Neutrons and Food 2016

31 May – 2 June 2016
Lund, Sweden

The increased consumer demand for safe, healthy and environmentally friendly food have made it more necessary than ever to understand the complex structure of food and food ingredients and how they are affected by processing and digestion.

This has increased the demand for more sophisticated non-destructive analysis techniques, such as neutron and x-ray scattering. Scattering techniques have been used to reveal the structure of casein micelles in milk, food protein structures in bulk and at interfaces, as well as emulsion structures.

In order to explore the opportunity these techniques offer, the international user community has initiated a series of conference “Neutrons and Food”, with the first meeting in Sydney 2010, Delft in 2012, and Paris in 2014. While the conference series focuses on the use of neutron techniques, there is a strong synergy with the application of x-ray and light scattering methods and we will discuss a wider range of techniques.

The 4th International Conference on Neutrons and Food will be held in Lund on 31st May – 2 June 2016. The conference welcomes participants and contributions from the wider community of food science and product development, food packaging, and nutrition, as well as scattering.
In particular we aim to have a mixture of talks from food scientists and technologists with problems *where scattering methods may be applicable*, as well as talks from those already using scattering, and presentation of methods that are available.

The primary goal of the meeting is to bring together the food community with the scattering community to see how they can work together to solve problems.

There is also be a dedicated session, part of the project entitled ’New X-ray Imaging Modalities for High Quality and Safe Food’ (NEXIM), which is lead by Robert Krarup Feidenhans’l at Copenhagen University.

Organising Committee:

- Prof. Tommy Nylander (Lund University)
- Dr. Andrew Jackson (European Spallation Source & Lund University)
- Prof. Robert Feidenhans'l (Copenhagen University)
- Dr. Niklas Loren (SP Food and Bioscience)
- Zsuzsa Helyes (European Spallation Source)

Programme

<u>**Tuesday, 31 May 2016**</u>

12:00 ESS Construction Site visit

13:00 Lunch at ESS

14:00 MAX IV Lab visit (until 15:00)

<u>**Wednesday, 1 June 2016**</u>

08:30 Coffee

09:00 Welcome

09:30 Methods

09:30 Keynote : Food Science @ ANSTO – The First Ten Years - Elliot Gilbert (ANSTO)

10:10 Challenges for Sustainable Food Processing – Fredrik Innings (Tetra Pak)

10:35 *Current Status of 40m SANS Instrument and SANS-related Research at HANARO* - Eunhye Kim (Korea Atomic Energy Research Institute)

11:00 Coffee Beak

11:15 Imaging and Tomography

11:15 *The NEXIM Project* - Robert Feidenhansl (University of Copenhagen)

11:30 *3D Structures of Processed Meat studied by X-ray phase-contrast Tomography* - Rikke Miklos (Department of Food Science, UCPH)

12:00 *Revealing the 3D nanostructure of extended colloidal networks in food emulsions using ptychographic X-ray computed tomography* - Mikkel Schou Nielsen (Danish Fundamental Metrology A/S, Lyngby, Denmark)

12:30 Lunch

13:30 Imaging and Tomography

13:30 *Application of X-ray dark-field imaging for monitoring barley seed germination* - Kasper Borg Damkjær (University of Copenhagen, NEXIM)

14:00 *Neutron radiography/tomography for visualising and quantifying the novel fruit pulp concentration process "Solar Assisted Pervaporation"* - Randi Phinney (Lund University)

14:30 *Layered Surface Detection in Micro-CT Tetra Pak Data* - Vedrana Andersen Dahl (Technical University of Denmark)

15:00 Coffee break

15:30 Fats, Emulsions and Emulsifiers

15:30 Keynote: *Quantification of fat crystallite thickness distributions by NMR and small-angle scattering techniques* - John van Duynhoven (Unilever)

16:10 *Soybean Oleosomes studied by Small Angle Neutron Scattering* - Andrew Jackson (European Spallation Source)

16:35 *SANS study of the self-aggregation of alkylglycoside surfactants with oligomeric head-groups* - Federica Sebastiani (Physical Chemistry Department, University of Lund - CR Competence AB, Lund)

17:00 -18:30 Posters and Mingle

19:00 Conference dinner at Hypoteket (until 22:00)

Thursday, 2 June 2016

08:30 Coffee

09:00 Nutrition and Health

09:00 Keynote: *The fate of nanoparticles in the GI tract* - Alan Mackie (Institute of Food Research)

09:40 New Sustainable Technology for Pure Water - an essential ingredient for food - Adrian Rennie (Uppsala University)

10:05 *Can neutron or X-ray scattering techniques support food scientists to reveal important information on food structures?* - Niklas Lorén (SP Food and Bioscience)

10:30 Coffee break

11:00 Lipids

11:00 Keynote: *New Insights on the Interaction of Flavonoids with Biomimetic Membranes* - Michael Rappolt (School of Food Science and Nutrition, University of Leeds)

11:40 *Lipid sponge phases and nanoparticle dispersions able to entrap large biomolecules* - Maria Valldeperas (Lund University)

12:05 *The Lipid Binding Interactions of Indolines and Thionins : Cationic Cysteine Rich Antimicrobial Proteins from Cereal Seeds* - Luke Clifton (ISIS Spallation Neutron and Muon Source)

12:30 Lunch

12:30 Biopolymers

12:30 Keynote: *Protein gels from the inside with neutron scattering* - Wim Bouwman (Delft University of Technology)

12:55 *Glass transition and hydraion of food protein studied by inelastic neutron scattering* - Hiroshi Nakagawa (Japan Atomic Energy Agency)

13:15 *Exploring plant protein structures in food and non-food products with the help of X-ray scattering and other techniques* – Ramune Kuktaite

13:35 *Effect of stabilizers on the mesostructure of cellulose microfibrils studied by small-angle X-ray scattering* - Evgenii Velichko (Delft University of Technology)

15:00 Coffee break

15:30 Dairy

15:30 Keynote: *Neutron and X-ray scattering applied to projects in the dairy industry* - Grethe Vestergaard Jensen (NBI, University of Copenhagen)

16:10 *Calcium caseinate fibrous structure investigated using neutron scattering and tomography* - Bei Tian (Delft University of Technology)

16:35 *Calcium phosphate nanocluster formation by phosphorylated proteins* - Tommy Nylander (Lund University)

17:00 Closing remarks (until 17:30)

Abstracts

1 Methods

1.1 Keynote: Food Science @ ANSTO – The First Ten Years - Elliot Gilbert (ANSTO)

On a daily basis, the most ubiquitous form of soft condensed matter with which we interact is food. Globally, there are major concerns around food, nutrition, bioenergy and the environment transcending borders. There are also significant challenges arising from the increasing food demand governed by a growing population, rapidly changing food preferences and increasing demand for high standards of food quality governed by economic developments, impact of agriculture on the environment, and the effect of climate change.

