LUND INSTITUTE OF ADVANCED NEUTRON AND X-RAY SCIENCE LINXS

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15th Nordic Workshop on Scattering from Soft Matter

17-18 January, 2018
Lund, Sweden

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The 15th Nordic Workshop on Scattering from Soft Matter will be held in Lund, Sweden on January 17 to 18, 2018.
There will be a full day program starting with lunch the first day and ending after lunch on the second day. The program includes invited and contributed talks, a poster session and a workshop dinner.

Confirmed keynote speakers include:

Prof. Jan Skov Pedersen, Department of Chemistry and Interdisciplinary Nanoscience Center (iNANO), Aarhus University, Denmark
Dr. Hanna Isaksson, Biomedical Engineering, LU, Sweden
Dr. Felix Roosen-Runge, Physical Chemistry, LU, Sweden
Prof. Dr. Christian Gutt, Department of Physics, X-ray Physics, University of Siegen, Germany

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Andrew Jackson (European Spallation Source & Lund University)
Zsuzsa Helyes (European Spallation Source)

Programme

Wednesday, 17 January 2018

Medicon Village, Auditorium

12:00 Registration and Lunch

13:00 Opening of the Workshop and Welcome

13:10 Overview of LINXS – Prof. Peter Schurtenberger, Lund University

13:30 - 15:15 Session 1

13:30 Keynote: Low radiation dose XPCS for dynamics in soft matter and biologial materials – Prof. Dr. Christian Gutt (University of Siegen)

14:00 Structrural characterization of cellulose nanocrystals using SAXS – Dr. Heike Ehmann (Anton Paar)

14:15 Short-time self-dynamics of immunoglobulin under bio- mimicking crowding conditions – Dr. Marco Grimaldo (ILL)

14:35 Interactions between Anionic Surfactants and Polymeric Micelles: stability and solubilisation kinetics – Synne Myhre (University of Oslo)

14:55 End of Cooperativity: Chain Exchange Kinetics in Mixed Polymeric Micelles with Partially Crystalline Cores – Nico König (Forschungszentrum Jülich / University of Oslo)

15:15 Coffee Break

15:45 - 17:45 Session 2

15:45 Keynote: Dynamics of Proteins in Solution Studied by Quasi-Elastic Neutron Scattering – Felix Roosen-Runge (Lund University)

16:15 High throughput biological solution SAXS instrumentation for state-of-the-art structural biology research and validation. – Søren Skou (Xenocs)

16:30 Probing the structural dynamics of photoreceptor proteins by time-resolved X-ray solution scattering – Dr. Oskar Berntsson (University of Gothenburg)

16:50 Probing the interaction between antimicrobial peptides and model biomembranes using small angle scattering techniques – Josefine Eilsø Nielsen (University of Oslo)

17:10 Surface and colloid properties of oligomeric alkylglycosides – Johan Larsson (Lund University)

17:30 Beamline Updates

17:45 Poster Session

19:30 Dinner

Thursday, 18 January 2018

08:00 Coffee

08:30 – 10:00 Session 3

08:30 Keynote: Scattering methods can unravel nano-structural changes in musculoskeletal tissues under loading – Hanna Isaksson (Lund University)

09:00 Structure Determination by SAXS in Protein Biophysics and Structural Biology – Jeppe Lyngsø (Århus University)

09:20 The Interaction of Perfluoroalkyl Substances with Mineral Surfaces and Biological Membranes – Shirin Nouhi (Uppsala University)

09:40 Investigating the mesoscale of fibril hydrogels – Dr. Christina Efthymiou (Uppsala University)

10:00 Coffee Break

10:40 - 10:30 Session 4

10:40 Keynote: Refolding of SDS-unfolded proteins by non-ionic surfactants: Equilibrium and kinetics – Jan Skov Pedersen (Århus University)

11:00 Liquid-Metal-Jet X-ray Source for In-situ SAXS studies in the Home Laboratory – Shichao Hu (Excillum)

11:15 Time-resolved SAXS reveals ionic liquid interaction with model membranes – Dr. Inkeri Kontro (University of Helsinki)

11:35 Characterization of liposomal formulations to treat Fabry Disease – Jannik Pedersen (Århus University)

11:55 Inverse- and real-space scattering of aqueous diblock copolymer micelles – Dr. Gregory Smith (University of Copenhagen)

12:15 Sum up and end of workshop

12:30 Lunch at Inspira

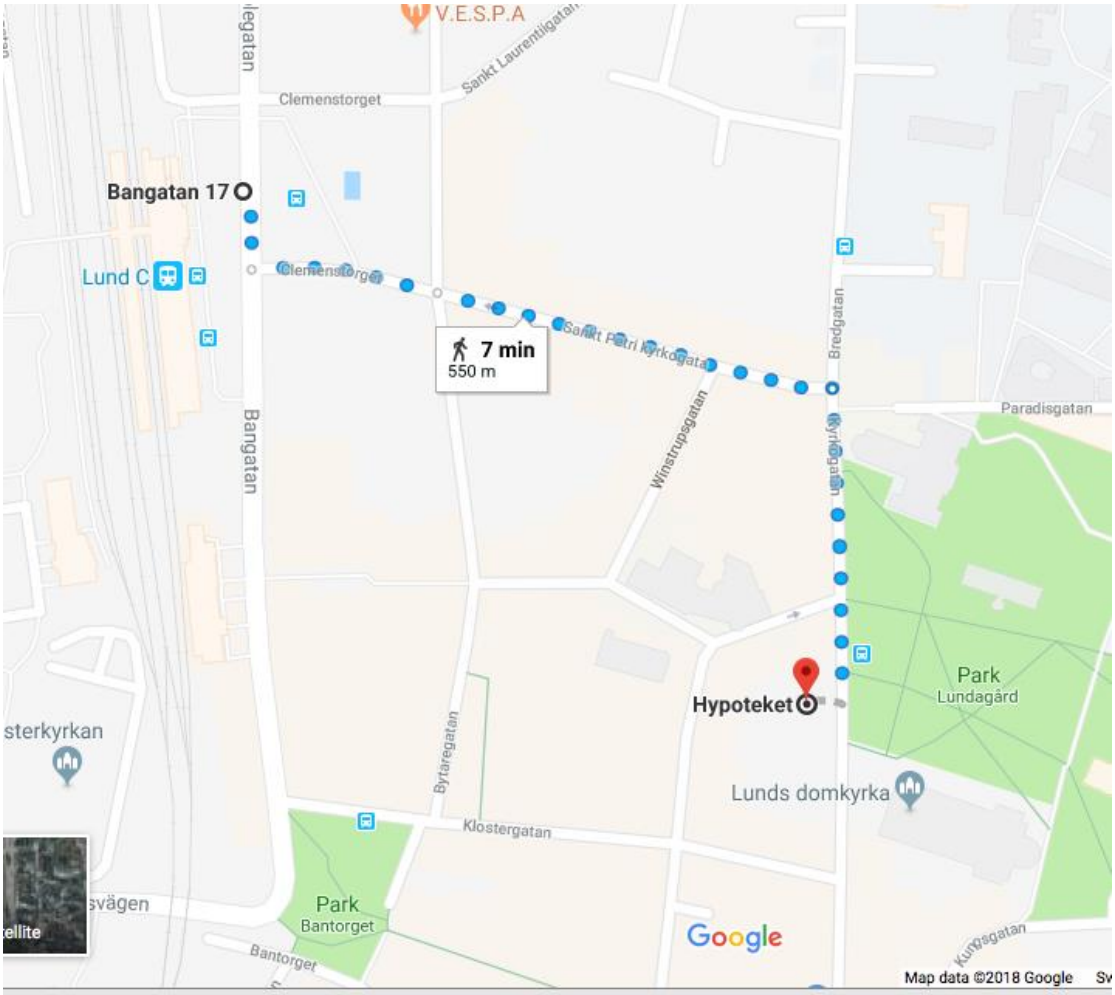
Dinner Venue

The conference dinner will be held on Wednesday, 17 January at 19:30 at Hypotek Våningen in downtown Lund.

Address:
Hypotek
Kyrkogatan 13
222 22 Lund

How to get there

Take bus 1 (in the direction of Klostergården) or 6 (in the direction Botulfsplatsen) from the bus stop called **Sparta** towards the city.
Get off at the stop called **Clemenstorget**.
From there, it Is a short 7 minute walk to the restaurant. Please see the map below.



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1.1 **Low radiation dose XPCS for dynamics in soft matter and biological materials –** Prof. Dr. Christian Gutt (University of Siegen)

X-ray radiation damage provides a serious bottle neck for investigating µs to s dynamics on nanometer length scales employing X-ray photon correlation spectroscopy. This limitation hinders the investigation of real time dynamics in most soft matter and biological materials which can tolerate only X-ray doses of kGy and below. Here, we show that this bottleneck can be overcome by low dose serial X-ray speckle visibility spectroscopy. The concept consists in spreading the dose needed for a correlation function over the entire sample volume by measuring at every spot on the sample the visibility of a speckle pattern as a function of exposure time. Mitigating the absorbed dose in this way is done at the expense of signal strength; the collected speckle patterns are sparse containing signal strength of 10-2 photons per pixel and possibly even less. We show that the speckle visibility correlation function can nevertheless be extracted by proper assignment of photon probabilities using a sufficiently large number of images. Employing X-ray doses of 640 Gy to 8.5 kGy and analyzing the sparse speckle patterns we follow as an example the slow nanoscale dynamics of an ionic liquid (IL) at the glass transition. Our method is especially relevant for the upcoming diffraction limited storage rings providing a two orders of magnitude increase in coherent X-ray flux.

1.2 Structrural characterization of cellulose nanocrystals using SAXS – Dr. Heike Ehmann (Anton Paar)

Cellulose is the main building block of trees and plants and the most abundant biopolymer in the world. It consists of highly ordered domains (nano crystals) and amorphous regions. These cellulose nanocrystals (CNC) have received significant interest due to their mechanical, optical, chemical, and rheological properties. CNC primarily obtained from naturally occurring cellulose fibers are biodegradable and renewable in nature and hence they serve as a sustainable and environmentally friendly material for most applications. These nanocrystals are basically hydrophilic in nature; however, they can be surface functionalized to meet various challenging requirements. The focus in this talk will be the structural characterization of CNC in different environments with small angle X-ray scattering (SAXS). It is employed to characterize the structure and shape of those crystallites directly after acid hydrolysis. Further, the rod-like crystallites are deposited and dried on different substrates and analysed using grazing incidence small angle X-ray scattering (GISAXS) and grazing incidence X-ray diffraction (GIXD).

Co-authors:
Dr. KEILBACH, Andreas (Anton Paar)
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1.3 Short-time self-dynamics of immunoglobulin under bio-mimicking crowding conditions – Dr. Marco Grimaldo (ILL)

Approximately 10-40% of the intra- and extracellular fluids of living organisms are occupied by macromolecules such as proteins. This macromolecular crowding condition was shown to influence reaction rates, and to lead to anomalous diffusion. The study of protein diffusion in such a crowded environment is, generally, not an easy task. Nevertheless, neutron backscattering (NBS) is well suited for measurements of the short-time self-diffusion of proteins in highly concentrated aqueous (D_2O) solutions [1-4]. We present a NBS study on the pico- to nanosecond self-diffusion of the antibody proteins immunoglobulins (Ig) in aqueous environment. To systematically investigate the effect of macromolecular crowding on protein dynamics we vary the concentration of cellular lysate, mimicking a cellular environment. The dynamics of Ig in lysate is then compared with that of Ig in pure (heavy) water as a function of its own concentration (self-crowding) [2]. Despite the high polydispersity and the not easily predictable variance in lysate composition, the measured diffusion of Ig as a function of the overall volume fraction are in rather good agreement with those of Ig in the self-crowded environment at comparable volume fraction, suggesting a crucial role of hydrodynamic interactions and hence, in principle, the applicability of colloidal theories to model the protein short-time diffusion even in a cell-like environment.

