Anniversary Symposium

"SAXS on Nanosystems: current trends and perspectives"
20 years of the Austrian SAXS Beamline at Elettra-ST

Trieste, Italy, October 10th – 12th, 2016

Scientific Committee
Chair: Heinz Amenitsch (Graz University of Technology)
Sigrid Bernstorff (Elettra-Sincrotrone Trieste)
Wim Bras (Netherlands Organization for Scientific Research)
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Peter Müller-Buschbaum (Technical University of Munich)
Lois Pollack (Cornell University)

Plenary Speakers
Peter Fratzl (Max Planck Institute of Colloids and Interfaces)
Stephan Roth (DESY)
Jean-Baptiste Salmon (CNRS, Univ. Bordeaux)

Invited Speakers:
Maja Buljan (Ruder Bošković Institute)
Pavo Dubček (Ruder Bošković Institute)
Stephan Förster (Univ. of Bayreuth)
Oskar Paris (Montanuniversität Leoben)
Jan Skov Pedersen (Univ. of Aarhus)
Javier Perez (Synchrotron SOLEIL)
Gianluca Grenci (MBI, T-Lab)

TU Graz
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Elettra Sincrotrone Trieste

Austrian SAXS beamline
Outstation of the Institute of Inorganic Chemistry
Graz University of Technology
c/o ELETTRA Sincrotrone Trieste
Strada Statale 14, km 163.5
34149 Basovizza, (TS) Italy

Graz University of Technology
Institute of Inorganic Chemistry
Stremayrgasse 9/IV
A-8010 Graz, Austria

and
ELETTRA-Sincrotrone Trieste
Strada Statale 14, km 163.5, in Area Science Park
34149 Basovizza, (TS) Italy

VENUE

ICTP - Budinich Lecture Hall
The Abdus Salam International Centre for Theoretical Physics
Strada Costiera 11,
34014 Trieste, Italy

	latitude	**longitude**
GD	45.703054886014236	13.71818482875824
GMS	N 45° 42' 10.998''	E 13° 43' 5.465''

Full program download at:

https://www.elettra.eu/Conferences/2016/SAXS20/Main/Program

"SAXS on Nanosystems: current trends and perspectives" - 20 years of the Austrian SAXS Beamline
Symposium program

Monday 10th				
Time		**Topic**	**Name**	**Title**
13:00	14:00	**REGISTRATION**		
14:00	14:40	opening remarks	**Alfonso Franciosi (President & CEO, ELETTRA-Sincrotrone Trieste)** **Horst Bischof (Vice Rector Research, TU Graz)** **Frank Uhlig (Dean TCVB & Head of Institute for Inorganic Chemistry, TU Graz)** **Heinz Amenitsch (Institute for Inorganic Chemistry, TU Graz)**	
14:40	15:20	**Plenary lecture**	Peter Fratzl	SAXS and complex biomaterials - Perspectives for synchrotron-based multiprobe imaging
15:20	15:50	**Invited lecture**	Peter Laggner	Structure and Change. A Personal Recollection of 50 Years with Bio-SAXS
15:50	16:10	BREAK		
				Chair: Heinz Amenitsch
16:10	16:25	NFFA	Giorgio Rossi	NFFA-EUROPE: An open access resource for experimental & theoretical science
16:25	16:40	CERIC	Jana Kolar	CERIC-ERIC: Future perspectives
16:40	17:10	**Invited lecture** SAXS	Jan Skov Pedersen	The new laboratory SAXS instrument at Aarhus University: Optimization, implementation and application to studies of low density lipoprotein (LDL) particles including a newly developed model
17:10	17:30	SAXS	Herwig Peterlik	In-situ X-ray scattering of single carbon fibers
17:30	17:50	SAXS	Michael Rappolt	The Art of Filling Space: Towards Understanding of Lipid Polymorphism
17:50	18:10	SAXS	Aldo Craievich	Size dependent melting and freezing temperatures and other properties of Bi nanoparticles confined in a glass matrix. Study by combined use of SAXS and WAXS
18:10	21:00	POSTER SESSION		

Tuesday 11th	MORNING SESSION			

Time		Topic	Speaker	Title Chair: Benedetta Marmiroli
9:00	9:40	**Plenary lecture** µfluidics	Jean-Baptiste Salmon	Pervaporation at the nanoliter scale: Microfluidic tools to investigate complex fluids and engineer micro-materials
9:40	10:10	**Invited lecture** µfluidics	Stephan Förster	Flow induced assembly processes in microchannels and microjets
10:10	10:30	µfluidics	Jens Meissner	Eutectic Crystallization of Salt Solutions in Nanopores: Accessing the Properties of the Crystallites by in-situ SAXS/WAXS
10:30	10:50	BREAK		
				Chair: Manfred Kriechbaum
10:50	11:20	**Invited lecture** GISAXS	Pavo Dubceck	Annealing Induced Evolution of Germanium Quantum Dots
11:20	11:40	chemistry	Christian Doonan	MOFs at the Biointerface
11:40	12:00	chemistry	Paolo Falcaro	Metal-Organic Frameworks from ceramics
12:00	12:20	chemistry	Ana Torvisco	From Aryltin Trihydrides to Nanosized Polymers- Solvent and Residue Effects on Material Morphology
12:20	12:40	chemistry	Attilio Cesaro	Nanoscale fractal aggregates of caffeine in hot-coffee conditions
12:40	13:00	pharmacy	Alexander Pichler	Structure Analysis of Drug Delivery Systems with SAXS in the Laboratory
13:00	14:00	LUNCH		

Tuesday 11th	AFTERNOON SESSION			
Time		**Topic**	**Speaker**	**Title** **Chair: Marcell Wolf**
14:00	14:30	**Invited lecture** chemistry	Oskar Paris	In-operando SAXS for Energy Applications
14:30	14:50	bioSAXS	Tobias Madl	Structural Characterization of Challenging Biomolecular Complexes by Integration of SAXS with Complementary Techniques
14:50	15:10	bioSAXS	Maria Grazia Ortore	From protein-protein interactions to amyloid aggregation: SAXS plays as an outsider
15:10	15:30	BREAK		
15:30	16:00	**Invited lecture** bioSAXS	Javier Perez	Synchrotron SEC – SAXS data as EXPERIMENTAL CONSTRAINTS to model the detergent corona around a membrane protein
16:00	19:00	VISIT AT THE AUSTROSAXS BEAMLINE AT ELETTRA-SINCROTRONE TRIESTE		
20:00	open	SYMPOSIUM DINNER, Antica Trattoria Menarosti, Via del Toro 12, 34125 Trieste		