When designing new food products for the market-place, it is of increasing importance to understand the relationships between the form and functional properties of food constituents, including food structure, nutrition, physiological and its sensory properties. The creation of novel functionalities of active ingredients in complex food systems requires knowledge of not only the structure of native agricultural materials but also the changes in their structure across a wide range of length scales brought about by food processing. It is the inherent complexity of modern food systems that calls for interdisciplinary scientific approaches to be applied.

To tackle many of these issues, ANSTO commenced the ‘Food Science Programme’ in 2005. The programme was conceived as having a focus on neutron scattering but naturally and necessarily utilises ANSTO’s broader infrastructure in X-ray scattering and diffraction, electron microscopy, the National Deuteration Facility and the Australian Synchrotron.

This presentation will cover the first ten years of the 'Food Structure and Dynamics' group at ANSTO and describe several examples including starch, oleogelators, triglycerides, digestion and food processing.

1.2 Challenges for Sustainable Food Processing - Fredrik Innings (Tetra Pak)

There is a heavy pressure, both from the community and from the industry itself, for a more sustainable food production. This can be a reduced energy and water consumption or lower losses in the food chain. Tetra Pak, as an equipment supplier are working hard to help our customers, the food industry, to reach their achieve this. We do this mainly by improving the efficiency in the equipment we deliver and the key to this is knowledge. By understanding the physics and chemistry of the unit operations we deliver, we can optimize them, and in many cases the key to this understanding is performing the right measurements. In this talk we will present a couple of real measurement tasks from the food packaging and processing field that we today don’t know how to handle.

1.3 Current Status of 40m SANS Instrument and SANS-related Research at HANARO - Eunhye Kim (Korea Atomic Energy Research Institute)

Small angle neutron scattering (SANS) is a very effective tool to study the structure of bulk nanoscale materials such as polymer, self-assembled materials, nano-porous materials, nano-magnetic materials, metal or ceramics. Understanding the importance of the SANS instrument in Korea, the 9m SANS instrument was initially installed at the CN beam port of the 30 MW HANARO research reactor in 2001. Since the 9m SANS instrument did not have a cold neutron source in the reactor core and its length is rather short, the beam intensity is fairly low and the Q-range is rather limited. In July 1, 2003, therefore, the HANARO cold neutron research facility project was launched and a state of the art 40m SANS instrument was selected as top-priority instrument. In April 2010, the development of the 40m SANS instrument was successfully finished, and it has been opened to outside users after conducting the beam test and optimization during 7 months. In this presentation, the characteristics (specification, performance and options) of the 40m SANS instrument and materials research results performed using 40m SANS instrument will be presented.

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2 Imaging and Tomography

2.1 *The NEXIM Project* - Robert Feidenhans'l (University of Copenhagen)

The Danish food industry is known for making high quality products. In order to maintain this reputation it is important to keep increasing food safety and quality.

These requirements motivate the collaboration of researchers working with x-ray and computing, members of the food industry, and members of the sensor system industry.

The goal of the project is to improve the detectability of unwanted materials incidentally present in food, development of new modalities for assesment of quality traits in food production, and development of a proof-of-principle of a conveyor belt solution.

The project runs from 2012 until 2016 and is funded by The Danish Council for Strategic Research

2.2 3D Structures of Processed Meat studied by X-ray phase-contrast Tomography - Rikke Miklos (Department of Food Science, UCPH)

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Application of X-ray imaging within food science has so far been limited due to the poor contrast in food products using the conventional X-ray absorption modality. However, the emerging phase-contrast modality is adding a new perspective to the potential of X-ray imaging within food science. Due to the increased sensitivity towards small variations in electron density in soft matter materials, phase-contrast imaging has demonstrated improved contrast for various food products such as processed meat. An overview of the experimental results using X-ray phase contrast imaging to study qualitative and quantitative structural changes related to changes in electron density in meat emulsions [1,2] and heat treated whole meat [3] is presented.

[1] H Einarsdóttir, MS Nielsen, R Miklos, R Lametsch, R Feidenhans'l, R Larsen & BK Ersbøll. Innovative Food Science and Emerging Technologies. (2013).
[2] R Miklos, MS Nielsen, H Einarsdóttir, R Lametsch, AIP Proceedings, ESAFORM 27-29 April, Nantes. (2016)
[3] R Miklos, MS Nielsen, H Einarsdóttir, R Lametsch. Meat Science, 100, p 217-221. (2015)