[1] Roosen-Runge F., Hennig M., Zhang F., Jacobs R.M.J., Sztucki M., Schober H., Seydel T., and Schreiber F. PNAS 108.29 (2011): 11815 [2] Grimaldo M., Roosen-Runge F., Zhang F., Seydel T., Schreiber F. JPCB 118.25 (2014): 7203. [3] Grimaldo M., Roosen-Runge F., Hennig M., Zanini F., Zhang F., Zamponi, M., Jalarvo N., Schreiber F., Seydel T. JPCL 6.13 (2015): 2577. [4] Braun M.K., Grimaldo M., Roosen-Runge F., Hoffmann I., Czakkel O., Sztucki M., Zhang F., Schreiber F., and Seydel T., JPCL 8.12 (2017): 2590.

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1.4 Interactions between Anionic Surfactants and Polymeric Micelles: stability and solubilisation kinetics – Synne Myhre (University of Oslo)

The kinetic processes involved in mixtures of surfactants and block copolymer micelles are not well understood. However, it is commonly known that surfactants exhibit rather fast equilibration kinetics, in the order of micro- to milliseconds, while polymers are much slower, in the order of minutes to months. In this contribution, we will present a study of the stability and solubilization kinetics of block copolymers micelles upon addition of sodium dodecyl sulphate (SDS) using small angle X-ray scattering (SAXS). We compare the ability of the surfactant to dissolve and form mixed micelles with two amphiphilic polymers; poly(ethylene propylene)-poly(ethylene oxide) (PEP-PEO) and end-capped PEO (C28-PEO). While the kinetics of C28PEO occurs on time scales on the order of minutes-hours on ambient temperatures, that of PEP1-PEO20 is known to be frozen on practical time scales. Addition of SDS to PEP1-PEO20 shows close to no change, even after extended period of time. However, upon addition of SDS to C28PEO5 we observe a fast dissolution and formation of mixed micelles, where the kinetics is seen to accelerate with the amount of added surfactant.

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1.5 End of Cooperativity: Chain Exchange Kinetics in Mixed Polymeric Micelles with Partially Crystalline Cores – Nico König (Forschungszentrum Jülich / University of Oslo)

Here we present a kinetic study on the chain exchange in mixed polymeric micelles containing partially crystalline cores. We are specifically interested in understanding how cooperative phenomena such as crystallization and melting affect the dynamics of self-assembled systems. As a model system we use n-alkyl-PEO (**CnH**2**n**+1−**O**−(**CH**2−**CH**2−**O**)100**H**) with a molecular weight of roughly 5 kg/mol. In water these molecules form star-like micelles with a strongly segregated alkane core that partially crystallizes. This creates an additional energy barrier that needs to be overcome during chain expulsion.[1] We employ time-resolved small-angle neutron scattering in combination with the kinetic zero-average contrast technique to track the exchange kinetics.[2]
We investigated mixtures of C28-PEO and C22-PEO and determined the respective melting enthalpies using differential scanning calorimetry (DSC) which was quantitatively compared to the kinetic data obtained from TR-SANS. We found that the core crystallization occurs cooperatively while the intermicellar chain exchange processes of C28-PEO and C22-PEO are virtually decoupled. Nevertheless the cooperative crystallization affects the separate exchange kinetics of both species.

[1] Zinn, T., Willner, L., Pipich, V., Richter, D. and Lund, R. Effect of Core Crystallization and Conformational Entropy on the Molecular Exchange Kinetics of Polymeric Micelles. ACS Macro Letters 4, 651-655, doi:10.1021/acsmacrolett.5b00197 (2015).
[2] Willner, L., Poppe, A., Allgaier, J., Monkenbusch, M. and Richter, D. Time-resolved SANS for the determination of unimer exchange kinetics in block copolymer micelles. Europhys. Lett. 55, 667-673, doi:DOI 10.1209/epl/i2001-00467-y (2001).

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1.6 Keynote: Dynamics of Proteins in Solution Studied by Quasi-Elastic Neutron Scattering – Felix Roosen-Runge (Lund University)

Information on protein dynamics is of central importance for the understanding how biological function is effectuated by individual proteins as well as by well-adjusted interaction cascades within the crowded cytoplasm. Protein dynamics comprises a hierarchy of processes ranging from fluctuations of side chains and the backbone over interdomain motions to self-diffusion of the entire macromolecule and collective and cage diffusion characterizing the structural relaxation in crowded protein solutions. The broad distribution of time scales from pico- to microseconds, and the variety of dynamical processes including simple as well as confined, anomalous diffusion renders investigating protein dynamics a challenging research field. In this context, quasi-elastic neutron scattering (QENS) provides unique information on both the nature of the underlying dynamical process and the related geometry of dynamical confinement. Neutron backscattering (NBS) and neutron spin echo (NSE) spectroscopy have proven particularly relevant for proteins in solutions, as their instrumental time scales around nanoseconds allow to access simultaneously global and internal dynamics. After a brief overview on the key characteristics of QENS techniques, the scientific potential of QENS for protein dynamics will be examplified with two recent case studies. First, the changes of hierarchical protein dynamics upon thermal denaturation have been studied by both real-time monitoring and an additional detailed characterization of selected states [1,2]. Interestingly, while global dynamics are irreversibly arrested after denaturation, local internal dynamics change reversibly, suggesting that localized internal dynamics are mainly affected by basic physicochemical properties. Second, scenarios of dynamical arrest have been examined in solutions of α, β and γ crystallins as model systems for the eye lens with potential implications for the understanding of cataract and presbyopia. While α crystallin solutions behave similar to hard-sphere systems with a repulsive glass transition at high volume fraction, γB crystallin experiences a dramatic slowing down of cage diffusion already at comparably low volume fractions, suggesting an dynamical arrest driven by weak anisotropic attractions [3,4].

[1] M Grimaldo, F Roosen-Runge, et al. Phys. Chem. Chem. Phys. (2015) 17, 4645-4655 [2] M Hennig, F Roosen-Runge, et al. Soft Matter (2012) 8, 1628-1633 [3] S. Bucciarelli, J.S. Myung, et al. Sci. Adv. (2016) 2, e1601432 [4] S. Bucciarelli, L. Casal-Dujat, et al. J. Phys. Chem. Lett. (2015) 6, 4470–4474

1.7 High throughput biological solution SAXS instrumentation for state-of-the-art structural biology research and validation – Søren Skou (Xenocs)

Low volume samples, high throughput capabilities and easily reconfigurable instrument parameters for biological small angle x-ray scattering have previously been reserved for measurements at state-of-the-art synchrotron beamlines.
With the introduction of the dedicated solution SAXS instrument, the BioXolver, Xenocs is not only moving the sample handling technology previously only seen at synchrotrons into the standard laboratory, but also providing it and the instrument itself with a level of automation that empower users at any skill level to obtain the best data for their particular sample without compromise.
The instrument has been developed to be a truly easy to use and stable workhorse for samples in solution. By integrating computer vision technology, sample volumes down to 5 uL are possible and the in-line UV/VIS absorption measurements facilitate concentration estimation on the exposed sample. Automated sample loading, sample cell cleaning and drying is done with a high precision pipetting robot that also ensures gentle transport from the 2x96 well tray sample containers to the sample cell.
No compromise is made on data quality as the detector is fully in vacuum, ensuring the lowest possible background. Furthermore, using a motorized detector stage and motorized scatterless slits, the sample to detector distance and flux can be automatically optimized to fit a large variety of protein complex sizes. Data reduction and analysis can also be automated and done using the open source software RAW that also includes integration and compatibility with the advanced software suite ATSAS from the EMBL.
Built on the platform of an already successful instrument design, the BioXolver is an excellent choice for stability and data quality.

1.8 Probing the structural dynamics of photoreceptor proteins by time-resolved X-ray solution scattering – Dr. Oskar Berntsson (University of Gothenburg)

The function of proteins is closely linked to their structure. Protein structures, however, are not static but rather dynamic and increased understanding of these structural dynamics is crucial for increased understanding of proteins. We have employed time-resolved X-ray solution scattering as a direct structural probe to study the signal transduction in different photoreceptor proteins. We have investigated members of the red-light sensing phytochromes, the blue light sensing cryptochromes and proteins with blue light sensing light-oxygen-voltage domains. Coupled with molecular dynamics simulations these investigations have revealed new information on how chemical changes at the light absorbing chromophore affects the global structure of the photoreceptor, in order to relay the signal further.

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1.9 Probing the interaction between antimicrobial peptides and model biomembranes using small angle scattering techniques – Josefine Eilsø Nielsen (University of Oslo)

Antibiotic resistance is one of the biggest threats to global health, according to the world health organization. Antimicrobial peptides (AMPs) is a group of molecules that are a natural part of the human immune system, shown to have effect against a broad spectrum of pathogens including both gram positive and gram negative bacteria.1 AMPs seem to be able to evade much of the bacterial resistance mechanisms, and are therefore promising candidates for future antibiotics. Instead of blocking specific biochemical pathways as most available antibiotic agents today, AMPs act physically on the cytoplasmic membrane itself. The precise microscopic mechanism for the perturbation of the membrane is not fully clear but several theories has been suggested including membrane deformation and pore forming which could lead to changes in the lipid dynamics, lateral and transversal composition and proton/ion transfer.2

Here we have used state of the art neutron and x-ray scattering techniques to investigate the microscopic mechanism of action of antimicrobial peptides with biomembranes as models for human and bacterial cell membranes. SAXS measurements on a model peptide, Indolicidin together with DMPC-DMPG-DMPE-PEG vesicles (with increasing amount of negatively charged DMPG) has shown that Indolicidin interacts with the model cell membrane causing a change in the contrast of the bilayer. Based on analysis of the results it seems that the peptide is situated at the interface between the lipid head group and the tail in the outer leaflet in the bilayer without significantly perturbing the structure of the bilayer. This is further supported by DSC where a shift and broadening of the melting lipid temperature upon addition of the peptide is observed. This indicates an associated disordering of the packing of the lipid tails in the bilayer of the vesicles. QCM-D experiments further confirms the disruption of the bilayer but also suggest removal of lipids upon flushing peptide solution over the bilayer surface. Preliminary SANS results do not indicate change in the lateral distribution of lipids which has been suggested as a possible mechanism on3).

When combining the results from scattering methods, together with other complimentary techniques, we gain profound biophysical understanding of these systems. This knowledge can be used in the development of new antibiotics for the future based on antimicrobial peptides designed specifically for the task.

1. Fjell, C. D.; Hiss, J. A.; Hancock, R. E. W.; Schneider, G., Nat. Rev. Drug Discov. 2011, 11 (1), 37-51.
2. Nguyen, L. T.; Haney, E. F.; Vogel, H. J., Trends in biotechnology 2011, 29 (9), 464-472.
3. Epand, R. M.; Epand, R. F., Biochimica et Biophysica Acta (BBA)-Biomembranes 2009, 1788 (1), 289-294.

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Prof. LUND, Reidar (Department of Chemistry, University of Oslo)

1.10 Surface and colloid properties of oligomeric alkylglycosides – Johan Larsson (Lund University)

The increasing demand for environmentally friendly surfactants has resulted in a comprehensive research to identify surfactants that are biodegradable, non-toxic and produced from sustainable raw materials. In this respect, alkylglycosides with one or more sugar molecules, have shown not only promising surface properties but also favourable characteristics regarding sustainability, especially those with an oligomeric head group. However, the self-assembly and interfacial behaviour of oligomeric sugar surfactants are not very well understood. The aim with this study is therefore to get a deeper understanding of oligomeric alkylglycoside behaviour at interfaces and in solution in relation to their structure. The properties of different alkylglycosides, with focus on hexadecyl-β-D-maltopyranoside (C16G2) and a polydisperse mixture with tails of both 16 and 18 carbon atoms and head groups lengths between 1 and 20 glucose molecules (TZ30), have been characterized both in terms of interfacial (tensiometry, ellipsometry) and in bulk behaviour (DLS, SAXS, SANS, cryo-TEM). C16G2 has been found to form long wormlike micelles when dissolved in water above the cmc. The size of these micelles are relatively unaffected of temperature changes from 25 °C up to 90 °C and NaCl concentrations up to 1 M, while they are growing slightly with increasing concentration (0.5-10 mM). TZ30 are both forming small spherical micelles and other larger aggregates when dissolved in water at concentrations above the cmc. Ellipsometry measurements have shown that both C16G2 and TZ30 adsorbs at a hydrophobised silica surface to a higher extent than PEG-surfactants of comparable size.