Wednesday 12th	MORNING SESSION			
Time		**Topic**	**Speaker**	**Title** **Chair: Sigrid Bernstorff**
9:00	9:40	**plenary lecture** GISAXS	Stephan Roth	Investigating rapid technological coating processes in real-time and in situ
9:40	10:00	GISAXS	Fernando Cacho-Nerin	µGISAXS on curved fluid-fluid interfaces: Following particle rearrangement and ejection upon surface compression
10:10	10:40	**Invited lecture** GISAXS	Maja Buljan	Application of GISAXS on study of three-dimensional quantum dot lattices
10:40	11:00	BREAK		

				Chair: Michael Rappolt
11:00	11:20	SAXS	Milos Steinhart	The performance of the High Pressure SWAXS System designed for the Austro-SAXS Beamline
11:20	11:40	bioSAXS	Michal Belička	Biomembrane Complexity at the Sub-Nanometer Level
11:40	12:00	bioSAXS	Domenico Lombardo	Interaction of Charged Dendrimers with Model Lipid Membrane: a SAXS Study
12:00	12:20	bioSAXS	Stefan Salentinig	In-situ small angle X-ray scattering reveals formation of highly organised nanostructures during digestion of milk fat
12:20	12:50	**Invited lecture** µfluidics	Gianluca Grenci	Easy and fast fabrication of a free-jet micromixer for SAXS
12:50	13:00	concluding remarks	Heinz Amenitsch	

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Lectures and Talks

PL1 SAXS and complex biomaterials - Perspectives for synchrotron-based multiprobe imaging

Peter Fratzl

Department of Biomaterials, Max Planck Institute of Colloids and Interfaces, Research Campus Golm, 14424 Potsdam, Germany

The permanent adaptation of tissues in animals or plants, including in our own body, creates materials with extreme heterogeneity in the three space dimensions and in time. In order to improve our understanding of biological materials such as bone, teeth, tendons or other organs, biological approaches need to be complemented by imaging techniques that provide multi-scale and multimodal information about structure, composition and physical properties. While synchrotron radiation is not generally suited for in-vivo imaging, it provides an exceptionally flexible tool to image composition, structure and properties in several dimensions, combining scanning-SAXS, x-ray diffraction, spectroscopy and tomography. The talk will showcase several examples, including bone from our skeleton, where scanning-diffraction and spectroscopy, in combination with tomography improves our understanding of the material and the effect of disease and treatment. Further development of these synchrotron-based methodologies is under way and this will provide a unique opportunity for research in biomaterials and in complex material systems in general

IL1 Structure and Change: A Personal Recollection of 50 Years with Bio-SAXS

Peter Laggner, Graz
Consultant to Bruker-AXS, Karlsruhe, and RCPE, Graz

Especially in the life sciences, where structure and change are the essence of being, SAXS has always played a key role, and continues to do so with ever more powerful techniques, such as synchrotron radiation. The cross-fertilization between synchrotron radiation sources and biosciences is a historical fact: promises of bio-medical breakthroughs have always been the key arguments to get the required funding, and – on the other hand - many important new developments in our knowledge have indeed become possible only through synchrotron SAXS. In fact, technological progress in X-ray sources and optics, detectors, computing, emerged closely in parallel to new scientific questions.
From the tedious beginnings in the Sixties with antibody-antigen complexes (at the famous Kratky camera), via blood lipoproteins, membranes, to the mechanisms of liquid crystal phase transitions and sub-millisecond SAXS cinematography at synchrotrons, the lecture will present a review of the success-story of SAXS: From the poor sister of X-ray crystallography to the quick and versatile, multivalent lady.
In more recent years, the technology gained at synchrotron facilities has started to feed back to laboratory instrumentation, to make the benefits of SAXS also available to daily analytical routine in research institutions and industry.

T1a NFFA-EUROPE: An open access resource for experimental & theoretical science

Giorgio Rossi

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CNR-IOM, CNR-IOM, Laboratorio TASC, Strada Statale 14 km 163,5 Basovizza, I-34149 Trieste ITALY

NFFA-EUROPE is an open access platform, granted by the EU for 48 months from September 1st, 2015, to carry out comprehensive projects for multidisciplinary research at the nano-scale extending from synthesis to nano-characterization to theory and numerical simulation.
Advanced resources, made available by the 20 NFFA-EUROPE partners, specialized on growth, nano-lithography, nano-characterization, theory and simulation and fine-analysis with Synchrotron radiation, Free Electron Laser (FEL) and Neutrons are integrated to develop frontier research on nanoscience and to enable European scientists from diverse disciplines to access state of the art and unique methods and tools.
NFFA-EUROPE enables coordinated access to nanoscience laboratories co-located at the large-scale infrastructures for fine analysis, or linked to High-Performance Computing (HPC) facilities as well as Joint Research Activities (JRA) and Networking Activities (NA).
The access management structure optimizes the services to the users to pursue scientific excellence as well as industrial and technological innovation. Proposals can be submitted through the *single entry point* at the NFFA.EU portal to apply for all NFFA-EUROPE methods and instruments, and a panel of international experts is in charge of the peer-review selection to ensuring the scientific excellence and/or innovation potential of the accepted proposals. Moreover, the experts of the Technical Liaison Network (TLNet) dialogue with and assist the user from the proposal submission to the technical feasibility check and to the personalized access programme optimizing the use of the NFFA-EUROPE infrastructure.
Within the NFFA-Europe offer, SAXS facilities are provided by Graz University of Technology (in both Graz and Trieste), DESY and SOLEIL. Additional informations are available at www.nffa.eu.

