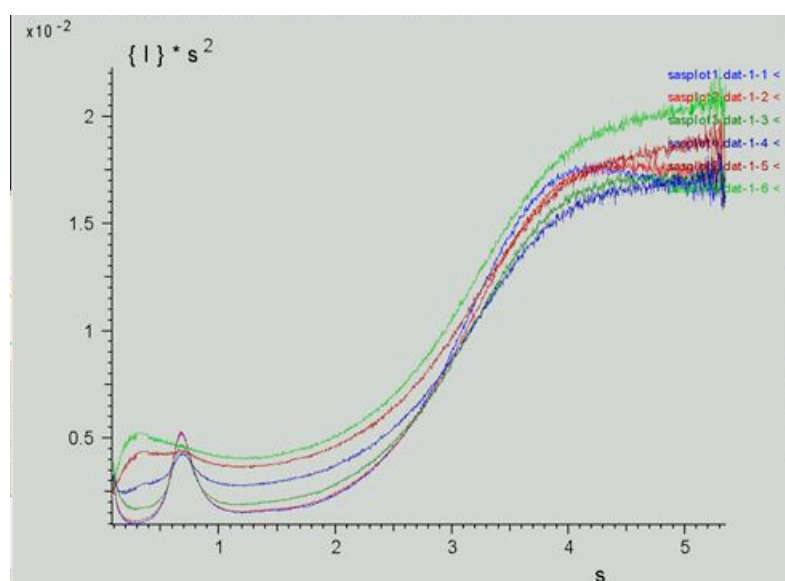


Starch emulsion based capsule before heating



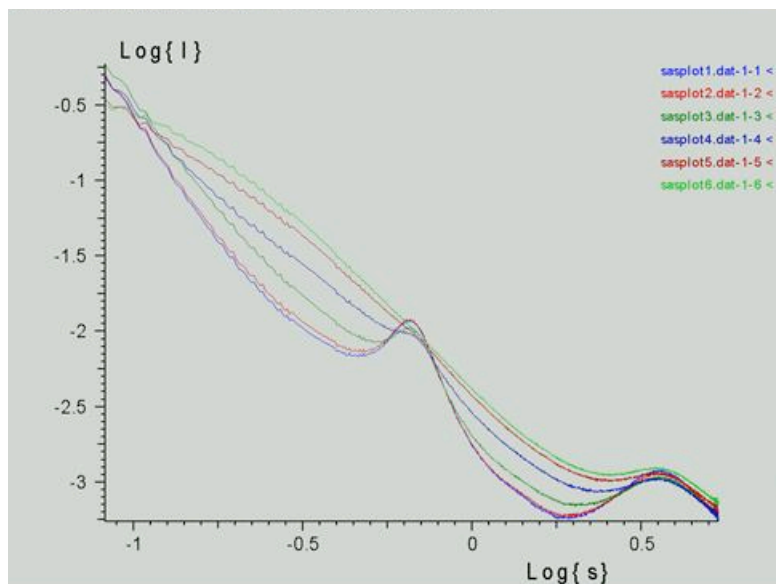
Starch emulsion based capsule after heating

Effect of heating, differently prepared samples:

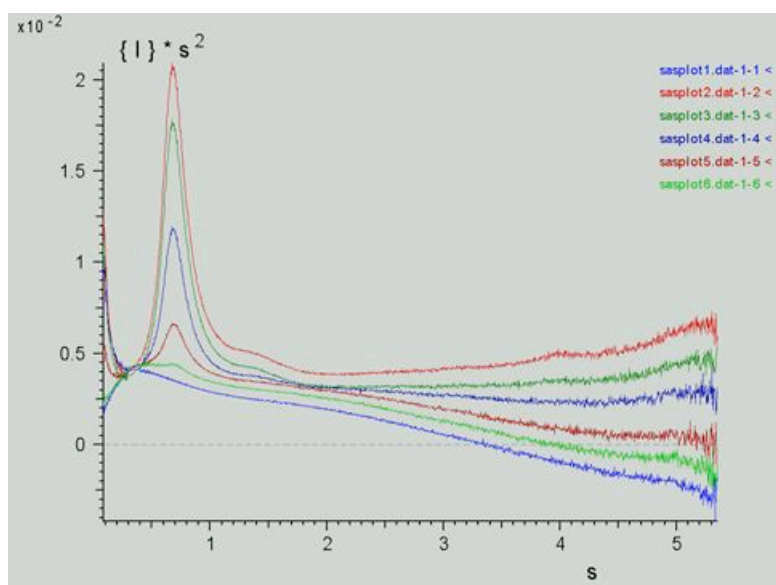


Iq2 vs q (t1, L(periodicity, crystalline/amorphous domains), ~9.2 nm)

Effect of heating, sample measured with temperature control:

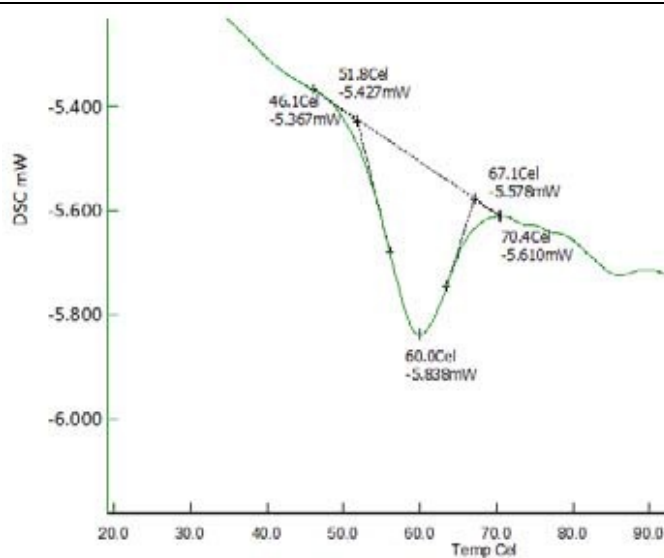


log I vs log q: Melting between T=60-65-75 C.



I q<sup>2</sup> vs q. Melting at 65-70 C.

Gelatinization endotherm of starch in water obtained by DSC:



dH vs T gelatinization peak at 60 C.