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Prof. WAHLGREN, Marie (Lund University)
Prof. ADLERCREUTZ, Patrick (Lund University)

1.11 Keynote: Scattering methods can unravel nano-structural changes in musculoskeletal tissues under loading – **Hanna** Isaksson (Lund University)

The musculoskeletal system enables locomotion of the human body, by force transfer through bone, cartilage, tendons and ligaments. Each tissue has a unique composition and hierarchical structure that result in an optimized mechanical function. Bone provides mechanical stability and support, while softer tissues such as tendons have a more damping function. With an aging population, the number of patients with musculoskeletal diseases, including fragile bones (osteoporosis), degenerated cartilage in the joints (osteoarthritis), and tendon pain (tendinopathy) is increasing.

We use scattering methods as one tool to understand how the quality of musculoskeletal tissues are affected by age and disease. In combination with other high-resolution imaging methods, we unravel how composition, structure and orientation in these tissues are affected on the macro-, micro- and nano-scales. By studying the tissues in-situ, under concurrent mechanical loading, we can link the alteration in nanostructure to mechanical competence of the tissues, and thereby the anatomical function.

As an example, we have combined experimental tensile testing inside SAXS and WAXS setups together with cameras on the surface and tomographic imaging, to study deformation simultaneous at multiple length scales in compact bone (Figure 1). We found that the orientation of the microstructure relative to the tensile loading influenced the strain magnitude on all length scales. Strains in the collagen fibers (measured by SAXS) were 2-3 times higher than the strains in the mineral crystals (measured by WAXS) for samples with microstructure oriented parallel to the loading. This will be extended to answer how the mineral crystal size and collagen fibre orientation and their response to load is altered in patients that are highly prone to bone fractures, e.g. osteoporosis.

Alterations in tissue quality can be addressed by combining the knowledge from advanced structural, compositional and mechanical analyses. A better understanding of tissue quality help predicting the functional integrity of the tissue and supports the development of better diagnostics methods and treatment options.

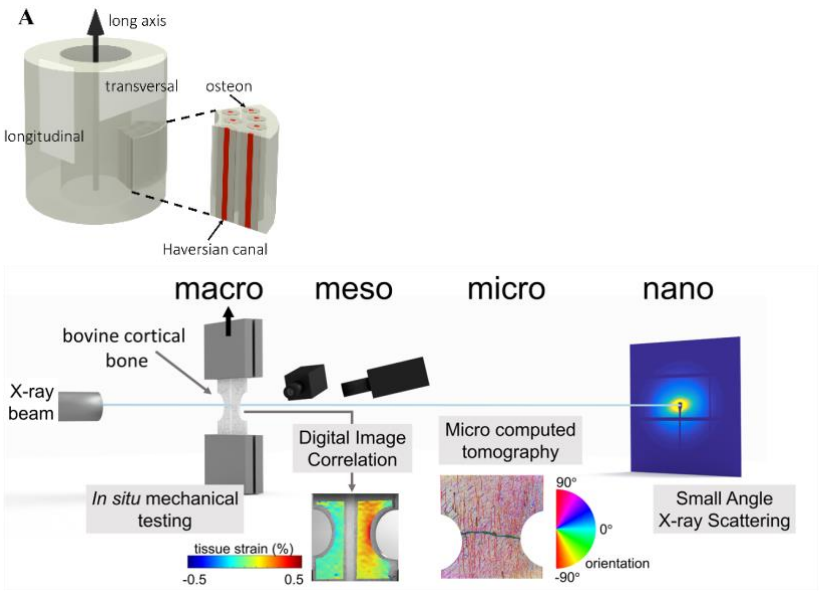

Figure 1. Setup of experimental study to unravel the nanoscale behaviour in bone in different microstructural directions in situ during mechanical loading (Gustafsson et al., 2018).

1.12 **Structure Determination by SAXS in Protein Biophysics and** Structural Biology – Jeppe Lyngsø (Århus University)

Jeppe Lyngsø[1], Eva Maria Steiner[2], Jodie Guy[2], Gleb Bourenkov[3], Ylva Lindqvist[2], Thomas R. Schneider[3], Gunter Schneider[2], Robert Schnell[2], and Jan Skov Pedersen[1]

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The ATSAS package is probably the most widely used program package for analysis of SAXS data on biomolecules and biomolecular assemblies. However, in connection with recent work on several projects, we have experienced short-comings with the available programs. The most restrictive circumstance is that only executable programs are available, thus limiting the application to ‘standard’ projects that fall within the range of applications considered by the authors of the programs. For example, modelling protein-detergent complexes and inclusion of structure factors to describe inter-particle interactions are not possible within ATSAS. This together with a somewhat inconsistent description of hydration layers of composite structures, and the need for faster tools have motivated us to develop alternative programs. In this talk, we will present one of these projects as a case study, where the new rigid-body refinement methods have been developed and utilized with great success in parallel with the traditional approaches, so that comparisons are possible. A bacterial enzyme, involved in remodeling of periplasmic peptidoglycan structures, and its various recombinant constructs have been characterized by solution SAXS. Data from both SAXS instruments at Aarhus University have been utilized in the project. Probable solution structures were determined and verified by various approaches for the individual constructs based on known high-resolution structures of the domains and ab initio modelling. This contributed valuable information to the overall collaborative study on this interesting enzyme.

1.13 The Interaction of Perfluoroalkyl Substances with Mineral Surfaces and Biological Membranes – Shirin Nouhi (Uppsala University)

Perfluoroalkyl substances (PFASs) have hydrophobic and hydrophilic characteristics which makes them useful in many commercial and industrial applications such as textile, leather and paper impregnation, detergents, fire-fighting foam, etc. These compounds are receiving increasing global concern due to their persistence, bioaccumulation and possible adverse effects on the environment and living organisms [1]. In this project, we have investigated the interaction of PFASs with biological and mineral model system interfaces using neutron reflection. The PFAS were selected to allow comparing the effect of hydrophobic chain length and hydrophilic functional group on their interactions with the interface.
The adsorption of PFASs was studied at two types of mineral surfaces (Al_2O_3, positively charged and SiO_2, negatively charged). The electrostatic interaction was shown to be the driving force in the sorption process. The adsorbed PFAS could be removed by gentle rinsing with water. The adsorption process was shown to be influenced directly by the solubility limit of the PFAS which changes with the chain length [2]. Phospholipids are the building blocks of cell membranes and are commonly used as a model system to understand the fundamental behavior of biological membranes. DMPC (1,2-Dimyristoyl-sn-glycero-3-phosphocholine) bilayer was chosen as the model interface in this study. PFASs have shown to penetrate into the bilayer and displace lipids to accommodate themselves. Off-specular data from bilayers which have been immersed to PFASs indicate rough and patchy structures. Extensive rinsing with water can remove some PFAS, but a less dense bilayer is left behind. The interaction of these PFAS was shown to vary with both head group and chain length, and strongly correlate with the PFAS solubility limit [3].

1. Hellsing et al. Chemosphere 159 (2016) 385e391.
2. Ahrens et al, Chemosphere 129 (2015) 33–38.
3. Nouhi et al, Journal of Colloid and Interface Science 155 (2018) 474-481.

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Mr. DE PEREIRA, Hugo (Swedish University of Agricultural Sciences)
Dr. AHRENS, Lutz (Swedish University of Agricultural Sciences)

1.14 Investigating the mesoscale of fibril hydrogels – Dr. Christina Efthymiou (Uppsala University)

Biomaterials widely presented in nature, like protein filaments in muscles, elastin in lungs, collagen in bones, cellulose fibers in fruits, and keratin in wool, self-assemble resulting in hierarchical structures that define their properties and uses. These hierarchical structures emerge from molecular and supra-molecular self-assembly systems organized on multiple length- and time-scales on the mesoscale. The study of such hierarchical structures at the mesoscale, where protein and carbohydrate self-assemblies like fibrils and tubes exist, is the particular interest of our research. Results from this study provide the knowledge to allow understanding the relationship between the architecture of fibril networks and their macroscopic properties, in order to connect the top (macroscale) to the bottom (microscale) and finally to understand how hierarchical systems, such as gels, are formed. This is achieved by measuring those properties using a combination of techniques; small-angle X-ray scattering for determining the molecular level self-assembly of the proteins and the resulting gel structure, cryo-scanning electron microscopy for investigating the 3D structural changes on the mesoscopic level, and bulk rheology for investigating the structural and kinetic changes on a macroscopic level.

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Prof. MCGRATH, Kathryn (Victoria University of Wellington, MacDiarmid Institute for Advanced Materials and Nanotechnology, New Zealand)

1.15 Keynote: Refolding of SDS-unfolded proteins by non-ionic surfactants: Equilibrium and kinetics – Jan Skov Pedersen (Århus University)

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The strong and usually denaturing interaction between anionic surfactants (AS) and proteins/enzymes has both benefits and drawbacks: For example, it is in good use in electrophoretic mass determinations (SDS-PAGE) but limits enzyme efficiency in detergent formulations. Therefore, studies of the interactions between proteins and AS as well as non-ionic surfactants (NIS) are of both basic and applied relevance. The AS sodium dodecyl sulfate (SDS) denatures and unfolds globular proteins under most conditions. In contrast, it has been shown that the NIS octaethylene glycol monododecyl ether (C12E8) protects bovine serum albumin (BSA) from unfolding in SDS. We have shown recently that globular proteins unfolded by SDS can be refolded upon addition of C12E8. Four proteins, BSA, α-lactalbumin, (αLA), lysozyme (LYZ), and β-lactoglobulin (βLG), were studied by small-angle X-ray scattering (SAXS) and both near- and far-UV circular dichroism (CD). All proteins form complexes with SDS with a structural organization as protein-decorated micelles, in which the protein preserves secondary structure. For βLG, there is a quite spectacular transition from mainly β-sheet structure in the native protein to mainly α-helical structure in the complexes. All proteins were attempted refolded by the addition of C12E8. Except for apo αLA, which has a molten globular state, the proteins did not interact with C12E8 alone. The addition of C12E8 to the protein-SDS samples resulted, except for aLA, in refolding of the tested proteins and dissociation from surfactant micelles. It was concluded that C12E8 competes with globular proteins for association with SDS, making it possible to release and refold SDS-denatured proteins by adding sufficient amounts of C12E8. The last part of the talk will describe recent work using synchrotron radiation SAXS in combination with stopped-flow techniques on the kinetics of unfolding and refolding with emphasis on βLG. For this protein, the preliminary analysis shows a fast aggregation, when SDS is added, and a gradual conversion of the structure to highly symmetric protein-decorated micelle structures that is nearly complete in 10 s. The refolding is significantly slower with time constants of minutes, however, CD and Trp fluorescence in our home lab reveal that there is also a much faster process that is not captured in the SAXS measurements, which is a secondary structure conversion. In the work, both simple analysis in terms of measured basis functions (SAXS data) as well as modelling on absolute scale have been used and will be briefly described.

Kaspersen JD, Søndergaard A, Madsen DJ, Otzen DE, Pedersen JS. Refolding of SDS-Unfolded Proteins by Nonionic Surfactants. Biophys J. 2017 112(8):1609-1620.