T1b **CERIC-ERIC: Future perspectives**

Jana Kolar[1], Ornela de Giacomo[1]
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Material science and nanotechnology are two of the main research fields to take up future challenges of Europe such as alternative energy sources and energy storage or biomedical and pharmaceutical materials. The scientific problems coming up in this fields have become more and more complex in the recent years and require an ever increasing number of instrumental and analytical techniques and disciplines. Such complexity requires the availability of expertise as well as open access to a wide range of probing techniques and many different complementary instruments.
To face this challenge Austria became a founding member of CERIC-ERIC, a research infrastructure that allows to access many complementary instruments (Fig. 1) with a single proposal. CERIC offers free open access based on peer-review for the best researchers from all over the world.
CERIC was set up as a European Research Infrastructure Consortium (ERIC) [2] and brings together excellent research facilities from Austria, Croatia, Czech Republic, Hungary, Italy, Poland, Romania and Slovenia. All partners offer free and open access CERIC-ERIC comprises synchrotron radiation, neutron radiation, microscopic techniques, ion-beam analysis methods and NMR.
Austria participates in CERIC through the TUGraz, in particular with it the AustroSAXS beamline, DXRL and the scattering facilities in Graz.

CERIC, its present instruments and possibilities for researchers will be presented, as well as the future perspectives.

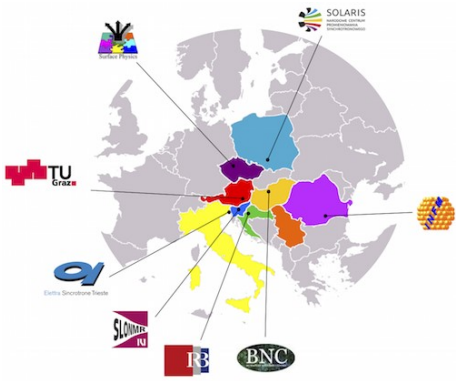

Fig 1: Members and Representing Entities of CERIC-ERIC.

References:

[1] Commission Implementing Decision, *Official Journal of the EU*, L184/49, **2014**
[2] Council Regulation (EC) No 723/2009 of June 25th **2009**.

IL2 The new laboratory SAXS instrument at Aarhus University: Optimization, implementation and application to studies of low density lipoprotein (LDL) particles including a newly developed model

Jan Skov Pedersen

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A new SAXS NanoSTAR (Bruker AXS) with a liquid metal Ga jet source has recently been installed at Aarhus University [1]. The instrument employs a long high-quality parabolic Montel multilayer optics from Incoatec and a compact scatterless pinhole slit with Ge edges built at Aarhus University. The combination of the powerful source and the optimized geometry gives an integrated X-ray intensity close to 10^9 photons/s for a standard range of scattering vector moduli, $q = 0.01–0.35$ Å^{-1}, where $q = (4\pi \sin\theta)/\lambda$, where λ is the Ga Kα wavelength of 1.34 Å and 2θ is the scattering angle.

The high intensity of the instrument means that even dilute samples of less than 1wt% of protein or surfactants can be measured in a few minutes. Therefore, in order to get an efficient use of the instrument a flow-through cell (built at Aarhus University) in combination with an automated sample handler has been installed at the instrument. The sample handler is based on the commercial Gilson GX-271 Injection system (delivered by Biolab, Risskov, Denmark), which also allows the samples to be stored under thermostated conditions. The sample handler inserts and removes samples, as well as cleans and dries the cell between measurements. The minimum volume of the flow-through capillary is about 25 μL.

A recent application of the instrument in a study of LDL particles as a function of temperature is described [2]. It demonstrates that high quality data can be obtained for the low concentration samples. A new semi-analytical SAXS model based on recent cryo-TEM results has been derived, which is fundamentally different from previous centro-symmetric layered models. The new model was fitted to the data providing detailed information on the melting of fatty core and the related cholesterol packing.

References

[1] J.S. Pedersen, J. Lyngsø, E. Konusch, and L. Bruegemann. High-flux SAXS instrument with liquid metal jet source and an automated sample handler and a stopped-flow apparatus (in preparation)

[2] S. Maric, J. Lyngsø, M. Cárdenas and J. S. Pedersen. A new semi-analytical model for simultaneous analysis of the inner and outer structure of low density lipoproteins using Small-Angle X-ray Scattering – insights into the fatty core phase transition and cholesterol packing.

T2 In-situ X-ray scattering of single carbon fibers

Harald Rennhofer[1], Oskar Paris[2], Herwig Peterlik[3]

[1]*University of Natural Resources and Life Science, Institute of Physics and Material Sciences, Peter-Jordan-Strasse 82, A-1190 Vienna, Austria*
[2]*University of Leoben, Institute of Physics,Franz-Josef-Straße 18, A-8700 Leoben, Austria*
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The internal structure of carbon fibers (CF) consists of interconnected nanocrystallites with a different degree of order. In modern words, they are a typical example for multi-layer graphene. As CF are available with constant quality due to the wide use as main load bearing component in composites, they are an ideal model material to study load and temperature dependence of carbon nanostructures. Whereas only fiber bundles can be investigated in a laboratory X-ray source, a microfocus from a synchrotron radiation source allows the in-situ measurement of single fibers, either with high position resolution in real space or for following structural evolution of carbon nanostructures during loading and heating. We present here an overview on *in-situ* SAXS and WAXS experiments, which allow obtaining information on both pore and crystallite structure. This ranges from structural asymmetry due to asymmetric loading conditions [1] up to structural dynamics at temperatures up to 2000 ^{o}C {2,3].