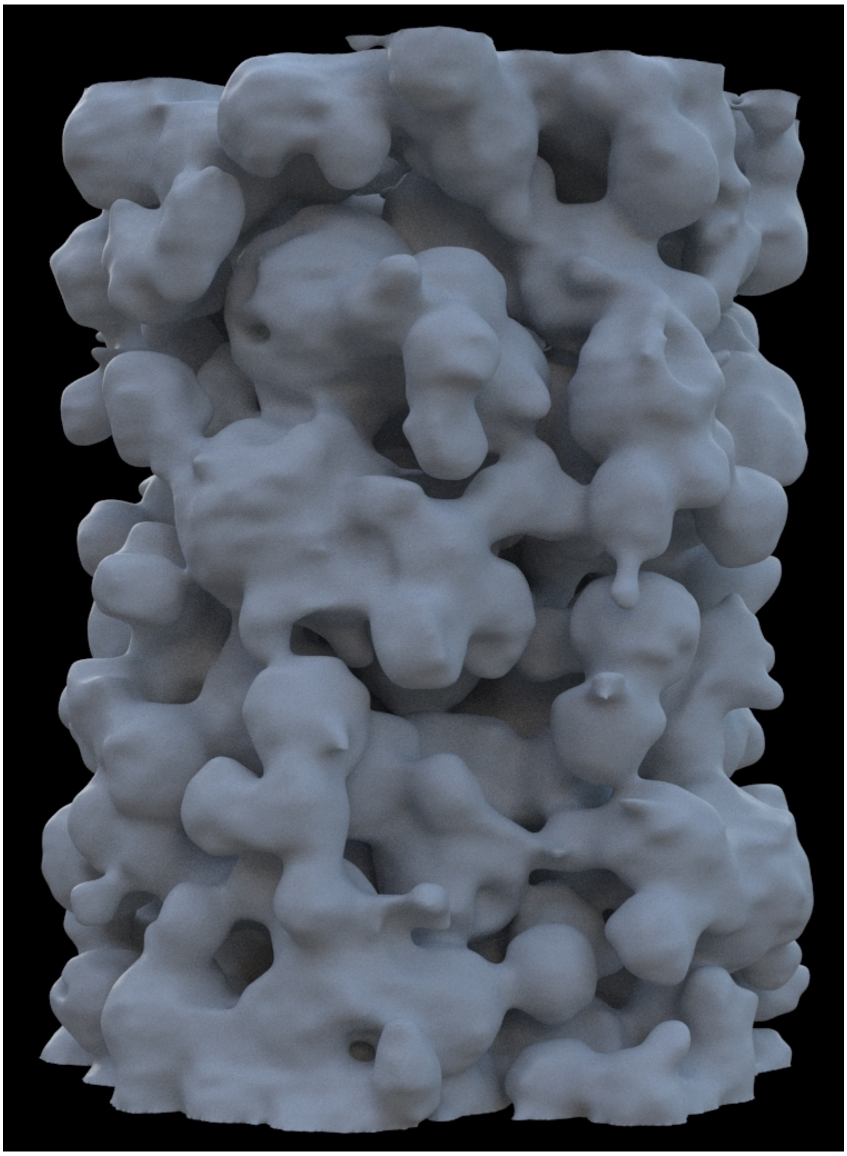
2.3 Revealing the 3D nanostructure of extended colloidal networks in food emulsions using ptychographic X-ray computed tomography - Mikkel Schou Nielsen (Danish Fundamental Metrology A/S, Lyngby, Denmark)

Extended colloidal networks are found in a variety of multi-phase food systems such as butter, chocolate, cream cheese, whipped creams, ice cream, cheese and yogurt. While composed of many of the same components, these systems display a wide range of mechanical and sensorial textural properties. Thus, focus has been to link the macroscopic properties of the food systems to the 3D microstructure of the colloidal networks. However, since the constituting network elements have submicron sizes, a nano-scale 3D imaging technique would be needed which has been missing so far.

In recent years, a range of non-destructive X-ray phase-contrast imaging methods have been developed at synchrotron facilities and applied to study the microstructure of food products. One of the most recent is ptychographic X-ray computed tomography PXCT which is a quantitative 3D nano-imaging technique. Besides offering a spatial resolution up to 16 nm in 3D, PXCT provides the full 3D electron density distribution of the sample.

We present results of quantitative PXCT applied to a palm kernel oil based oil-in-water emulsion. The 3D structure of the extended colloidal network of fat globules was obtained with a resolution of around 300 nm which was limited by the stability of the sample. In addition, the reconstructed electron density values were within 4% of reference values. Through image analysis of the network structure, the fat globule size distribution was computed and compared to previous findings.

The study demonstrates the potential of applying PXCT for food systems. As PXCT is well-suited for diffraction-limited synchrotron sources such as MAX IV, an increasing availability of this technique is expected in coming years.



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2.4 Application of X-ray dark-field imaging for monitoring barley seed germination - Kasper Borg Damkjær (University of Copenhagen, NEXIM)

X-ray transmission and dark-field imaging were used to investigate the imbibition and distribution of water in barley seeds, including structures and structural changes on the micrometer scale during a 48 hour germination period at 19°C.

With the use of dark-field imaging it was possible to observe structural changes in barley seeds during imbibition and the following germination. This was not possible with traditional X-ray transmission.

X-ray dark-field imaging also made it possible to observe and locate stress cracks in the barley endosperm which was also not possible with X-ray transmission.

2.5 Neutron radiography/tomography for visualising and quantifying the novel fruit pulp concentration process "Solar Assisted Pervaporation" - Randi Phinney (Lund University)

Quantitative neutron imaging was used to investigate internal mass transport processes and drying uniformity during "Solar Assisted Pervaporation" (SAP) - a novel process for drying fruit pulps using vapour permeable, liquid water impermeable bags and solar energy. Internal mass transport was visualised and quantified by injecting D2O into a bag of apple purée and monitoring its redistribution over time. The process was found to be diffusion-driven, most likely attributed to the viscous nature of the purée. Juice and purée were found to dry differently inside vertical bags, even though both were distributed homogeneously in the beginning. With the purée bag, local moist spots were observed posing a food safety risk. This first investigation of the compatibility of neutron imaging with SAP has shown that radiography and spatiotemportal tomography are promising techniques for quantifying convective and diffusive transport rates, local drying rates and drying heterogeneity. Future experiments will enable the validation of a multiphysics model of the process, and increase the understanding of the internal mass transport and the food safety risks associated with non-uniform drying.

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2.6 *Layered Surface Detection in Micro-CT Tetra Pak Data* - Vedrana Andersen Dahl (Technical University of Denmark)