1.16 Liquid-Metal-Jet X-ray Source for In-situ SAXS studies in the Home Laboratory – Shichao Hu (Excillum)

High-end x-ray scattering techniques such as SAXS, BIO-SAXS, non-ambient SAXS and GISAXS rely heavily on the x-ray source brightness for resolution and exposure time. Traditional solid or rotating anode x-ray tubes are typically limited in brightness by when the e-beam power density melts the anode. The liquid-metal-jet technology has overcome this limitation by using an anode that is already in the molten state.
We have previously demonstrated prototype performance of a metal-jet anode x-ray source concept with unprecedented brightness in the range of one order of magnitude above current state-of-the art sources. Over the last years, the liquid-metal-jet technology has developed from prototypes into fully operational and stable X-ray tubes running in many labs over the world. Small angle scattering has been identified as a key application for this x-ray tube technology, since this application benefits greatly from high-brightness and small spot-sizes, to achieve a high flux x-ray beam with low divergence. Multiple users and system manufacturers have since installed the metal-jet anode x-ray source into their SAXS set-ups with successful results. With the high brightness from the liquid-metal-jet x-ray source, in-situ SAXS studies can be performed – even in the home laboratory.
The influence of the size of the x-ray source and its distance to the x-ray optics on the divergence will be discussed, and how to minimize the divergence and maximize the flux in SAXS experiments targeted to specific applications.
This presentation will review the current status of the metal-jet technology specifically in terms of stability, lifetime, flux and brightness. It will also discuss details of the liquid-metal-jet technology with a focus on the fundamental limitations of the technology. It will furthermore refer to some recent SAXS and GISAXS data from users of metal-jet x-ray tubes.

1.17 Time-resolved SAXS reveals ionic liquid interaction with model membranes – Dr. Inkeri Kontro (University of Helsinki)

Ionic liquids (ILs) are solvents that have many desirable properties. For example, they are easier to handle than volatile organic solvents and they dissolve cellulose. However, many are harmful. The toxicity of ILs is in part due to their effects on cell membranes. In this study, we have observed effects of (tetradecyl)tributylphosphonium acetate ([P$**{14444}][OAc]),** **trioctylmethylphosphoniumacetate ([P**{8881}][**OAc**]), **tributylmethylphosphoniumacetate** ([**P**_{4441}$][OAc]) and 1-ethyl-3-methylimidazolium acetate ([emim][OAc]) on model membranes composed of L-α-phosphatidylcholine multilamellar vesicles (MLV) by time-resolved small-angle X-ray scattering (SAXS).
The SAXS experiments were conducted on beamline ID02 at ESRF, Grenoble. The penetration of ILs into MLVs was studied at millisecond resolution using a stopped-flow mixing device. The results show that all studied ILs penetrated the lipid bilayers. [emim][OAc] and [P4441][OAc] caused a thinning of the lamellar distance but did not induce disorder in the lamellae. The ILs with larger cations destroy the lamellar order. The results give insight into the ways in which ILs can penetrate the cell membrane and cause changes to it.

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1.18 Characterization of liposomal formulations to treat Fabry Disease – Jannik Pedersen (Århus University)

Fabry disease is a lysosomal storage disorder, where the lack of α-Galactosidase A (GLA) causes accumulation of glycosphingolipids leading to damage of the kidneys, heart and nervous system. With current treatments, free GLA is injected intravenously in patients causing instability of GLA, high immunogenicity, and low bioavailability. To overcome this, liposomes has been developed that can encapsulate GLA and improve the performance. Further development of the liposomes is necessary and currently being done to archive perfect control of the assembly process. The liposomes consists of several different components that can alter the size, polydispersity and structure of the liposomes, and characterization of different formulations is therefore important in the development of new liposomal formulations. We have used an In-house small-angle X-ray scattering (SAXS) setup to get information on the average liposomal structure in presence of different lipids. We use a para crystalline model (Guinier, A. (1963). X-ray Diffraction, San Francisco: Freeman) with a finite number of layers and disorder between layers, while a set of Gaussians is used to describe the cross-section profile of the lipid bilayer. Scattering from GLA contributes to the total signal and is in the model added to the scattering from the bilayers, so that an estimate of the amount of GLA can be obtained. Using the model for fitting the data, we are able to follow incorporation of GLA in the liposomes and changes in bilayer thickness as well as changes in the amount of multi-lamellar structures and bilayer ordering. The incorporation of GLA in the formulations correlates well with the results from the SAXS data, and it is also observed that the distance between layers is increased when larger molecules are incorporated in the liposomes. The number of bilayers determined with SAXS correlates well with that seen with cryo-TEM. Altogether, the SAXS data can give us a lot of information on the structure, composition and polydispersity of the liposomes. We also used static and dynamic light scattering to obtain information on the size and polydispersity of the samples and can use this in characterizing the homogeneity of the samples. This information is important for further development of new liposomal drug formulations and will be an important tool in the design of liposomes that has a low polydispersity, are stable and can help in improving the treatment of Fabry Disease. This project has received funding from the European Union's Horizon 2020 research and innovation programmer under the grant agreement No 720942.

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1.19 Inverse- and real-space scattering of aqueous diblock copolymer micelles – Dr. Gregory Smith (University of Copenhagen)

Poly(glycerol monomethacrylate)–poly(benzyl methacrylate) (PGMA–PBzMA) diblock copolymer micelles were synthesized via polymerization-induced self-assembly (PISA) using reversible additional–fragmentation chain-transfer (RAFT) aqueous emulsion polymerization in D2O. PISA reactions produce polymer nanoparticles in situ during the reaction and can be performed at high particle concentrations. PGMA–PBzMA synthesized by PISA is known to form only spherical micelles, making it an ideal model system for exploring new characterization methods. The structure of the polymer micelles was obtained using small-angle X-ray scattering (SAXS) and, a more recently developed form of neutron scattering, spin-echo small-angle neutron scattering (SESANS). Fitting the scattering data from these techniques showed that the inverse-space (SAXS) and real-space (SESANS) scattering gave structural parameters that compare very favorably. As far as we are aware, this is the first report of polymer micelles being studied by SESANS. Using both an inverse-space and a real-space scattering technique, with optimal sensitivity at different lengthscales, has made it possible to gain interesting information about both the structure of and interactions between polymer micelles as a dilute dispersion (SAXS) and directly in the synthesis medium (SESANS).

2 Poster Abstracts

2.1 HEXOSOMES BASED ON OMEGA-3 LIPID EICOSAPENTAENOIC ACID MONOGLYCERIDE - AL-HOSAYNI, Sabah (University of Copenhagen)

ω-3 PUFA supplements are not only available in free fatty acid, triglyceride, ethyl ester, and phospholipid-enriched forms but they are also available in the form of monoglycerides: eicosapentaenoic acid (MAG-EPA), docosahexaenoic acid (MAG-DHA), and docosapentaenoic acid (MAG-DPA) monoglycerides, respectively. Recent studies demonstrated the potential therapeutic use of these newly synthesized omega-3 (ω-3) polyunsaturated fatty acid (PUFA) monoglycerides owing to their beneficial health effects in various disorders including cancer and inflammation diseases. To date, the research was mainly focused on exploring the biological effects of these functional lipids. However, to the best of our knowledge, there is no report on the hydration-mediated self assembly of these lipids that leads to the formation of nanostructures, which are attractive for use as vehicles for the delivery of drugs and functional foods. In this contribution, we present the temperature-composition phase behaviour of eicosapentaenoic acid monoglyceride (MAG-EPA), which is one of the most investigated ω-3 PUFA monoglycerides, during a heating-cooling cycle in the temperature range of 5-60 °C. Experimental synchrotron small-angle X-ray scattering (SAXS) evidence on the formation of a dominant inverse hexagonal (H2) lyotropic liquid crystalline phase and its temperature-induced transition to an inverse micellar solution (L2 phase) is presented for the fully hydrated bulk MAG-EPA system and its corresponding dispersion.1 We produced colloidal MAG-EPA hexosomes with an internal H2 phase in the presence of F127, a well-known polymeric stabilizer, or citrem, which is a negatively charged food-grade emulsifier. We show also that MAG-EPA hexosomes can be produced by vortexing MAG-EPA in excess aqueous medium containing F127 at room temperature. This low-energy emulsification method is different than most reported studies in the literature that have demonstrated the need for using a high-energy input during the emulsification step or adding an organic solvent for the formation of such colloidal non-lamellar liquid crystalline dispersions.

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2.2 Formation Kinetics Of Poly Ion Comlexes Studied With Time-Resolved SAXS - Dr. AMANN, Matthias (Department of Chemistry, University of Oslo)

Micellar aggregates formed by oppositely charged polyelectrolytes (Poly Ion Complexes, PIC) have recently attracted a large interest due to their possible applications in the biomedical field[1]. Although showing promising properties to act as carriers providing a high drug loading and triggered release by external stimuli like pH or salt concentration, the formation process of these polyelectrolyte complexes is still barely studied. Understanding the formation kinetics is a crucial key in practical formulations and being able to selectively tailor and design PICs as drug delivery systems. The electrostatic interactions require cooperative movements of the polyelectrolyte chains that result in very different kinetics to micelle formation in classical amphiphilic systems. In this contribution we present a time-resolved small-angle X-ray scattering (SAXS) study on the formation kinetics of PICs formed by mixtures of Poly(Sodium 4-StyreneSulfonate) (PSSS) with 20 to 160 SSS units and Poly(ethyleneglycol)-block-Poly((Vinylbenzyl)Trimethylamonium chloride) (PEG-b-PVBTA) with a 2 kg/mol PEG and 5 to 30 VBTA units. The polyelectrolytes are mixed at a fixed 1:1 charge ratio, focusing on the influence of molecular weight Mn and concentration of the components on the complexation kinetics. Our results show a fast formation of metastable large aggregates directly after mixing which rearrange into polydisperse spherical micelles over time. The initial clusters are formed immediately in all cases but the kinetics of the rearrangement into spheres shows a strong dependency on molecular weight of the polyelectrolytes, in particular to the number of charges per polyelectrolyte chain. With fixed PSSS molecular weight, the rearrangement is fast for low molecular weight PVBTA and becomes significantly slower with increasing Mn(PVBTA). Above a certain threshold Mn, which corresponds to an equal number of charges on PSSS and PVBTA, no rearrangement is evident on the experimental time scale at all. Similar trends can be observed when fixing the PVBTA molecular weight and varying Mn(PSSS). In order to quantify these general observations, the data are fitted with a spherical core-shell model including graded interfaces. Large scale aggregates are taken into account by modeling clusters of Nclu randomly connected micelles. This allows to follow the formation kinetics by describing the time dependence of crucial parameters like the aggregation number P, micellar dimensions Rcore and Dcorona and the number of clusters Nclu in detail.

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2.3 Structure of Starblock Copolymer Thin Films – Mr. ARIAEE, Sina (Roskilde University)

Using advanced methods of chemical synthesis, linear polymer chains can be linked together to form a multitude of different multiblock copolymers with sophisticated molecular architectures and interactions, allowing vast possibilities in tailoring materials with nano-scale architecture [1]. Recently, ABC star-block copolymers, where three different polymers are linked together at a common core have come into focus for use in e.g. nanolithography [2,3]. When forming ordered morphologies, ABC star-block copolymers are restricted compared to linear block copolymers, since the block junctions have to be located on a line. In addition, ABC star-block copolymers have competing interactions between the three different arms and thus a very rich phase diagram [4]. We present structural studies of thin films of Star(polyisoprene-block-polystyrene-block-poly(2-vinylpyridine)) and Star(polyisoprene-block-polystyrene-block-poly(methyl-methacrylate)) with systematic variation of arm length ratio, using both AFM (Atomic Force Microscopy), SEM (Scanning Electron Microscopy) and GISAXS (Grazing Incidence Small-Angle X-ray Scattering). .