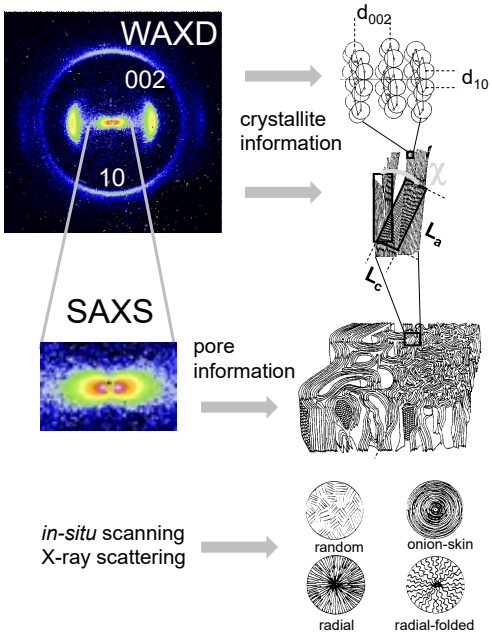

Figure 1: figureTypcial SAXS and WAXS image of a carbon fiber and the corresponding structure.

References:

[1]. D.Loidl, O.Paris, M.Burghammer, C.Riekel and H.Peterlik, Phys. Rev. Lett. **95** (2005) 225501.

[2] H.Rennhofer, D.Loidl, S.Puchegger, and H.Peterlik, Carbon **48** (2010) 964-971.

[3] H.Rennhofer, S.Puchegger, S. Pabisch, C. Rentenberger, C.H. Li, S. Siegel, A. Steiger-Thirsfeld, O. Paris, and H.Peterlik, Carbon **80** (2014) 373-381.

T3 The Art of Filling Space: Towards Understanding of Lipid Polymorphism

Michael Rappolt[1]

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In this presentation an overview on the most prominent lyotropic lipid phases shall be given, i.e. discussing various nanostructural water-in-oil (w/o) and oil-in-water (o/w) phases. This includes elucidating the mechanical and structural properties of planar membranes, bicontinuous cubic phases, columnar/tubular phases and several micellar aggregation forms. Alongside fundamental bending and packing concepts are explained and latest developments in X-ray scattering techniques will be highlighted. Especially using time-resolved, simultaneous small- and wide-angle synchrotron X-ray scattering, the formation pathways of these nanostructures and liquid to solid-state formations can be revealed in great detail.

T4 Size dependent melting and freezing temperatures and other properties of Bi nanoparticles confined in a glass matrix. Study by combined use of SAXS and WAXS

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Nanomaterials that can easily be studied by SAXS for quantitative and precise characterization of phase transitions are those composed of a dilute set of spherically shaped nanoparticles with identical sizes. In a recent study, a method exclusively based on SAXS was used for the determination of the melting and freezing temperatures (T_m ad and T_f, respectively) of a dilute set of Pb nanoparticles with a narrow radius distribution embedded in a glass matrix [1].

For the investigation described here, several glass samples containing Bi nanoparticles were prepared as described in a previous work [2]. This led to a set of glass samples, each of them containing spherical Bi nanoparticles with several average radii <R> and different radius distributions N(R).

The present study allowed us to determine the transition temperatures of Bi nanoparticles over a very wide radius range, from 1 to 10 nm circa. The experimental results confirm previous conclusions indicating a continuous decrease of both, T_m and T_f, for decreasing radius, and linear dependences of T_m and T_f versus 1/R functions, over a (1/R) range much larger than that of previous work [3]. The results of the present investigation agree with those of the classical Couchman and Jesser theory [4], which predicts the decrease of melting and freezing temperatures of nanocrystals and linear dependences for increasing 1/R. The straight lines in T_m versus 1/R and T_f versus 1/R plots meet at R=1.8 nm, which corresponds to a critical radius at which undercooling vanishes. This implies that in Bi nanoparticles with a radius R<1.8 nm neither crystal-to-liquid nor liquid-to-crystal transitions occur and suggests that Bi nanoparticles below the melting point are not totally crystalline. The absence of undercooling in Bi nanoparticles with R<1.8 nm can be explained by assuming that all nanoparticles below their melting points exhibit a complex structure which can schematically be described as consisting of a crystalline core with different sizes surrounded by a 1.8 nm thick disordered shell.

These experimental results confirm previous conclusions about the radius dependence of the transition temperatures and their agreement with theoretical predictions, in the current case for a much wider radius range, and allowed us to quantify the reproducibility of SAXS/WAXS experimental results for a number of different samples. The present investigation also verified the robustness of the SAXS/WAXS procedure previously proposed for the determination of the melting and freezing temperatures for dilute and polydisperse sets of spherical nanoparticles. Additionally, from WAXS results it was demonstrated that both lattice parameters of Bi nanocrystals and their respective temperature dependences exhibit clear differences with respect to those of bulk Bi crystals

[1] G. Kellermann, A. Gorgeski, A.F. Craievich, L.A. Montoro. J Appl Cryst. **48**, 520 (2015).

[2] G. Kellermann, A.F. Craievich. Phys Rev B. **65**. 134204 (2002).

[3] G. Kellermann, A.F. Craievich. Phys Rev B. **78**, 054106 (2008).

[4] P.R. Couchman, W.A. Jesser. Nature. **269**, 481 (1977).

PL2 Pervaporation at the nanoliter scale: Microfluidic tools to investigate complex fluids and engineer micro-materials

Jean-Baptiste Salmon
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We exploit water pervaporation though dense poly(dimethylsiloxane) membranes embedded in microfluidic devices to concentrate solutes or particles at a controlled pace within microchannels [1]. These tools enable us to dynamically "explore" the phase diagrams of complex fluids at the nanoliter scale, from copolymer solutions to colloidal dispersions, and to measure out-of-equilibrium transport coefficients [2,3,4].

Ultimately, this continuous concentration process may lead to the formation of micro-materials (colloidal crystals, dense assemblies of nanoparticles, polymer composites…) which grow within the network of microfluidic channels [5,6,7]. Combining microfluidic pervaporation and Quake valves makes also possible the engineering of micro-materials with controlled and programmable composition gradients [8].