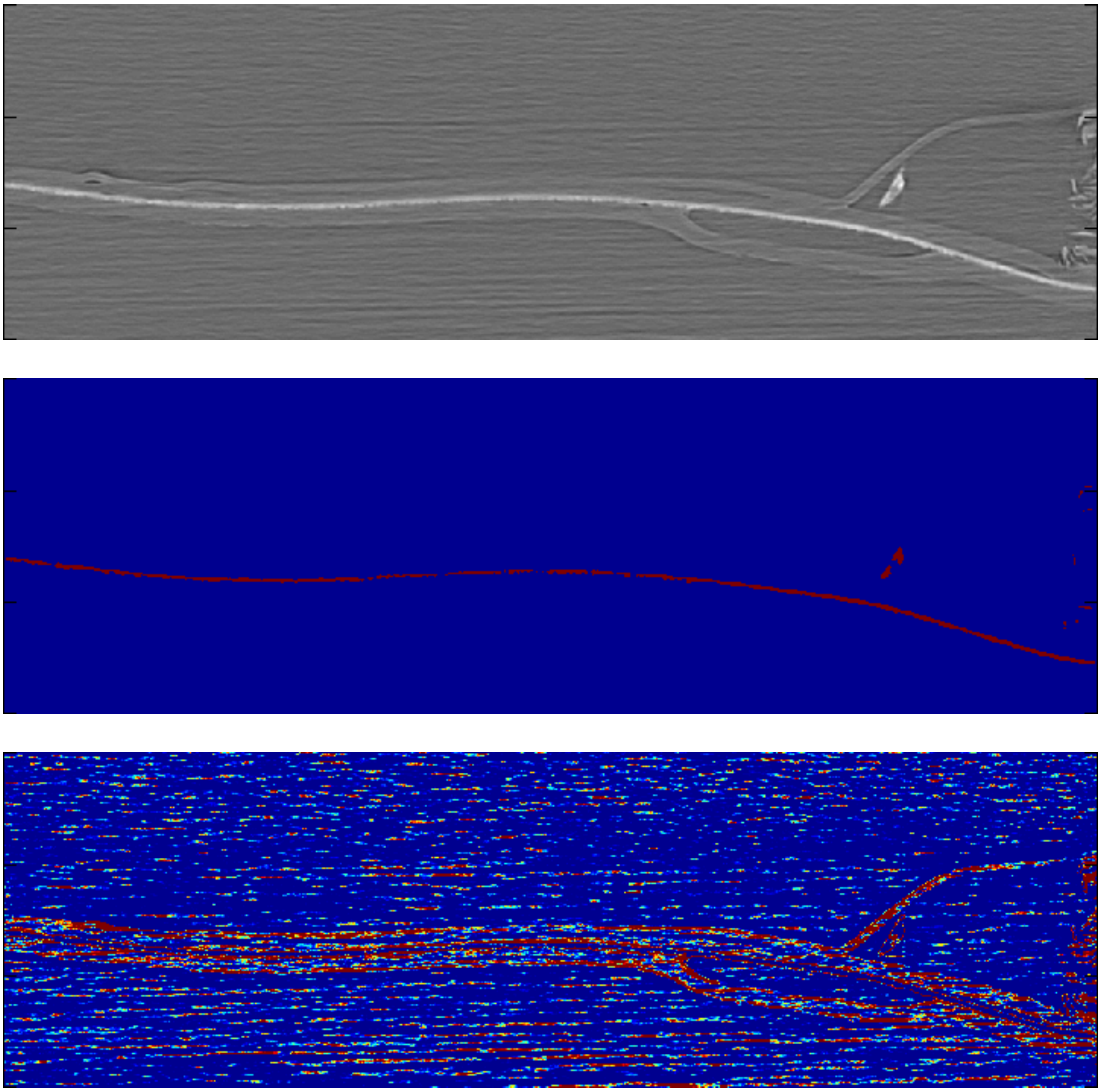
Exact geometry is important for product development and quality control of Tetra Pak carton packages for liquid food products, leading to better performance and lower cost. For packages intended to be used with the drinking straw, the membrane covering the pre-punched straw hole has to hold the liquid content inside the package, while still allowing for an easy opening. Furthermore, wider straw allows for the better flow of the liquid product, but it requires a larger and a more resilient membrane. Product development aims at meeting those opposing objectives, as well as increasing converting production speed and package filling machine speed. Gaining knowledge on the geometry of the straw opening is valuable, since accurate geometry can be used directly as an input to the virtual simulation models for product development.

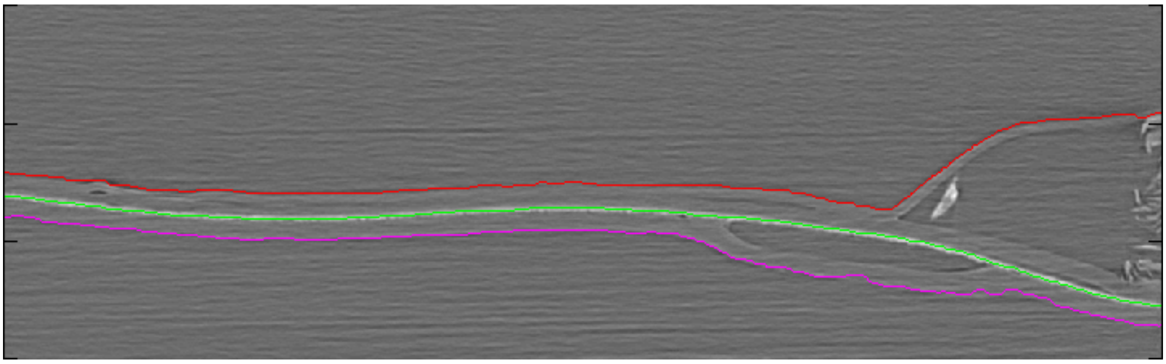
To investigate the possibility of the automated method for extracting the accurate geometry of the straw hole and the membrane, an experiment was conducted at DTU Imaging Industry Portal. The straw hole of Tetra Pak packages is covered by the laminated membrane consisting of an aluminium foil sandwiched between the thin layers of polymer material. The layer of aluminium is 6 µm thick. A sample containing the straw hole and the membrane was scanned at DTU Imaging Industry Portal using a micro CT scanner, with the resulting voxel size of 1.9 µm. Fig. 1 shows a part of the reconstructed volumetric data. The aluminium foil is visible as a bright horizontal line, and small part of the carton is visible on far right.