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2.4 Salt-induced temperature-dependent protein cluster formation: access to binding entropies and enthalpies - Mr. BECK, Christian (ILL)

With increasing Yttrium Chloride (YCl_3) salt concentration c_s , aqueous Bovine Serum Albumin (BSA) protein solutions subsequently change from a visually transparent (regime I) to a turbid phase (regime II) and back to a transparent phase (regime III: reentrant dissolution). Within regime II, a lower critical solution temperature (LCST) associated with a liquid-liquid phase separation (LLPS) can be observed. Quasi-elastic neutron scattering (QENS) data as a function of c_s and the protein concentration c_p lend support to the formation of protein clusters when approaching regime II [1]. By applying the Wertheim theory for patchy particles [2] and the Flory-Stockmeyer cluster size distribution function, it is possible to describe the temperature dependent cluster formation quantitatively [3]. The ion-binding and protein- protein bridging entropies and enthalpies associated with the clusters can be determined directly from a simultaneous fit of the model to a large set of QENS spectra for different c_p , c_s , and T. The results are compared with calorimetry measurements [4].

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2.5 A surface active enzyme and colonization factor investigated by x-ray and neutron scattering - Dr. BJERREGAARD-ANDERSEN, Kaare (University of Oslo)

Colonization and biofilm formation on biosurfaces are important in early development of infectious diseases. The bacterial family **Vibronaceae** contains several strong human and fish pathogens e.g. **Vibrio cholerae** and **Vibrio salmonicida**. The secreted adhesin GbpA is found commonly in the **Vibrionaceae** and is essential to the initial reversible attachment of the bacterium to the surface as it mediates interaction. GbpA is an attractive target for developing strategies for intervention such as binding inhibitors or vaccines. Moreover, GbpA carries lytic polysaccharide monooxygenase (LPMO) catalytic activity, which has high potential of use in the production of biofuels, while the role of this activity in virulence is unknown. Also GbpA is under regulation of proteins involved in quorum sensing. Altogether, this makes GbpA a pivotal study target in the understanding of vibrio colonization, however, relatively little is known about the structure-function relationship of the protein.
In this project, we seek to investigate the structural biology of the GbpA interaction with carbohydrates using x-ray and neutron scattering techniques. The study encompasses crystallography, small angle scattering and reflectometry to develop a complete model of the surface interaction of GbpA.
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2.6 Liposomes as a model system for the study of surface active peptides - Ms. BJØRNESTAD, Victoria Ariel (University of Oslo)

Antimicrobial peptides (AMPs) are interesting agents for the development of future antibiotics, but the lack of knowledge about their mode of action makes AMP-based drug design difficult. A proposed method of studying the AMPs interactions with the membrane is designing liposomes that mimic the bacterial and mammalian membranes which can then be measured using small angle X-ray/neutron scattering (SAXS/SANS) techniques to find changes in the structure of the lipid bilayer upon addition of AMPs. Liposomes alone, however, have been found to precipitate in the presence of AMPs, and therefore model systems using different polyethylene glycol derivatives have been tested. Addition of both free n-alkane-polyethylene oxides (Cn-PEO) to the liposomes and incorporation of polyethylene oxide ("PEG")-modified phospholipids in the liposomal bilayer was tested and found to efficiently stabilize the liposomes through steric repulsions. However, although addition of n-alkane-polyethylene oxide makes the system more versatile and simpler in terms of preparation and modelling, the n-alkane-poly(ethylene oxide) was found to interact and modify the membranes to a great extent. At 37 °C, we also find that Cn-PEO, in addition to forming mixed micelles with the lipids seem to interact with the AMPs themselves, yielding unpredictable results with both dynamic light scattering, small angle scattering and calorimetric measurements. These results suggest that the better approach is to use PEG-modified phospholipid, although this means incorporating the PEG both on the inner and outer leaflet of the liposomes. PEG-modified liposomes do stabilize the liposomes against aggregation without modifying the bilayer significantly and the PEG does not seem to prevent the AMPs' interaction with the membrane either, making it a good system for studying their microscopic mode of action.

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2.7 Development of a sample environment for the study of mechanically confined and sheared geometries - BOYD, Hannah (Malmö University)

The study of soft materials confined between surfaces is central to the understanding of interactions that lead to adhesion, lubrication and colloidal stability. Force measurements have long been able to describe the potential that exists between various coated surfaces, but it is only more recently that scattering techniques, particularly reflectometry, have permitted the detailed study of surface layers under confinement. Specifically, the recent development of a confinement cell that consists of a solid surface confined by an expanding flexible film has been able to address key factors that have hampered these investigations for a long time by achieving parallelism and good contact over a large area. These advances have enabled new fields of research for studying surfaces under confinement using neutron reflectometry [1]. Within the framework of a project recently funded by the Swedish Research Council, researchers from Malmö University and the collaborative team behind the development of this cell will work with researchers at both ESS and ISIS to build on this success towards a next-generation surface confinement apparatus. Specifically, the key deliverable will be to develop a sample environment capable of applying shear under confinement which can be integrated with GI-SANS and reflectometry geometries to provide information on the structural changes in all dimensions. To achieve this goal, the cell will be designed and manufactured in collaboration with ISIS and fully optimized for full integration on to the beamlines at ESS. There is significant private and public sector interest in understanding lubrication and developing nanomaterials to mimic nature. Thus, the huge potential of this project to significantly expand the research field accessible with neutrons will be demonstrated by employing it to investigate the mechanism underlying the outstanding performance of biological lubricants, in particular salivary pellicles and mucus blankets [2]. In this communication we provide an overall presentation of the planned implementation and goals of this project.

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2.8 **In-house SAXS For Biological Samples -** Dr. BUCCIARELLI, Saskia (Copenhagen University)

Small angle X-ray scattering (SAXS) is a powerful tool to study biological macromolecules in solution, yielding information on their shape, aggregation state, low-resolution structure, as well as on protein-protein interactions, without interfering with the solution. Here we show a selection of applications of the BioXolver, a dedicated in-house bioSAXS instrument developed in collaboration between SAXSLAB and the BioSAXS group at UCPH. The BioXolver is, amongst others, equipped with a flow-through exposure cell and a sample-handling robot allowing robotic mixing and automated loading of a series of samples. The applications presented here are

Low-resolution ab initio protein shape determination within a matter of minutes. As SAXS data is obtained from unperturbed solutions, these ab initio models can serve as a validation of higher-resolution models, obtained for example by crystallography.

Investigation of complex systems containing different species, such as for example fibrillating proteins. Amyloid-like protein fibrillation is associated with various neurodegenerative diseases, amongst others Parkinson's and Alzheimer's. During the fibrillation process, originally monomeric proteins associate into larger oligomers and finally form mature micrometer long fibrils. The current hypothesis in the field is that the transiently formed oligomers are the cytotoxic species. It is thus highly relevant to structurally characterize these oligomers, but the fact that they always exist in equilibrium with monomers and fibrils and can not be physically isolated renders such structural studies very challenging. SAXS, combined with advanced analysis software, is optimally suited to study such systems containing multiple species in solution, as it does not require their physical isolation.

Determination of protein-protein interactions in different solvent conditions. The robotic mixing functionality of the BioXolver allows to quickly an easily screen different solvent conditions and protein concentrations, thus yielding information on two-body protein interactions, through the osmotic second virial coefficient.

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2.9 **Modelling of Flexible Proteins -** Mrs. CRAGNELL, Carolina (Theoretical chemistry, Lund University)

The existence of functional disordered (unstructured) proteins has been recognized for many years. However, due to the classical structure-function paradigm, the functional role of intrinsically disordered proteins has only recently been recognized. Biochemical evidence has since shown that these proteins are functional, and that the lack of a folded structure is related to their function.
We would like to present results from a combined experimental and theoretical study, where the aim is to develop a model for flexible proteins and to relate the lack of structure of the proteins in solution with their function and structure when adsorbed to surfaces. For this purpose, we are combining atomistic and coarse-grained modelling, with simulation techniques such as molecular dynamics and Monte Carlo simulations. The simulations are compared with SAXS experiments of a model protein (Histatin 5).
There are good agreement between the scattering curves for Histatin 5 obtained from SAXS and the simulations. At high salt concentration, the protein behaves as a neutral polymer, and at low salt concentration, a repulsive peak is obtained at low q. In the latter regime, it is the net charge of the protein that is of importance for the inter molecular interaction and not the charge distribution. Preliminary results also indicates that the peptide is more streched out in low pH solutions (in the salivary pH range) as well as in prescence of divalent ions such as Zn^{2+}, Mg^{2+}, and Ca^{2+}, This indicate that electrostatic interactions indeed are important for Histatin 5 bulk structure.

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2.10 Structure Analysis of Drug Delivery Systems with SAXS in the Laboratory - Dr. EHMANN, Heike (Anton Paar)

Small-Angle X-ray Scattering (SAXS) draws increasing attention in the field of pharmaceutical engineering. SAXS is a versatile technique used for shape and size characterization of nanostructured materials between 1 nm and 200 nm. Biological samples, like proteins or viruses are already well known to be investigated with SAXS. Furthermore drug delivery systems like drug loaded vesicles (see example in figure 1), where size and shape parameters of the vesicle and the drug are found or granulate powders, where the internal surface obtained by SAXS correlates with the tablet hardness, are interesting examples of applications in pharmaceutical research.
In this contribution we present select applications of biological samples, employing a multifunctional laboratory Small and Wide Angle X-ray Scattering (SWAXS) system, the SAXSpoint. The SAXSpoint system enables SAXS and WAXS studies at ambient and non-ambient conditions, GI-SAXS, in-situ tensile SWAXS experiments and satisfies the advanced user with a wide range of dedicated sample stages, full experimental flexibility and highest resolution. The system provides simple operation, short measurement times and excellent angular resolution, enabled by a smart beam formation concept which includes a brilliant X-ray source, advanced X-ray optics and optimized scatterless collimation while maintaining a laboratory-friendly compact size and small footprint.
Different scattering studies on biological and pharmaceutically relevant samples were performed on the presented SAXSpoint system. Some of the samples required high resolution, i.e. a very low minimum scattering angle in order to resolve large structural dimensions. The unique sample-positioning mechanism enabled WAXS measurements to determine crystallinity without re-aligning any part of the SWAXS system. The presented studies clearly show that high-resolution and high-quality SWAXS data can be obtained with a laboratory SWAXS system.

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2.11 The Effect of Temperature and Ionic Strength on Micellar Aggregates of Oppositely Charged Termoresponsive Block-co-polymer Polyelectrolytes - Mr. FEHÉR, Bence (Eötvös Loránd University, Budapest; Aarhus Univesity, Interdisciplinary Nanoscience Center, Aarhus)

In recent years block-co-polymers have been investigated intensely due to their potential application as polymer nanocapsules [1]. One possibility is to use termoresponsive block-co-polymer with polyelectrolyte blocks for making nanocapsules, which may have controlled drug release properties. For the relatively low molecular mass polymers in pure water, cylindrical micellar-like structures formed due to the electrostatic interaction, and these were stable also at higher temperatures. In our study we investigated the structure of mixtures of the cationic pNIPAM-b-poly((3-acrylamidopropyl)trimethyl ammonium chloride) and the anionic pNIPAM-b-poly(4-styrenesulfonic acid sodium) of higher molecular mass with 1:1 charge ratio by Small-Angle X-Ray Scattering (SAXS) with the main aim to investigate the dependence of the structure of the molecular aggregates on temperature and NaCl concentration. We performed our measurements at eight different salt concentration (0, 1, 5, 10, 25, 50, 100, 500 mM), at seven different temperatures (20, 25, 30, 35, 40, 45, 50 Celsius). The data were fitted by a spherical micelle model, which has a smoothly decaying radial profile and a Gaussian star term that describes the internal structure of the micelle and possible attractive interactions between the polymer chains. At high temperature and high salt concentration, a cluster structure factor is used for describing the formation of bulky clusters of the molecular aggregates. The perfect fits of the model to the SAXS data suggest that water is not really a good solvent for pNIPAM at the relatively high polymer concentration in the micellar corona. According to our results, below 10 mM salt concentration and below 40 Celsius, micellar, aggregates with quite high aggregation numbers are present due to the strong electrostatic attraction between the oppositely charged blocks. From 10 mM salt concentration, we found a decrease of the aggregation number at constant temperature, which can be explained by the screening of the electrostatic interactions. Above 40 Celsius and at high salt concentrations, the aggregation number increases again due to pNIPAM becoming insoluble in water and there is a possible formation of inverse micelles driven by the hydrophopic forces. It is also at these conditions that bulky aggregates are formed.