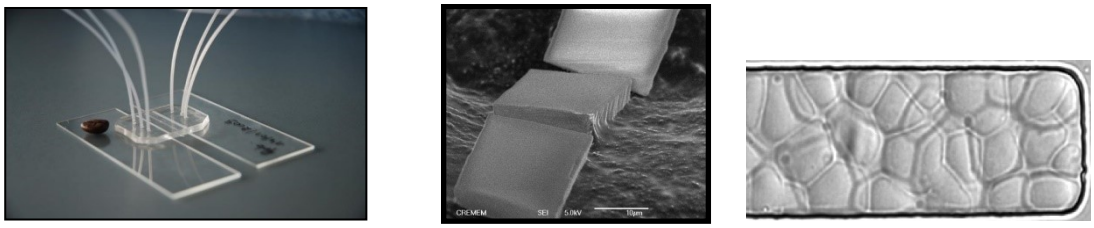

Figure: Left: microfluidic pervaporation for phase diagram screening. Middle: SEM view on a dense assembly of gold NPs. Right: nucleation and growth of colloidal crystals in a microfluidic pervaporation channel.

References:

[1] J. Leng, B. Lonetti, P. Tabeling, M. Joanicot, A. Ajdari, Phys. Rev. Lett. **96,** 084503 (2006)

[2] L. Daubersies, J. Leng, J.B. Salmon, Lab Chip **13,** 910 (2013)

[3] N. Ziane, M. Guirardel, J. Leng, J.B. Salmon, Soft Matter **11,** 3637 (2015)

[4] N. Ziane, J.B. Salmon, Langmuir **31,** 7943 (2015)

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[6] J. Angly, A. Iazzolino, J.B. Salmon, J. Leng, S. Chandran, V. Ponsinet, A. Désert, A. Le Beulze, M. Tréguer-Delapierre, and M. Correa-Duarte , ACS Nano **7,** 6425 (2013)

[7] C. Laval, P. Poulin, and J.-B. Salmon, Soft Matter **12,** 1810 (2016)

[8] C. Laval, A. Bouchaudy, J.B. Salmon, Lab Chip **16,** 1234 (2016)

IL3 Flow induced assembly processes in microchannels and microjets

Stephan Förster, Mathias Schlenk, Eddie Hofmann, Carolin Fürst
Physical Chemistry I, University of Bayreuth, Germany

Microfluidics emerges as a versatile sample environment for *in-situ* SAXS/WAXS-investigations of fast structural changes. We investigate the flow-induced orientation of anisotropic particles in microchannels and study fast nucleation & growth, the kinetics of amphiphile self-assembly and fiber-formation in hydrodynamic flow-focusing experiments.

For anisotropic cylindrical (1D) and sheet-like (2D) particles we observe that particles orient perpendicular to the flow-orientation after passing narrow channel sections. This is caused by the strong extensional forces perpendicular to the flow direction in the downstream widening section of the channel. The detailed flow-induced orientational distribution of the particles can be followed *in-situ* by scanning microfocus-SAXS at dedicated synchrotron beamlines.

We further investigate the kinetics of self-assembly of low molecular and polymeric amphiphiles, and study the nucleation and growth of spherical and cylindrical nanoparticles by using flow-focusing or stopped-flow SAXS/WAXS/UV-Vis experiments. Here we can test predictions from classical nucleation and growth theory and find for the formation kinetics of gold nanoparticles and nanorods good agreement between experiments and theory.

Using capillaries and microfluidic devices it is possible to generate free microjets. Using scanning microfocus-SAXS we can map the orientational distribution of anisotropic particles in free jets which is interesting as the flow field in free jets differs fundamentally from the parabolic flow fields in channels. Free microjet devices offer new possibilities for future studies at free-electron lasers, where versatile sample delivery devices are needed.

T5 Eutectic Crystallization of Salt Solutions in Nanopores: Accessing the Properties of the Crystallites by *in-situ* SAXS/WAXS

Jens Meißner, Albert Prause, Gerhard H. Findenegg

Stranski Laboratory of Physical and Theoretical Chemistry, Technical University Berlin, Berlin, Germany.

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Phase transitions of pure compounds under confinement has been in the scientific focus for years and much theoretical and experimental work has been done to understand the fundamental differences to freezing/melting in bulk.[1,2] Much less effort has been devoted to the phase transition of multicomponent systems at the nanoscale. Salt crystallization in nanoporous matrices is of great importance to multiple fields, such as salt impregnated advanced adsorbents and catalysts, thermal weathering of building materials and cultural heritage.
Recently, we could show that the crystallization of eutectic alkali halide solutions inside the porous matrix of SBA-15 and MCM-41 silica differs strongly from the bulk behavior.[3] This study highlights the implications of confinement on the phase transition at a nanoscale, but also reveal the necessity to clarify important aspects. Intriguing open questions are:

(i) What is the size of the crystallites and does it depend on the pore size?
(ii) How are the crystallites distributed in the pore space?
(iii) Which salt (hydrate) is formed in the pores?

In order to address those open questions we employed the AustroSAXS at the Elettra Synchrotron in Trieste. Using a temperature-controlled sample stage, we were able to record *in-situ* SAXS and WAXS intensity profiles simultaneously, covering a temperature interval from 130 to 290 K. A combination of both, small and wide angle scattering information gives a comprehensive picture about the phase transition in the system. The WAXS data provides information about the crystal structure and show transitions between different hydrates. On the other hand, synchrotron SAXS allows us to monitor several orders of Bragg reflexes with a very high q-resolution. The Bragg pattern originates from the regular 2D hexagonal pore structure and contains information about the lattice constant and the scattering contrast. By evaluating the relative intensity of each Bragg reflex we will be able to gain information about the freezing induced change in the scattering length density inside the porous matrix.[4] This will allow us to locate the salt crystallites inside the pore matrix.
In this contribution we will summarize the effects caused by secondary confinement in narrow pores and present how synchrotron x-ray scattering techniques can develop a much more in-depth view for several aspects.

References:

[1] C. Alba-Simionesco, B. Coasne, G. Dosseh, G. Dudziak, K. E. Gubbins, R. Radhakrishnan, M. Sliwinska-Bartkowiak, *J. Phys.: Condens. Matter* **2006**, 18, R15−R68.