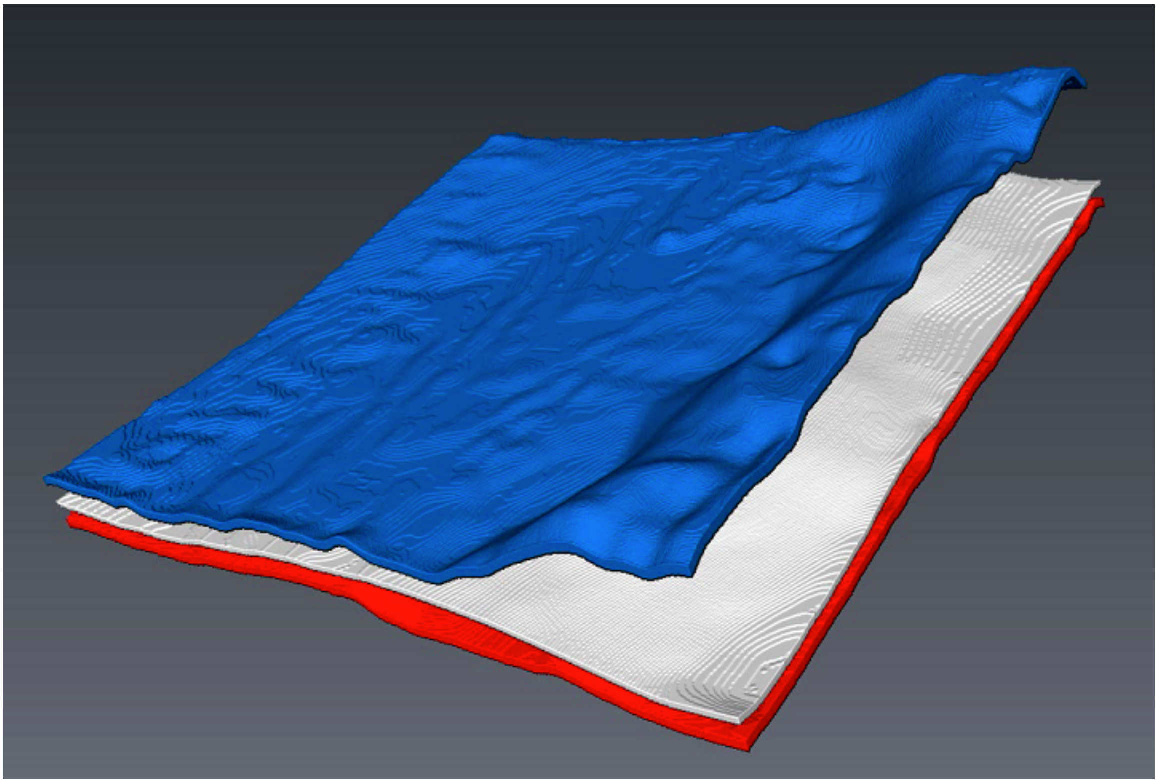
It is easy to detect the aluminium foil in the obtained volumetric data using simple segmentation techniques, as its voxel intensities are much brighter than the surrounding matter. For example, thresholding for the aluminium results with the binary response in Fig. 2. On the other side, detecting the layers of polymer is challenging, at its intensities resemble those of the surrounding air. Estimating polymer intensity distribution from the intensity histogram, and calculating the probability of the voxel belonging to this distribution results with the probabilistic polymer response in Fig. 3. Incorporating edge detection does not fully alleviate the problem, due to the edge-like noise in the background.

To extract the accurate geometry of the membrane, including the polymer layers, we applied a layered net surface approach by Li et al., which incorporates geometric information into the detection model. The geometric information is based on our knowledge of the sample: aluminium and polymer may be modelled as terrain-like, smooth, layered and non-intersecting surfaces. Those geometric constraints significantly reduce the number of acceptable surface detection outcomes. From all the remaining outcomes, we use a graph-cut based search to find the surfaces with optimal response throughout the whole volume. As an initial goal, we set up a model for three surfaces: a surface defining the aluminium foil, a surface defining the upper edge of the upper polymer layer, and a surface defining the lower edge of the lower polymer layer. We used a brightness response when detecting the aluminium layer, and polymer-to-air transition response when detecting two edge layers. See Fig. 4 for results visualized on one slice, and Fig. 5. for visualization of detected surfaces.

The resulting framework demonstrates the feasibility of the automated approach, allowing for the accurate detection of the layered materials comprising the straw hole membrane. To be even more valuable, the method should be coupled with the assessment of the quality of the fit. This is because the geometric constraints will always be met regardless of the data (e.g. the surfaces will be found even when the membrane is punctured).







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Li, Kang, et al. "Optimal surface segmentation in volumetric images-a graph-theoretic approach." Pattern Analysis and Machine Intelligence, IEEE Transactions on 28.1 (2006): 119-134.

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3 Fats, Emulsions and Emulsifiers

3.1 Keynote: Quantification of fat crystallite thickness distributions by NMR and small-angle scattering techniques - John van Duynhoven (Unilever)

Understanding the behaviour of fat crystal dispersions in oil under industrial processing conditions is of considerable industrial interest. Knowledge of the transient structural hierarchy in fat crystal networks under dynamic conditions will impact directly the rational design and engineering of lipid-based food materials with enhanced shelf-life stability and sensorial quality. Moreover, this will provide the necessary means to develop the relationship between processing and the growth of the multi-length scale structure of a fat network. Fat crystal network formation predominantly depends cooling rate and lipid composition. While knowledge on the crystal polymorphism exists, we only have limited understanding how nanoscale fat crystallites promote hierarchical multiscale networks. The average crystallite thickness (ATD) can be derived from the Full Width at Half Maximum (FWHM) of the first order diffraction line by making use of the Scherrer equation. This approach relies on long range order and may be biased towards assessment of thicker plates. Hence we also implemented an NMR method that solely relies to the low molecular mobility in the crystalline phase. First the magnetization of the crystalline phase was selected by means of a double quantum filter subsequently the redistribution of magnetisation toward the mobile and non-crystalline phase was monitored. By fitting a one-dimensional model for spin-diffusion this approach also yielded ATD values and these compared well with those obtain from SAXS and the Scherrer equation. These NMR and SAXS methods could be implemented on lab-based equipment and were applied on model and real-life complex fat blends subjected to different cooling regimes; average crystallite thicknesses as obtained by these methods were in good agreement. Depending on blend composition and cooling regime also a distribution in crystallite thickness can be introduced. The aforementioned approaches do not take such heterogeneity into account, hence we implemented the Bertaut-Warren-Averbach method to derive crystallite thickness distributions (CTD) from SAXS data obtained at the ID02 beamline at ESRF. Thus CTD signatures could be recognized that were specific for blend composition and cooling regime, both in semi- as in full food products.