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2.12 **Ferrihydrite-Soil Organic Matter Interactions: joining scattering and microscopy -** Dr. GENTILE, Luigi (Lund University)

The intricate associations of soil organic matter (SOM) with minerals are mainly investigated because of their role in determining the long-term retention of SOM. The interaction between aqueous solutions containing dissolved organic matter (DOM) extracted from boreal forest soil and ferrihydrite nanoparticles has been investigated at the nanoscale by using small angle light and X-rays scattering along with cryo-transmission electron microscopy (TEM). This work demonstrates the self-assembly of ferrihydrite (iron-oxides) nanoparticles in presence of dissolved organic matter extracted from soil. Soil Continuum Model considers that the organic material is transformed by decomposing organisms, such as fungi and bacteria, from large molecular fragments to smaller molecules. Here the interaction between soil organic matter and ferrihydrite nanoparticles is highlight at the nano-scale where specific kind of colloidal clusters are individuated. The organic matter is reteined against microbial decomposition through incorporation into molecular aggregates and interactions with mineral particles. Moreover, these aggregates could leach from the litter (O-horizzon) to the upper part of the A-horizon.

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2.13 **Porphyrin Adsorption by Stabilized TiO2 Nanoparticles** - Mr. GÖTZ, Klaus (Institute for Crystallography and Structural Physics (ICSP))

The use of titanium dioxide (titania) nanoparticles in dye-sensitized solar cells is considered a low-cost alternative to classical semiconductor devices. Antase is known to exhibit the best optoelectronic properties for this application out of the three natural crystalline modifications of titania (rutile, anatase and brookit). We synthesized tunable titania nanoparticles with a diameter of ~2-3 nm using a hot injection method. The produced nanoparticles are stabilized by oleic acid. Special emphasis of our work is focused on the exchange process of the oleic acid with porphyrins designed for a particular application.

This process has been studied by small angle x-ray and neutron scattering (SAXS and SANS). As X-rays interact mainly with the electrons, SAXS yields information essentially about the inorganic core of the nanoparticles. Neutrons on the other hand are very sensitive to hydrogen and therefore SANS is well suited to get information about the organic stabilizer shell. A combined analysis of SAXS and SANS data provides detailed information on the structure of layered core/shell systems. This information can be further improved by additional contrast variation measurements. For SANS this has been realized by variation of the mixing ratio of the protonated and deuterated solvent.

In the talk a synthesis route for anatase nanoparticles with tunable morphology will be introduced. The morphology of the produced particles will be discussed on the basis of our extensive SAXS and SANS studies. Furthermore, first results on the ligand exchange from oleic acid to porphyrins will be presented.

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2.14 Measuring dynamics of anisotropic particles with XPCS - Dr. HOLMQVIST, Peter (Lund University)

Anisotropic colloidal particles (such as rod- or platelet-like) have taken an increasingly larger space in the industrial and scientific community in recent years. Due to their anisotropic nature both in shape and interaction they have the ability to organize themself in different structures. The disorder to order transition for hard thin rods is an isotropic to nematic transition where the particles have orientational order but no positional order. With more elaborate anisotropic particle, in both shape and interaction, a multitude of different transitions and phases can be found. Due to their versatility anisotropic particles can be found in large abundance in nature and is frequently used in industrial applications. To investigate and understand these systems not only the structural and static information has to be investigated but also the dynamics. A major challenge in investigating anisotropic particles is to cover the whole length scale of the particles. For anisotropic particles in the colloidal domain x-ray scattering experiments are well suited since they can cover a large part of the reciprocal space that is with in the interesting length scales of the particles. In order to investigate the dynamic responses in theses systems one has then to turn to XPCS. I will present XPCS investigation on platelet particles. Due to the good amplitude of the measured correlation function a broad q-range could be accessed. From this the rotational diffusion could be extracted. I will also show XPCS measurements on ellipsoids. As these particles align, either with concentration or external field, the scattering pattern becomes anisotropic and hence the dynamic. With XPCS the anisotropic dynamics can then be investigated at different q-vectors at different phase states, i.e. concentration and/or external field.

2.15 Re-entrant Phase Behavior in Charged Nanoparticle Solutions - Dr. KUMAR, Sugam (Stockholm University)

The anionic silica nanoparticles have been observed to show interesting re-entrant phase behaviour where stable nanoparticles undergo a transformation from one-phase (individual) to two-phase (nanoparticle aggregation) and return back to one phase system with tuning of the system parameters. Such phase behaviour can be achieved by playing with the degree of repulsive and attractive interactions in the system. Here, we show that addition of polymer and/or multivalent ions render similar re-entrant phase behaviour in charged nanoparticle solution, however utilizing completely different mechanism. Small-angle neutron scattering (SANS) and dynamic light scattering (DLS) along with the other macroscopic techniques have been used to investigate the system and responsible interactions.

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2.16 SAXS as Sample Environment at the ILL D22-SANS-Beamline - LAGES, Sebastian (Friedrich-Alexander-Universität Erlangen-Nürnberg,Department of Condensed Matter Physics, Institute for Crystallography and Structural Physics)

Small-angle X-ray and small-angle neutron scattering experiments often complement each other. A series of contrast variation SANS experiments can be complemented by an additional SAXS experiment, and in many cases the additional contrast provided by the X-ray scattering data allows for a more detailed analysis of the data. An example is the study of growth processes of inorganic nanoparticles that are stabilised by a shell of organic residues. It is often difficult, if not impossible, to prepare the samples in a way that both, SANS and SAXS experiments, are carried out under the same initial conditions. This project aims at overcoming this limitation in a collaboration with the Institute Laue-Langevin (ILL).
A lab-scale SAXS instrument is going to be installed as a sample environment at the D22-SANS-instrument of the ILL. The SAXS instrument will not only be highly automated but it will also be integrated into the instrument control software of the D22 to guarantee the best possible performance when executing simultaneous SAXS-SANS experiments at the very same sample volume.
This talk presents the design and the ongoing construction of the SAXS instrument as well as some scientific cases where simultaneous SAXS and SANS experiments are highly useful.

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2.17 Development of Chemical-Space Screening by Small-Angle X-Ray Scattering (SAXS) - Mr. LYNGSØ, Jeppe (Aarhus University)

Developments in laboratory X-ray sources and in laboratory instrumentation for SAXS during the last decade have been astonishing. The introduction of the liquid Ga metal-jet X-ray source by Excillum has given about a factor of about ten higher brightness than one has for rotating anode X-ray sources. For the SAXS instrumentation there have been impressive developments in X-ray optics in the form of elliptical or parabolic multilayer mirrors, however, the most important development is probably the introduction of 'scatterless' slits (Li et al. J. Appl. Cryst. (2008). 41, 1134-1139), where the edges are made of a single crystal of Ge or Si. With the divergent source that one has in the laboratory, this gives a factor of 5-10 higher flux at the sample position compared to a conventional 3-pin-hole collimation system. With the increase in flux, data acquisition times have decreased correspondingly down to a few minutes even for weakly scattering dilute samples. Therefore, automatization of data acquisition and sample handling have become important. At Aarhus University, we have a Bruker AXS Nanostar SAXS instrument with an Excillum liquid metal-jet source, optimized optics and geometry, employing homebuilt scatterless slits. For this instrument, we have introduced an automated sample handler based mainly on commercial Gilson components. The sample handler is connected to a homebuilt flow-through capillary, can pick up the sample from various sample racks including a thermostated block, place the sample in the capillary for the measurements and clean and dry the capillary after use. The sample handler has led to a very efficient use of the SAXS instrument

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2.18 **NON-LAMELLAR LIPID LIQUID CRYSTALLINE PHASES – CONTROLLING THE FORMED STRUCTURE USING LIPOLYTIC ENZYMES WITH DIFFERENT SPECIFICITY -** Dr. MARIA, Wadsäter (Lund University)

Lipids in living organisms do not always confined in bilayer structures, but they can also assembly into intriguing 3D structures. Well-defined model system will help us understand the biological implication as well as develop new applications, for e.g. biomedical devices and targeted delivery. Such different structures can be generated or evolved with the help of specific lipolytic enzymes. Here we will demonstrate that the lipolysis-induced evolution of a particular structure from reverse lipid phases formed by mixtures of lipids, which invoke different curvature, is indeed controlled by the type of lipolytic enzyme. For this purpose we used highly structured cubic micellar (Fd3m) nanoparticles of 50/50 (wt%/wt%) soy phosphatidyl choline (SPC)/glycerol dioleate (GDO). The two types of lipolytic enzymes used were phospholipase A2 (PLA2) that catalyses degradation of the phospholipid component, SPC, and porcine pancreatic triacylglycerol lipase (TGL) that facilitate the hydrolysis of the diglyceride, GDO. Phospholipase A2, which promotes the hydrolysis of the lamellar forming component, SPC, induces a reversed micellar phase. However triacylglycerol lipase, which hydrolysis the reverse phase forming compound, GDO, induces a lamellar phase. The lipid particles were found to retain their integrity throughout the whole time of reaction studied.

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2.19 Self- and co-assembly of block copolymer micelles - Mrs. MORTENSEN, Henriette (Aarhus University)

Poly(ethylene oxide)-b-Poly(methacrylic acid sodium salt) (PEO-b-PMANa) is a weak polyelectrolyte. Its ability to form micelle depends on the ionization degree, which can be controlled through pH. At low pH, the polymer is on the protonated form Poly(ethylene oxide)-b-Poly(methacrylic acid) (PEO-b-PMAA) and it is believed to form micelles either due to PMAA hypercoiling[1] or to intrachain interactions between PEO and PMAA[2]. The polymer exists as single chains at higher pH [1, 2]. Polymer micelles forms at intermediate pH when PEO-b-PMANa is mixed with Ca2+ [1]. In the present study small-angle x-ray scattering (SAXS) is used to detect micelle formation and modeling of the SAXS data can give a more detailed description of shape and size of the formed micelles. Nuclear magnetic resonance is used to investigate solubility and to reveal solid-like cores. Multiple polymers with varying polymerization degrees are studied. The dependence of micellization on concentration, pH, charge neutralization degree and temperature is investigated. Preliminary results from SAXS indicate that micelles form at low pH (low ionization degree). Micelle formation is not seen in all polymer samples and the presence of micelles does not correlate with polymer size. This is attributed to differences in polymer solubility and to possible differences in chemistry with regard to polymer end groups. The same inconsistency is seen from modeling of SAXS data from samples at high pH, where micelles are found in some of the samples. One specific polymer is studied together with Ca2+ at varying charge neutralization degrees and temperature. Micelles do not form at low polymer concentration (0.5 wt%). For 2 wt% samples, micelles decrease in size, as charge neutralization degree (Ca2+ concentration) increases. For the micelle-forming samples, micelle size decreases as temperature is increased.

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2.20 GISANS simulations on lipid covered nanowires - Ms. MOTHANDER, Karolina (Physical Chemistry)

Reveal how membrane curvature affects protein interactions, using lipid bilayer on GaP nanowires. GISANS simulations on the nanowires arrays and lipid bilayer covered nanowires are preformed with the software BornAgain. Form the simulation we found the highest intensity from short nanowires, with a small increase in intensity with addition of the lipid bilayer.