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IL4 Annealing Induced Evolution of Germanium Quantum Dots

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We explored self-assembled Ge quantum dots (QDs) formation in dielectric matrix. The starting structures were thin films consisting of (Ge+SiO_2)/SiO_2 multilayers grown by RF magnetron sputtering at room temperature. QD nucleation started already during deposition and related inhomogeneities of about 2nm in size were detected by GISAXS. Subsequent annealing of such films up to 600°C in inert atmosphere resulted in Ge quantum dots formation at the position of the Ge rich SiO_2 layers, with a high lateral density (about 10^{12} cm^{-2}) of Ge QDs having a good crystallinity. The SiO_2 spacer layers separated well the adjacent Ge rich layers, where the Ge QDs were formed with a diameter about the size of the (Ge+SiO_2) as-deposited layer thickness. The QDs are arranged with a good vertical repeatability confirmed by the appearance of a Bragg sheet in 2D Grazing Incidence Small Angle X-ray Scattering (GISAXS) patterns. The structural analysis, by wide angle X-ray diffraction, GISAXS and transmission electron microscopy, has shown that the described processing of the films induced large compressive stress on the formed QDs. Additional annealing at higher temperatures resulted in a QD deterioration where germanium leaked out and left vacancies of somewhat larger size.

T6 MOFs at the Biointerface

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Metal-organic Frameworks (MOFs) are well known for their exceptionally high surface area and have been extensively studied for their gas adsorption properties. We have recently uncovered a method for using MOFs to encapsulate biomacromolecules that mimics natural biomineralisation. Like the biomineralisation process the crystalline MOF shell protects the biomolecule from the external environment (1). A key aspect of this protective capacity is that the MOF tightly encapsulates the biomolecules within the framework. This crucial information was quantified by modelling SAXS data. New developments in this burgeoning area of MOF chemistry will be canvassed.

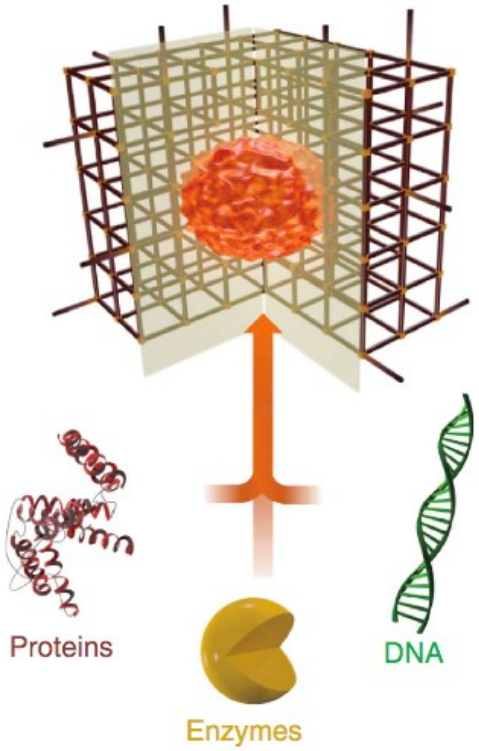

Figure: Schematic of a MOF encapsulating a biomacromolecule

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T7 Metal-Organic Frameworks from ceramics

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Metal-Organic Frameworks (MOFs) are an emerging class of porous crystalline materials holding much promise for application in gas storage, separation, catalysis, sensing, biotechnology, microelectronics.[1] In the early stage, the preparation of MOFs was typically performed applying solvothermal methods to a solution containing a ligand and a soluble salt for metal node of the framework. During several hours of thermal treatment, the self-assembly process leads to the formation of porous crystals. More recently, different alternative synthetic approaches have been proposed highlighting how different processing methods allow for the synthesis of MOFs with different interesting futures. Some of these new approaches involve the exploitation of ceramics or insoluble inorganic precursors either as seeding agents for the rapid MOF growth, or as feedstock materials for the structuralization of MOF into complex architectures.[2,3,4] In other cases, these new methodologies allows for the rapid transformation from minerals into the porous crystals, while recent examples highlight how MOFs can be obtained by gas phase processing of thin oxide layers.[5,6] In this presentation we will describe the recent advance in the field with emphasis on the use of SAXS as a tool for the investigation of the MOF formation.

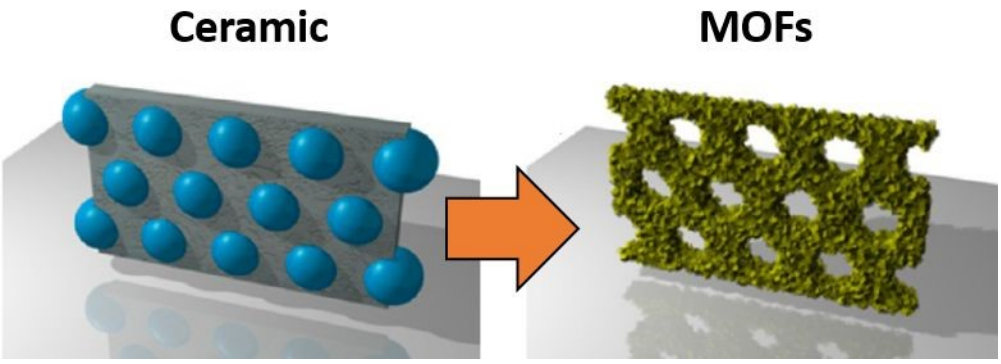

Figure 1: schematic representation of the structuralization of MOFs from a ceramic material.[2]

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T8 From Aryltin Trihydrides to Nanosized Polymers- Solvent and Residue Effects on Material Morphology

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Organotin dihydrides (R_2SnH_2) have been extensively studied as precursors in the formation of polymeric materials (polystannanes) exhibiting a linear backbone of covalently bonded tin atoms and can be seen as a molecular metal wire (Sn-Sn) embedded in an organic jacket, featuring an increased degree of electron delocalization by catenation leading to promising materials in charge-transfer devices [1]. Similarly to organotin dihydrides, organotin trihydrides ($RSnH_3$) undergo Sn—Sn bond formation upon loss of hydrogen *via* a reductive dehydrogenative coupling reaction in the presence of the cheap and easy to handle amine base TMEDA (N,N,N',N'-tetramethylethylenediamine) as polymerization catalyst forming hitherto unknown aryl decorated tin nanoparticles (aryl@Sn) (Figure 1a) [2][3][4]. The nature of the aryl residue as well as the donor capacity of the reaction solvent can affect the morphology and common correlation length of the nano-Sn/C composite material. Therefore, a range of aryl substituted tin trihydrides ($arylSnH_3$) including *o*-tolylSnH_3, 1-naphthyl, *p*-^{n}butylphenylSnH_3 was successfully converted to novel aryl decorated tin nanoparticles (aryl@Sn) (Figure 1a) [3][4]. SAXS measurements on the isolated material revealed that the nature of the aryl substituent alters the size of the formed nanoparticles.