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3.2 Soybean Oleosomes studied by Small Angle Neutron Scattering - Andrew Jackson (European Spallation Source)

Although oleosomes, the lipid storage organelles of plants, have attracted growing interest during recent years, not all of their structural features are known yet in detail. This is especially true for their stabilizing outer protein layer, mainly composed of oleosins. These are challenging to study in extracted form due to their long hydrophobic domains, which in turn are crucial for the stabilization of the oil droplets. Small angle neutron scattering (SANS) offers the possibility to investigate those structures non-destructively in their native state. Using the contrast variation method in order to enhance the scattering signal from the protein layer, the thickness of this layer has been estimated to be 6-8 nm. In order to study the oleosins temperature stability, SANS maesurements were also performed at elevated temperatures. Up to 80 °C, no significant change in the thickness of the protein layer was observed, indicating the high temperature stability of the oleosomes. Oleosomes encapsulated by pectin via electrostatic deposition have also been studied. An increase in shell thickness of 10 nm due to the depostition of pectin has been found.

3.3 SANS study of the self-aggregation of alkylglycoside surfactants with oligomeric head-groups - Federica Sebastiani (Physical Chemistry Department, University of Lund - CR Competence AB, Lund)

The increased effort to preserve the environment has driven extensive research toward the identification of surfactants that are nontoxic, biodegradable, and synthetized from sustainable resources (1). Alkylglycosides, which have a head-group consisting of one or several sugar moieties, promise to meet these demands. Alkylglycoside surfactants with functionalised oligomeric head group (>3 sugars) have recently proved possible to synthetize by enzymatic means (2,3). This novel class of surfactants has been specifically designed to ensure biocompatibility and controlled biodegradability, and hence lend themselves to applications within the area of in vivo controlled release (e.g. food additives). Our study focused on a surfactant comprising a long alkyl chain, 16 carbons, and a long glucose chain, 8 glucose units, which is referred to as C16G8. Since the functionalities and possible applications of C16G8 can compete with the widely used Polysorbate 80, we investigated thoroughly the self-aggregation mechanism. We characterised the system with several techniques, such as light scattering, both static (SLS) and dynamic (DLS), NMR, SAXS and SANS. The complementary use of neutrons and x-rays was crucial to determine the structure of the aggregates, since the contrast between the glucose chain and the alkyl chain differs when probed with x-rays and neutrons. We will discuss the effect of temperature and concentration on the size and shape of the aggregates and, furthermore, the effect of different anomeric configurations (4). The combination of these techniques allowed us to reveal the features of this novel sugar surfactant and build a fundamental knowledge required for identification and development of applications.

Acknowledgements This work has received funding from the People Programme (Marie Curie Actions) of the European Union's Seventh Framework Programme FP7/2007-2013/ under REA grant agreement n° 606713. The SANS study was allowed by allocations of beam time at the ILL (Grenoble, France).

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4 Nutrition and Health

4.1 Keynote: The fate of nanoparticles in the GI tract - Alan Mackie (Institute of Food Research)

Nanoparticles (NPs) are increasingly being used in a wide range of applications including packaging, functional foods and pharmaceuticals. Indeed there is even evidence that they spontaneously form in the gastrointestinal (GI) tract. Although the range of applications is wide, the final fate of the NPs once consumed is of great interest to all. Regardless of the origin of the nanoscale particles, their properties can be significantly altered by exposure to the changing environment of the GI tract and this can affect the way that they interact with the body. The first place that the orally delivered NPs come into contact with the body is at the GI epithelium and although the cells themselves are not readily amenable to scattering experiments, the cell membranes are. Thus supported lipid bilayers (SLBs) can be used as a model of the cell membrane and the interaction with NPs can be investigated using neutron reflectance.
In this study, a combination of neutron reflectance and QCM-D were used to measure the interactions between a range of NPs and SLBs. Among pristine NPs only bigger carboxylated polystyrene NPs induced a permanent effect on the SLB. The presence of adsorbed proteins levelled the behaviour for all NPs, inducing a weak and non-specific adsorption on the SLB. Under these conditions the more weakly adsorbed soft corona showed more binding that a tightly bound hard corona. This is also broadly in agreement with experiments using in vitro digestion and cellular models and has implications for the fate of ingested NPs, regardless of origin.

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4.2 New Sustainable Technology for Pure Water - an essential ingredient for food - Adrian Rennie (Uppsala University)

Pure water is essential for good health. It is necessary for food preparation and sanitation. Supply of water is still a major challenge and technology that can be used without specialist support in remote and rural areas is still needed in many countries. An extract from the seeds of the *Moringa oleifera* tree that is principally a low molecular mass protein is known to be efficient as a coagulating agent for water purification. A purification process based on traditional methods used in tropical and sub-tropical Africa is attracting interest for wider use as a sustainable, environmentally friendly technology. Flocs have been investigated by small-angle scattering and ultra small-angle scattering [1]. The structure of adsorbed layers at different oxide surfaces such as silica and alumina has been investigated by neutron reflection [2]. The studies have identified that very compact flocs of impurity particles can be formed that are easy to separate. The amount of protein required to cover various different surfaces and the structure of interfacial layers has been determined. These results allow optimisation of the water purification and the process attracts interest at present in a number of countries.

1. M S. Hellsing, *et al*. *Colloids and Surfaces A: Physicochem. Eng. Aspects* **460**, (2014), 460-467
2. H. M. Kwaambwa, M. S. Hellsing and A. R. Rennie *Langmuir* **26**, (2010), 3902-3910.

4.3 Can neutron or x-ray scattering techniques support food scientists to reveal important information on food structures?

- Niklas Lorén (SP Food and Bioscience)

Foods are complex soft materials that consist of many different components. Polysaccharides, proteins, lipids and water are the most common components. The way the different components are spatial distributed, their inherent properties and the interaction between the different components will strongly influence the macroscopic properties of the food. Thus, the food microstructure codes the food properties. The typical length scales of the structure are also very important for thefood properties. For instance, the oil droplet size distribution of mayonnaise at the micrometer level determines much of the mouthfeel. In addition, the stability of the mayonnaise is determined by the protein network at the nanometer level. Food structures are also very hierarchical. Imagine chocolate, there the fat phase consists of TAGs at molecular level. The TAGS assemble into lamellar structures at length scale of tens of nanometers which assemble into nanoplatelets at the length scale of fifth nanometers. These nanoplatelets then assembled into fractal like network structures at the micrometer level. The shelf-life and mechanical properties of the chocolate is determined by the fat crystal structure there different length scales act together to give the macroscopic properties. Thus, nice techniques to reveal the food microstructure at different length scales in both the real and reciprocal space are needed. In this presentation, food microstructure in general will be discussed.