2.21 **Polymer growth dependence on crystal orientation -** Mr. NAGY, Bela (Linköping University. Sweden)

Neutron reflectometry is an essential tool for investigating the hydration in polymer thin films. Since the method is based on the interference of waves reflected from different interfaces, the recorded reflectograms are greatly influenced by the roughness of the layers in a sample. To decrease these parameters only well controlled methods, such as surface initiated atom transfer radical (SI-ATRP) and reversible addition-fragmentation chain transfer polymerization, are used to produce such films. Furthermore, the polymers are usually deposited on the oxidized <111> face of silicone blocks as they can be polished to the required smoothness with less effort. To reduce costs, for the initial optimization of the growth process, silicon wafers with <100> orientation are used. Here we report a discrepancy between growth rate of hydroxyethyl methacrylate (HEMA) and poly(ethylene glycol) methacrylate (PEGMA) random co-polymer films with 1:1 monomer ratio grafted with the SI-ATRP method depending on the substrate facing. The surface coverage of the initiator was compared by x-ray photoelectron spectroscopy. To decouple the surface from the crystallographic orientation of the wafer, layers were also grafted on samples that had been annealed at 800°C. The growth dynamic of the films was investigated by recording Br- ion depth profiles using time-of-flight secondary ion mass spectroscopy.

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2.22 Interfacial properties of compounds derived from hemicellulose - Ms. NAIDJONOKA, Polina (Lund University)

Softwood hemicellulose (galactoglucomannan, GGM) is one of the most abundant polysaccharides found in plant cell walls. It makes up to 25% of the wood in Sweden, however, currently only a small fraction is used as a renewable resource and the rest is discarded in waste-streams from agriculture and forest industries. Our aim is to develop sustainable and efficient methodology based on biocatalytical processes to transform GGM into products with added value like surfactants for detergent formulations and coatings for wood and paper. This study is focused on revealing relevant physical properties of compounds derived from hemicellulose. One key property is the interfacial behaviour of the compounds which will be presented. This includes the surface tension and micelle formation of the novel hexyl mannoside surfactant synthesized within the project by means of enzymatic catalysis using β-mannanase and alcohol acceptor (Morrill et al 2017). Furthermore, using ellipsometry measurements we will show how the adsorption properties of unmodified GGM samples to spin coated cellulose surface are affected by different extraction techniques. We will demonstrate that the adhesion can be controlled by GGM branching and molecular weight. In order to study the influence of the galactose side groups on the adsorption kinetics of GGM, another enzyme, α-galactosidase, is used to alternate galactose substitution degree (von Freiesleben et al 2016, Reddy et al 2016).

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2.23 Fixing colloidal particles at solid/liquid interfaces using Moringa oleifera seed protein as 'glue' - Ms. NOUHI, Shirin (Uppsala University)

Colloidal particles such as polymer latices can form large crystalline structures close to, but not directly on a solid/liquid interface by combination of flow, packing constraints and charged repulsion imposed by the solid boundary [1]. These structures can be used directly to modify large areas and as templates for two-dimensional metallic structures using lithography. Such materials are not yet used widely since preserving the required structure on drying remains as a major challenge. Particles tend to move and the structures often crack. Proteins extracted from the seeds of Moringa oleifera trees have been shown to be effective flocculants for particles dispersed in water [2] to which they bind strongly. These proteins also adsorb irreversibly to mineral surfaces such as silica and alumina [3, 4]. These features of the proteins allow them to be used as a 'glue' to stick particles in position and to retain their self-organized structure. In this study, we have used quartz crystal microbalance, atomic force microscopy and neutron reflectometry to show the use of a crude extract of Moringa protein that is pre-adsorbed on silica surfaces as a means to bind particles, with two sizes, in a controlled manner at interfaces [5].

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2.24 Field Induced Self Assembly and Dynamics of Anisotropic Magnetic Particles - Dr. PAL, Antara (Lund University)

Anisotropic particles are known to exhibit a rich phase behaviour. In addition to the usual gas, liquid, crystal and glassy states found for spherical particles, anisotropic particles such as rods are known to exhibit additional liquid crystalline phases. Here we present the field induced self-assembly and dynamics of a novel anisotropic colloidal particle whose shape resembles a peanut. Being made up of hematite cores and silica shells, these particles align in a direction perpendicular to the applied external magnetic field. Ultrasmall-angle x-ray scattering (USAXS) studies on their self-assembled structures in sedimented samples reveal the formation of a nematic like phase. The anisotropic dynamics of these particles was investigated using multispeckle ultrasmall-angle x-ray photon correlation spectroscopy (USA-XPCS). Our results indicate that along the direction of the magnetic field the relaxation follows a compressed exponential behaviour while in the perpendicular direction (also the direction of gravity) an advective term together with a purely diffusive one explains the observed behaviour.

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2.25 Effects of oxidative stress on physicochemical properties and disruption of polyunsaturated phospholipid membranes -

Dr. PARRA, Elisa (University of Copenhagen)

Oxidation of lipid membranes is widely known to play a crucial role in many cellular processes and pathological conditions such as apoptosis, inflammation, infection, and sepsis, through direct damage of the cell membrane. Both enzymatic and non-enzymatic oxidation, the latter occurring via direct exposure to reactive oxygen species (ROS), are pathways leading to lipid peroxidation in living cells and tissues. ROS attack lipids containing C-C double bonds, especially those containing polyunsaturated fatty acids (PUFA). This leads to lipid degradation into oxidation products such as truncated phospholipids or carbonyl compounds like malondialdehyde (MDA). PUFAs are very abundant in biological tissues, representing between 30-60% of the total fatty acid composition depending on the tissue and animal, so their inclusion in model systems designed to study oxidation of biologically relevant membranes is crucial. Amongst them, arachidonic acid, with 4 double bonds, is the most abundant species. In this study, we have used a combination of different experimental techniques in order to characterise the effect of oxidation on the structure, stability, and disruption of PUFA-containing membranes. Liposomes consisting of 1-palmitoyl-2-oleoyl-sn-glycero-3-phosphocholine/1-palmitoyl-2-arachidonoyl-sn-glycero-3-phosphocholine (POPC/PAPC) mixtures have been subjected to different oxidative conditions, including heat and UV exposure, in the absence or presence of H2O2. The liposomes were then tested using different fluorescence assays of lipid peroxidation and MDA production. They were further analysed in terms of size, surface charge, permeability, bilayer deposition, and stability using dynamic light scattering, fluorescent leakage assays, and quartz crystal microbalance with dissipation monitoring (QCM-D). In general, higher PAPC contents lead to higher lipid peroxidation, lower stability, and a greater tendency to disruption under oxidative stress. Furthermore, small angle X-ray scattering (SAXS) has been employed to investigate the bilayer structure and thickness of these systems. It was found that both increasing PAPC contents and UV exposure appear to induce bilayer thinning. Together, these experiments will establish the conditions to induce and quantify membrane oxidation in cell-like model membranes as a precursor to future experiments addressing nanoparticle-induced oxidative stress and disruption pathways.

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2.26 CoSAXS AT MAX IV Laboratory. First InstaLLATIONS, SAMPLE ENVIRONMENTS AND COMMISSIONING PLAN - Dr. PLIVELIC, Tomás Sigfrido (MAX IV Laboratory. Lund University)

The time for first light at the CoSAXS beamline is rapidly approaching and a major milestone in the project has been achieved with the initial installation of optical components at the beamline. The full optical and diagnostic element installation will be completed by Spring 2018. CoSAXS aims to take full advantage of the 3 GeV diffraction limited MAX IV design (low emittance and high brilliance) preserving the coherent properties of the source. The SAXS vacuum vessel construction has been awarded to an external company and this key project is moving forward towards the final design specifications with installation expected by Autumn 2018. The SAXS flight tube will be a 17 m long, 1 m diameter chamber with working vacuum pressures of 0.1 mbar and two in vacuum 2D position sensitive hybrid pixel X-ray detectors. The expected available q-range is projected to be 6 x 10-4 < q < 6 Å-1 (from 1 μm to 1 Å d-spacings) in simultaneous SAXS/WAXS detection. The experimental area is now in design but with significant contributions from the user community, specifically to bring together a suite of modern sample environments. These include: i) the high throughput BioSAXS set up (BioCUBE system from Xenocs, already in commissioning phase); ii) the new flow through cell for combined x-ray scattering/spectroscopy experiments (SUrF-II, collaboration project: CoSAXS and SWING at SOLEIL); iii) stand-alone devices such as stop-flow, heating stages, rheometers and microfluidics setups (MAX4ESSFUN collaboration sub-projects in 2016-2018). In IT developments, the beamline control will be based on Tango and Sardana with specific APIs for the different sample environment configurations. SAXS/WAXS data collection and data reduction software is Python based and undergoing testing. Commissioning activities with light is expected in November 2017 (for the insertion device and optics). First commissioning experiments and expert (friendly) user activities are planned for Q1-Q2 2019. One call for external users is expected in 2019.

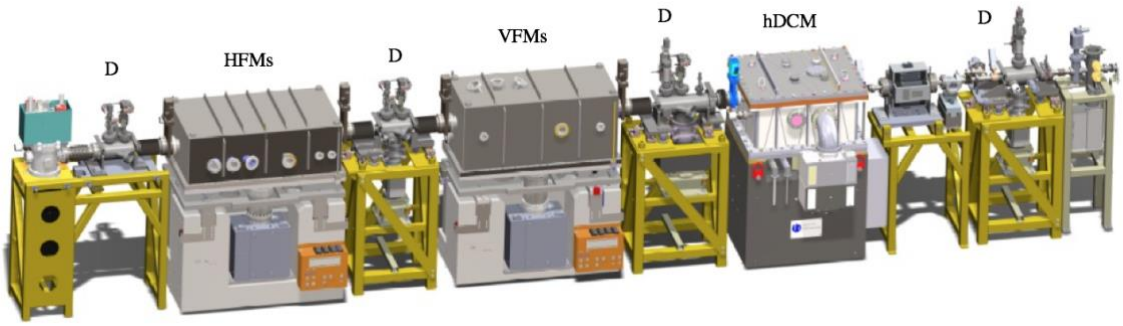

Figure 1: 3D drawing of the CoSAXS optical and diagnostic elements. hDCM: monochromator; VFMs:vertical focusing mirrors; HFMs: horizontal focusing mirrors.D: diagnostic units

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2.27 Mesoporous silica: in situ SAXS investigation of synthesis processes -

Ms. RASMUSSEN, Helena (Master student),
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In this study, synthesis routes for SBA-15 will be investigated, primarily using in situ small-angle X-ray scattering (SAXS). Furthermore, the effects of the swelling agents TMB (1,3,5-Trimethylbenzene) and TIPB (1,3,5-Triisopropylbenzene) are investigated when the swelling agent is added in several stages during the synthesis, together with the Pluronic P123 and tetraethyl orthosilicate (TEOS), which is the silica source for the formation of the ordered structure. The results from in situ measurements are further complemented by studies of nitrogen absorption, SAXS on the as-synthesized and calcined powder samples, transmission electron microscopy (TEM) and scanning electron microscopy (SEM). As will be shown, a detailed structural investigation is obtained for several synthesis conditions, indicating the correlation between the synthesis parameters and structural parameters obtained from the characterization techniques and advanced modeling of the SAXS data [1]. The information obtained from this study is very important since it enables a fine-tuning of the pore sizes and structural ordering for the hexagonal phase of this mesoporous material, with many potential applications [2,3].