Furthermore, *in situ* synchrotron SAXS measurements of the reductive dehydrogenative coupling reaction of *o*-tolylSnH_3 in a variety of solvents including diethyl ether, toluene, DME and cyclohexane were employed. These investigations reveal the effect of the nature of the applied solvent on the nano morphology evolution over reaction time (Figure 1b). These results successfully provide a "chemical file" allowing for a fine tuning of the material's characteristic on a nanometer scale.

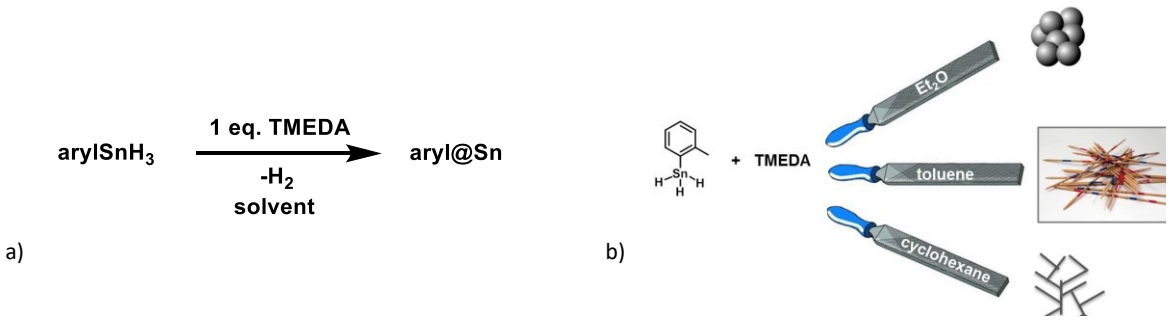

Figure 1. a) Synthesis of aryl decorated nano particles **aryl@Sn** *via* dehydrogenative coupling reaction of aryltin trihydrides. b) Morphology fine tuning of ***o*-tolyl@Sn**.

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T9 Nanoscale fractal aggregates of caffeine in hot-coffee conditions

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The dynamical and structural properties of caffeine solutions at the solubility limit have been investigated up to 80 °C by means of MD simulations, Raman and static- and dynamic light scattering and small angle neutron and X-rays scattering experiments [1,2].
At short range, the Raman investigation clearly shows that dipolar interactions play a fundamental role in determining stacking of caffeine molecules in solution. In order to reach this achievement, polarized ultraviolet Raman resonant scattering experiments have been carried out on caffeine aqueous solutions as a function of concentration and temperature. The investigation pointed out at the aggregation and solvation properties, particularly at elevated temperatures. The NCE concentration dependence shows that caffeine aggregation at 80 °C occurs by planar stacking of the hydrophobic faces. The data clearly indicate that dipolar interactions determine the reorientational motion of the molecules in solution and are the driving force for the stacking of caffeine. In parallel, the observed dephasing times imply a change in caffeine interactions as a function of temperature and concentration. A decrease, at low water content, of the dephasing time for the ring breathing vibration mode indicates that self-association alters the solvation structure that is detectable at low concentration.
At large range, a clear picture unambiguously supported by both experiment and simulation emerges: caffeine self-aggregation promotes the formation of two distinct types of clusters; linear aggregates of stacked molecules, formed by 2 to 14 caffeine molecules depending on the thermodynamic conditions; and disordered branched aggregates with a size in the range of 1000-3000 Å. While the first type of association is well known to occur under room temperature conditions for both caffeine and other purine systems, the presence of the supramolecular aggregates has not been reported previously. MD simulations indicate that branched structures are formed by caffeine molecules in a T-shaped arrangement. An increase of the solubility limit (higher temperature but also higher concentration) broadens the distribution of cluster sizes, promoting the formation of stacked aggregates composed by a larger number of caffeine molecules. Surprisingly, the effect on the branched aggregates is rather limited. Their internal structure and size do not change considerably in the range of solubility limits investigated. This study provides a basis for further investigation of the temperature evolution of caffeine supramolecular structures, as well as for the complexes that can be formed by the addition of other molecules such as simple sugars.

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T10 Structure Analysis of Drug Delivery Systems with SAXS in the Laboratory

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Small-Angle X-ray Scattering (SAXS) draws increasing attention in the field of pharmaceutical engineering. 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.

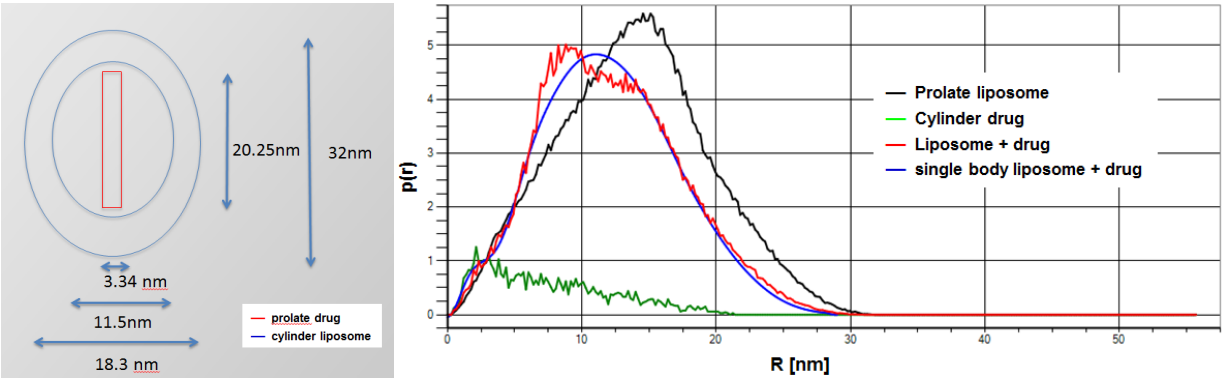

Figure 1: Study of a liposome drug carrier system. Data, obtained from a SAXS-measurement, yielded with aid of single body simulation (simulated Pair Distance Distribution Function on the right side) in the depicted model of the drug-loaded liposome (on the left side).