Hydrogels are important as thickeners and stabilizers among others in foods. Hydrogels consist of a continuous gel strand network that is percolating the whole structure. The gel strands are dispersed in a continuous water phase. One important class of hydrogels is carrageenan. The carrageenan concentration, cooling rate, salt concentration, and type of salt are strongly influencing the hydrogel microstructure as seen by transmission electron microscopy1-3 and the mass transport and mechanical properties. The gelation of kappa-carrageenan and some of the challenges that remain to be answered will be discussed. Maybe neutron or x-ray techniques can shine some extra light on the gelation of kappa-carrageenan and other hydrogels?

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5 Lipids

5.1 Keynote: New Insights on the Interaction of Flavonoids with Biomimetic Membranes - Michael Rappolt (School of Food Science and Nutrition, University of Leeds)

After an introduction into global scattering analysis methods for bilayer membrane systems and after some remarks on how one can exploit even poor diffraction data, recent studies on the interaction of flavonoids with different model membranes systems are presented. Flavonoids are of great importance concerning the health benefits of foods and food additives due to their potential anti-oxidative, anti-inflammatory and immune regulatory actions. It is eminent that flavonoid consumption reduces the risk of several chronic diseases, including cancer and diabetes. Interactions of flavonoids with lipids, via their adsorption at the membrane water interface or insertion into the lipid chain region, can alter the lipid bilayers features (e.g. thickness, fluctuations and fluidity), and thus, influence their therapeutic potentials. The overall aim is to understand the structure-function relationship of flavonoids in the biomembrane in order to design functional food with an higher bioavailability profile.

5.2 Lipid sponge phases and nanoparticle dispersions able to entrap large biomolecules - Maria Valldeperas (Lund University)

Nonlamellar lipid liquid crystalline phases have many potential applications, such as for drug delivery, protein encapsulation or crystallization.1-4 Lipid liquid crystalline sponge phase (L3) has not been very much considered in these applications, but have advantages in terms of its capacity of forming large aqueous pores able to encapsulate large bioactive molecules. This is more challenging to obtain with other reverse mesophases, such as reverse cubic (Q2) and reverse hexagonal (H2) phases. Basically, all of these reversed mesophases in excess aqueous solutions can be dispersed into colloidal stable particles dispersions.1,5,6 Here, we report a novel lipid system able to form highly swollen L3 phases, both in bulk and as nanoparticle dispersion. This system has been characterized by Dynamic Light Scattering (DLS), Cryo-Transmission Electron Microscopy (Cryo-TEM) and Small Angle X-ray Scattering (SAXS) to determine the size and shape of disperse particles as well as the liquid crystalline structure as a function of lipid composition. Water pores up to 13 nm were achieved, making the system suitable for entrapment of bioactive macromolecules such as proteins. Preliminary data on protein inclusion will also be presented.

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5.3 The Lipid Binding Interactions of Indolines and Thionins : Cationic Cysteine Rich Antimicrobial Proteins from Cereal Seeds - Luke Clifton (ISIS Spallation Neutron and Muon Source)

Indolines and thionins are cationic, amphiphillic, antimicrobial proteins found in a series of cereal species. The antimicrobial activity of both protein families is thought to be related to their ability to disrupt pathogen membrane function via direct interactions with the lipid component of the membrane. We have been examining the lipid binding activity of a series of indoline and thionin types and comparing the molecular solution structuring to the nature of the hydrophobic surface of the proteins.

The hydrophobic region of the puroindolines is a unique Tryptophan rich domain which is fully conserved on the major isoform puroindoline-a and partially conserved on the minor isoform puroindoline-b. We have shown that puroindoline-a uniquely forms highly prolate protein micelles in solution. This self assembly is likely driven by the amphiphillic nature of the molecule and in particular the hydrophobicity of the Trp rich domain. During its lipid binding interactions with phospholipid monolayers the protein is unable to penetrate the lipid acyl chain region on condense phase anionic phospholipid monolayers probably due to the interactions between the tryptophans and the lipid carbonyl groups.

Conversely, all thionin types examined showed high penetration into condense phase DPPG monolayers, as well as removing lipids from the interface during the protein adsorption process. When comparing the lipid binding interactions of α1 and α2-purothionin, two highly related protein which are similar in charge but differ in their relative hydrophobicity (with α2-Pth being more hydrophobic than α1-Pth), we observed a correlation between the ability of these protein to remove lipids from the interface, with the more hydrophobic protein able to remove more lipid from the interface.

These findings have implication for our understanding of the role of small cationic amphiphillic proteins in defending the seed endosperm from bacterial and fungal pathogens and in the indolines role in wheat endosperm hardness.

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6 Biopolymers

6.1 Keynote: Protein gels from the inside with neutron scattering - Wim Bouwman (Delft University of Technology)

Systems of practical relevance to the food industry are often hard to investigate non-invasively. This is caused by the fact that most food emulsions are opaque and soft materials. The relevant length scales are often micrometers. Spin-echo small-angle scattering (SESANS) operates at these length scales and benefits from the high penetrating power of neutrons. SESANS yields directly the scattering length density correlation function, which facilitates visual data-analysis [1].
In the presentation the possibilities of SESANS will be illustrated with studies on the structure of cross linked casein micelles gels [2], the water holding capacity of ovalbumin gels [3] and the effect of the presence of gelatine on whey protein gels [4].

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6.2 Glass transition and hydraion of food protein studied by inelastic neutron scattering - Hiroshi Nakagawa (Japan Atomic Energy Agency)

Hydration and vitrification is an essential factor determining the physical property and quality of food. Inelastic neutron scattering is a promising experimental method for analysis of water mobility and glass transition of food materials. Neutron has high permeability in materials, and can observe non-destructively the molecular dynamics of materials involved in food. Molecular Dynamics simulation gives the information on the molecular structure and dynamics with atomic scale. It was found that the hydration level dependence of the onset of protein glass transition is correlated with the percolation transition of hydration water. In addition, the hydration water dynamics change significantly at the transition temperature. The percolation of hydration water induces the protein glass transition.