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2.28 Orientation distribution of cellulose nanofibrils in material processes using small angle X-ray scattering -

Dr. ROSEN, Tomas (Chemistry Department, Stony Brook University, NY, USA; Linné FLOW Center and Wallenberg Wood Science Center, Royal Institute of Technology (KTH), Stockholm)

New advanced materials from cellulose nanofibrils (CNF) have recently shown great potential to become a key component meeting the demands of a sustainable society. Through flow-assisted assembly of CNF, there is also a possibility to control the structure of the material and thus also its macroscopic properties, as demonstrated by Håkansson **et al.** [1]. Understanding the orientational dynamics of dispersed nanofibrils in different flows is thus crucial for optimizing the material process. The orientation distribution function (ODF) of the CNF can be obtained using small angle X-ray scattering (SAXS) experiments. However, these experiments typically only provide the projected ODF in a plane perpendicular to the beam direction. Using numerical simulations, we demonstrate the problems that arise when comparing the projected alignment with the alignment based on the 3D orientation in different flows [2]. We also provide a simple method for reconstructing the three-dimensional ODF from the projected ODF, when fibril orientation symmetry can be assumed around the flow direction. Finally, the reconstruction method is used to revise the results by Håkansson **et al**. [1] as well as proposing a mechanism for the evolution of the ODF during drying of the material.

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2.29 Sample environment for soft condensed matter research at the ESS – Harald Schneider (European Spallation Source ERIC)

The FLUCO platform as part of the ESS sample environment will provide sample environment equipment and methods neutron scattering experiments on soft condensed matter. We present the plan for the available equipment for the start of the user operation, from all 3 parts, instrument specific, pool equipment and collaborational (external) equipment and methods.

2.30 Alkylglycoside surfactants with oligomeric head-group: investigation of self-assembly and its implications for future applications - Dr. SEBASTIANI, Federica (Physical Chemistry Department, University of Lund and 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 life sciences. 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. Light scattering showed the presence of large aggregates (RH ~ 60 nm), whereas data from NMR diffusometry are compatible with aggregates that are ten times smaller. SANS was crucial to collect information on the structure of both aggregate types at the same time, and to quantify their relative presence. The larger aggregates have a disc-like shape, most probably a portion of bilayer lamella, while the small aggregates have an elongated shape. Consequently, C16G8 is a surfactant in which two different modes of self-aggregation operate side by side. 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. 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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2.31 In Search of Novel Sample Environments and Next-generation Materials – SEGAD, Mo (Arrhenius Laboratory, Stockholm University, Advanced Light Source, Lawrence Berkeley National Laboratory, and X-ray Scattering Facility, Materials Research Institute)

Elucidating the structure and dynamics of polymeric-, protein- and hybrid-based materials is an under explored area that needs to be addressed further to solve many challenging problems in designing next-generation materials. Targeting these materials, using hard/tender/soft X-ray range and advanced sample environments along with real-time data treatment can probe the complexity of nano- and meso-scale materials. Combining tender resonant X-ray scattering with tender X-ray absorption spectroscopy, at the newly developed end-station TReXS, is a unique chemical sensitive structure probe to identify the chemical components of multi-component systems. Tuning X-ray photon energies to match the absorption spectrum of different chemical components, the scattering contributions from different components can also be selectively enhanced, enabling a better characterization of the complex morphologies with more details. In this talk, I will briefly describe the capabilities of TReXS beamline that has recently been developed at the Advanced Light Source (ALS) in Berkeley California. I will also present scattering results for some polymer, bio and multi-component systems, collected at the ALS, LCLS, ESRF, NSLS-II and ORNL, as the objective of this talk is not only to describe TReXS beamline and the advanced sample environments but also to promote collaboration among researchers in different disciplines particularly in soft matter.

2.32 Microfluidics with in-situ SAXS to probe the time evolution of the lamellar-microemulsion transition induced by a concentration jump - Dr. SILVA, Bruno (INL - International Iberian Nanotechnology Laboratory)

The use of microfluidic devices with in-situ small-angle X-ray scattering (SAXS), offers new interesting possibilities for the study of soft materials under out-of-equilibrium conditions [1,2]. In particular, when flowing in micron-sized channels, fluids become easier to manipulate, allowing an experimental control (e.g. rate of mixing, shear rate, concentration gradients, confinement) and reproducibility, that has been previously unavailable, and opening the possibility for new experiments. In this work we study the SDS-pentanol-water ternary system's lamellar to oil-in-water (o/w) and lamellar to water-in-oil (w/o) microemulsion transitions, induced by mixing a lamellar phase with water or pentanol in a crossed microchannel configuration. By manipulating the individual flow-rates, one can carefully tune the final composition following the concentration jump, and furthermore, probe different time-scales of the transition. The ongoing structural evolution is simultaneously monitored in-situ with SAXS. The main findings show that the lamellar to o/w droplets transition (by mixing with water) occurs through a gradual stripping down of bilayers from the lamellar phase, with a microemulsion SAXS signature coexisting with the initial lamellar peak since very early mixing times. Conversely the lamellar to w/o reverse droplets transition (through mixing with pentanol) involves the formation of an intermediate lamellar phase with a smaller spacing before giving place to the reverse droplets.

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2.33 Salt-induced temperature-dependent protein cluster formation: access to binding entropies and enthalpies - Mr. BECK, Christian (ILL)

With increasing Yttrium Chloride (YCl3) salt concentration cs , aqueous Bovine Serum Albumin (BSA) protein solutions subsequently change from a visually transparent (regime I) to a turbid phase (regime II) and back to a transparent phase (regime III: reentrant dissolution). Within regime II, a lower critical solution temperature (LCST) associated with a liquid-liquid phase separation (LLPS) can be observed. Quasi-elastic neutron scattering (QENS) data as a function of cs and the protein concentration cp lend support to the formation of protein clusters when approaching regime II [1]. By applying the Wertheim theory for patchy particles [2] and the Flory-Stockmeyer cluster size distribution function, it is possible to describe the temperature dependent cluster formation quantitatively [3]. The ion-binding and protein- protein bridging entropies and enthalpies associated with the clusters can be determined directly from a simultaneous fit of the model to a large set of QENS spectra for different cp , cs , and T. The results are compared with calorimetry measurements [4].

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2.34 Vapor sorption of polar solvents by graphene oxide films studied by in-situ neutron reflectivity - Dr. TALYZIN, Alexander (Umeå University, Department of Physics)

Permeation of multilayered graphene oxide (GO) membranes by vapors and liquid polar solvents is known to correlate with swelling properties and amount of sorbed solvent. However, quantitative estimation of sorption using standard (e.g. gravimetric) methods is technically challenging for few nanometers thick GO membranes/films. Neutron reflectivity (NR) method provides unique opportunity to study sorption properties of rather thin films which consists of only 25-35 of GO layers. Analysis of NR data recorded from GO film exposed to vapors of polar solvents provide information about change of film thickness due to swelling, amount of intercalated solvent and selectivity in sorption of solvents from binary mixtures. Our earlier study reported swelling properties of GO films exposed to D2O, ethanol and D2O–ethanol vapors. It was found that the solvent adsorbed by the GO film is D2O enriched compared to the composition of mixed D2O–ethanol solution. 1 Recently we extended this study to include several other solvent and binary solvent mixtures. Quantitative study of GO film sorption was performed for D2O, d-methanol, ethanol, dimethyl sulfoxide (DMSO), acetonitrile, dimethylformamide (DMF) and acetone. Using isotopic contrast we estimated selectivity in sorption of ethanol/ d-methanol mixtures by the GO film. Estimation of sorption selectivity was also performed for D2O/acetonitrile, D2O/dimethylformamide, D2O/dimethyl sulfoxide and D2O/acetonitrile binary mixtures.

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2.35 Interfacial behavior of lipid sponge-like nanoparticles on hydrophilic silica surface and their interactions with proteins - Ms. VALLDEPERAS, Maria (Lund University)

The interest in using nonlamellar lipid liquid crystalline phases has grown in many applications, such as drug delivery, protein encapsulation or crystallization. However, it is still challenging to form inverse mesophases with large aqueous pores able to encapsulate large bioactive molecules. Here, we present a novel lipid system able to form sponge phases (L3) with water pores up to 12 nm of diameter. We will also unveil the interfacial properties of sponge –like nanoparticles (L3 NPs) formed by the L3 phase in excess water on hydrophilic silica. Finally, preliminary results on the adsorption of two enzymes, aspartic protease (34 KDa) and beta-galactosidase (465 KDa), on the lipid layer formed by L3 NPs will be also shown.

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2.36 Interpretation of the SAXS from cellulose microfibrils as ensembles of rods - Mr. VELICHKO, Evgenii (Delft University of Technology)

Nanocomposite materials have a variety of applications ranging from food and cosmetics to wings of a space shuttle. Cellulose microfibrils can form a scaffold in such nanocomposites, thus defining such properties of the materials as rigidity, tensile strength, Young's modulus and yield stress. The crucial property of microfibrils is their high aspect ratio, which can reach values of several thousand. What makes these particles even more attractive is their natural origin, renewability, and environmental friendliness.

Mechanical properties of the nanocomposite material are defined by its internal arrangement on the nanometer scale. However very few techniques can provide information about this arrangement within intact samples in their native, often wet state. Small-angle X-ray scattering(SAXS) can provide structural information in the range of length scales from several Angstrom up to several micrometers. Moreover, SAXS can be applied to the materials in their native state, without disturbing their structure. The main obstacle in the scattering data interpretation is so-called "phase problem" – loss of part of the structural information in the scattering experiment. A possible way of overcoming this problem is to assume possible structures and compare the calculated and experimental scattering curves.

In the presented work we developed several scattering models based on elementary fibrils as building blocks of microfibrils. The elementary fibrils were arranged with their axes parallel to each other and forming various cross-sectional structures, which were averaged azimuthally. The microfibrils, formed in this way, were consequently averaged over all orientations. This approach yield analytical solutions for structure factors describing various arrangements of the elementary fibrils in the samples. The models derived were successfully applied to the suspensions of microfibrils varying in concentrations and the way of preparation.

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2.37 Recent upgrade of the polarized neutron reflectometer Super ADAM @ ILL - Dr. VOROBIEV, Alexei (Uppsala University)

The upgraded Super ADAM reflectometer is a Swedish infrastructure operated at Institut Laue-Langevin, Grenoble, France. The instrument is characterised by an unrivalled flexibility in incident beam optics, sample environment and detector settings, enabled by its modular design and large accessible floor space. The redesign of the instrument took an advantage of the major reconstruction of the ILL22 experimental hall allowing the completion of a high flux mode, which is currently under commission. The improved flux combined with the outstanding resolution and polarisation (99.8% incoming and 99.4% outcoming) has significantly strengthened the performance of the instrument. The high-resolution mode of the instrument offers therefore already unique research opportunities for investigations of magnetic thin films and multilayers. Several examples of recent studies will be presented, including soft matter studies on solid-liquid interfaces, demonstrating the capabilities of the instrument. 1 A. Vorobiev, et al. Neutron News, Volume 26, Number 3, 2015 Super ADAM.

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2.38 A Microfluidic platform for the continuous production and characterization of lipid nano-self-assemblies -

Dr. YAGHMUR, Anan (Department of Pharmacy, Faculty of Health & Medical Sciences)

Microfluidic devices have gained popularity for a wide range of technological applications including tissue engineering and organ-on-a-chip. Microfluidic devices are also attractive and promising platforms for pharmaceutical applications. They have been used to form lipidic and polymeric nanoparticulate formulations such as liposomes with controllable size by mixing miscible liquids and/or controlling the diffusion process by implementing hydrodynamic flow focusing (HFF) mixing. Combined with advanced analytical techniques such as X-ray and Neutron scattering (SAXS and SANS), microfluidic platforms allow the online study of protein aggregation and continuous producing and real-time tracking of lipid nano-self-assemblies. For instance, real-time monitoring of chemical reactions can be performed by the combination of SAXS with a suitable microfluidic device that allows adjusting the mixing time down to a few tens of microseconds. In this contribution, we present our recent investigations on the combination of microfluidics with synchrotron small angle X-ray scattering (SAXS) to monitor the early dynamic structural features occurring during the continuous production of nano-self-assemblies. We report also on the dependence of the nanoparticle size and size distribution on the microfluidic device geometry, the flow rate ratio (FRR) which is the ratio of sheath to center stream, and the total volumetric flow rate (TFR). Moreover, we present our recent study on monitoring the effect of mixing calcium ions with negatively charged cubosomes.

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