6.3 Exploring plant protein structures in food and non-food products with the help of X-ray scattering and other techniques - Ramune Kuktaite (Dr)

For production of healthy and environmentally safe products the use of plant proteins is recognized as of highest importance. Plant proteins are an important group of molecules, offering a number of molecular interactions during processing and play a central role in the manufacturing of protein based food/non-food products. In this context, the plant protein structure and function are closely related, and a full control of them would guarantee the successful manufacturing of plant protein products. More understanding is needed on how plant protein structure and function are developed during processing of targeted protein products for innovative protein foods and proteins suitable for packaging applications.

Here we present a brief overview of the nano- and microstructure dynamics of plant proteins in various protein products by the use of X-ray scattering (SAXS and WAXS) in combination with other microstructural methods (SEM, TEM and X-ray tomography). The impact of additives (glycerol, cross-linking enzyme) and processing conditions (temperature and composition of the blend) during processing will be discussed. When studying wheat gliadin and glutenin films with different amounts of glycerol, we found that the nano-structural morphology of gliadin-glycerol films was greatly impacted by 20% and the higher amounts of glycerol (1). For the gliadin-enzyme food products, we found that the gliadin protein microstructure can be greatly steered by the use of specific additives and the type of enzyme.

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6.4 Effect of stabilizers on the mesostructure of cellulose microfibrils studied by small-angle X-ray scattering - Evgenii Velichko (Delft University of Technology)

Cellulose microfibril (CMF) dispersions find many applications in food industry [1]. They are typically prepared by defibrillation of plant cell wall material under high shear [2]. Aqueous CMF dispersions have to be protected from agglomeration by a stabilizer, typically soluble polymer, such as pectin, which coexists with cellulose in plant cell walls. In industrial processing, CMFs undergo series of processing steps, most of which apply shear-stress on CMFs. Under certain shear-rates plant CMFs start to agglomerate, despite the presence of stabilizers [3]. This behaviour could be mimicked in dispersions of bacterial cellulose (BC) (already produced in microfibrillar form) with water-soluble CMC [4]. The mesostructure of CMF dispersions and it's changes with addition of stabilizer is poorly understood so far. In order to understand effect of stabilizer on CMF, two systems were chosen for investigation: citric fibrils with pectin (CF-P) as a natural heterogeneous system and bacterial cellulose with CMC as a better defined model system. Series of both systems with different component ratios were prepared. All prepared samples were studied by small angle X-ray scattering, allowing to see characteristic features in lengths range from about few nm up to few μm. Dependencies of mesostructure on concentration of stabilizer have been discovered for both systems. Peculiar similarities and mesostructural differences between both types of CMF dispersions are revealed.

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7 Dairy

7.1 Keynote: Neutron and X-ray scattering applied to projects in the dairy industry - Grethe Vestergaard Jensen (NBI, University of Copenhagen)

Neutron scattering holds a lot of relatively unexploited potential for food science, including industrial research and development. Examples of applications from the dairy industry will be presented, showing how small-angle scattering of neutrons and X-rays (SANS and SAXS) can be applied to study milk protein in the form of casein micelles.
The internal structure of casein micelles, re-suspended from a powder isolate, is studied for samples supplied by Arla Food Ingredients Group P/S. The sample processing affects the structure of the micelles, and thereby also their functionality, related to the various potential applications of the powder.
The self-association of casein micelles upon acidification is investigated in collaboration with DuPont Nutrition Biosciences ApS. To avoid macroscopic aggregation, and the associated unpleasant mouth-feel, the casein micelles can be stabilised with pectin, and the structural effect of different pectins is determined.

The studies were conducted as part of the NXUS initiative (Neutron and X-ray User Support). NXUS is jointly funded by the University of Copenhagen and the Capital Region of Denmark, and is one of the projects that have been initiated following the heavy Danish investments in the European Spallation Source (ESS) and the synchrotron facility MAX IV in Lund. The aim is to investigate the potential and barriers for increased industrial and academic use of this type of facilities, and to gain experience on which role specialised university groups might play in this agenda. A series of case studies have been conducted with different companies, ranging from pharma- and biotech to manufacturers of paint and catalysts, serving as illustrative examples. The work now continues in the industry portal LINX, which is supported by Innovation Fund Denmark, includes partners from industry and universities, and covers more experimental techniques.

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7.2 Calcium caseinate fibrous structure investigated using neutron scattering and tomography - Bei Tian (Delft University of Technology)

Mild heat and shear to a 30% calcium caseinate and transglutaminase (E:S = 1:20) results in a material with distinctive fibrous structure that is arranged in a hierarchical order. Such a material can form the basis for a next generation meat analogue. It is important to understand this structuring mechanism to find the optimal ingredient ratios, process conditions and to maintain good quality control. Neutrons can be a powerful tool to study the structuring mechanism of such systems, not only because it is non-invasive but also because it can cover a broad range of length scales. Here, two neutron techniques: Spin Echo Small Angle Neutron Scattering (SESANS) and neutron tomography have been used to understand the micro- and meso-structures of samples with structure that range from dispersion to gel state. Models are chosen to describe the sample structure and to extract physically relevant parameters.

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7.3 Calcium phosphate nanocluster formation by phosphorylated proteins - Tommy Nylander (Lund University)

Milk and some milk products are almost unique among foods in containing high concentrations of calcium but in spite of this have a remarkably high stability during storage and processing. The main reason for the high stability is that most of the calcium is in the form of nanoclusters of an amorphous form of calcium phosphate sequestered by casein. Recent research from our group has shown that similarly stable nanoclusters can be formed and sequestered by some non-casein phosphoproteins. We have focused our attention on osteopontin because it is widely distributed in vertebrate tissues and plays key roles in the control of biomineralisation. The osteopontin nanoclusters have been characterized by elastic and inelastic scattering, by neutron diffraction and complementary X-ray methods. Some recent results using neutron small-angle scattering and spectroscopy that probe the formed structures and molecular mobility will be presented. Results have been compared with casein nanoclusters to provide an insight into the structural and compositional requirements and other physico-chemical principles needed for stable biofluids and foods. This new knowledge can be applied in the design of stable dairy products as well as to produce other types of stable, high-calcium foods, and novel materials for use in nanotechnology and medicine.

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