IL5 In-operando SAXS for Energy Applications

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Transport and re-distribution of ions in storage- and conversion devices for electrical energy is complex. Beside atomistic simulations, comprehensive in-situ experimental approaches are in high demand to gain a more fundamental understanding of the basic mechanisms determining the charge storage ability of, for example, supercapacitors and related technologies.[1] In recent years, in-operando techniques have come up particularly in battery research, aiming at "visualizing" such phenomena during operation of real working devices. Recently, we have introduced in-operando small-angle X-ray scattering at the Austrian SAXS beamline at ELETTRA to track ions in aqueous electrolytes within the carbon nanopores of supercapacitor electrodes during charging and discharging.[2]

Due to the complex pore geometry and the multi-phase character of the system (carbon, water, and at least two types of ions), the analysis of the in-operando SAXS data is challenging. Contrast variation by using different ions can help to establish simple models for global- and local ion redistribution as a function of applied voltage.[2] In order to obtain more direct information on the arrangement of ions within the nanopores, we introduce a novel strategy for SAXS data analysis. Real-space models of the disordered carbon nanopore structure are derived from an analytical fit to the SAXS intensity of the empty carbon electrode using the concept of Gaussian random fields (GRF). The computer generated real-space pore structure is then filled with electrolyte and the equilibrium positions of the ions within the pores are determined as a function of the applied voltage using Monte Carlo simulations. Subsequent Fourier Transformation of the simulation box (weighted by the corresponding electron densities) yields simulated SAXS curves which can be compared with experimental in-operando SAXS measurements. As a major outcome we observe that charge is most effectively stored in sites of the carbon structure with highest possible geometrical confinement, whereby also the ion hydration shell is partly lost.

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T11 Structural Characterization of Challenging Biomolecular Complexes by Integration of SAXS with Complementary Techniques

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Structural analysis of multi-domain protein complexes is a key challenge in current biology and a prerequisite for understanding the molecular basis of essential cellular processes. The use of solution techniques is important for characterizing the quaternary arrangements and dynamics of domains and subunits of these complexes. As experimental data for large protein complexes are sparse, it is advantageous to combine these data with additional information from other solution techniques.

In my presentation I will show our recent achievements in integrating Small-Angle X-ray Scattering (SAXS) data with complementary data from Nuclear Magnetic Resonance Spectroscopy, X-ray crystallography, electron microscopy, and mass spectrometry to study structure and dynamics of large disease-related proteins and protein complexes [1-9]. By using our integrated approach we were able to provide a comprehensive and accurate description of protein complex structure and dynamics in a native-like environment. This underscores the central role of SAXS for structure determination of protein complexes and ensures its unique role and contributions in integrated structural biology approaches in the future.

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T12 From protein-protein interactions to amyloid aggregation: SAXS plays as an outsider

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Small Angle X-ray Scattering experiments on dilute protein solutions are exploited to achieve several and different aims. SAXS can determine protein structure at low resolution [1] and refine the crystallographic structure of in solution proteins [2]. In the case of model proteins, SAXS provides information concerning their interactions and even hydration features [3]. In a few examples, we present how SAXS can unveil important protein features, which are not easily determined from other experimental techniques.

A combination of SAXS and elastic and quasi-elastic neutron scattering experiments on model proteins correlated dynamical changes induced by non-denaturing high pressure treatment with protein-protein interactions, suggesting that density and compressibility of water molecules in contact with the protein are key parameters to regulate the protein flexibility [4].

SAXS experiments on proteins leading to amyloid fibrils have been performed in more diluted conditions in order to evidence and to describe the structure of the aggregates. In these experiments, some intriguing aspects of the aggregation processes have been determined. The rate of an aggregation process, in the range of milliseconds, was observed, as well as the exact amount of each species in each time step was evaluated [5]. In the case of septins, GTP-binding proteins involved in many human pathologies and able to form hetero-oligomeric complexes, we have observed how temperature increase can induce the formation of aggregates, evidencing their amyloid nature. SAXS data analysis by a global fit has provided enthalpy, entropy and heat capacity variations governing dimer-monomer dissociation equilibrium, and suggested that the lower molecular weight species triggers the amyloid formation [6]. Recently, SAXS experiments on amyloid beta peptide in presence of heat shock proteins performed at Elettra provided noticeable information and perspectives on future BIO-SAXS applications.

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IL6 SYNCHROTRON SEC–SAXS data AS EXPERIMENTAL CONSTRAINTS TO model the detergent corona around a membrane protein

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The application of small-angle X-ray scattering (SAXS) to structural investigations of transmembrane proteins in detergent solution is hampered by two main inherent hurdles. On the one hand, the formation of a detergent corona around the hydrophobic region of the protein strongly modifies the scattering curve of the protein. On the other hand, free micelles of detergent without a precisely known concentration coexist with the protein–detergent complex in solution, therefore adding an uncontrolled signal. To gain robust structural information on such systems from SAXS data, in previous work, advantage was taken of the online combination of size-exclusion chromatography (SEC) and SAXS, and the detergent corona around Aquaporin-0, a membrane protein of known structure, could be modelled. A precise geometrical model of the corona, shaped as an elliptical torus, was determined [1]. The geometrical approach was revisited by more thoroughly examining the correlations between all fitting parameters, and derive some rules about which strategy to adopt in further studies with different proteins [2]. The program Memprot has been developed to systematize the SAXS calculations from the geometrical models and is accessible to the community [3]. In a subsequent development of our software, for cases in which the protein contour is less isometric than that of AQP-0, we developed a parameterized geometrical model of the detergent corona which adheres more closely to the actual shape of the protein. A recent output of this new strategy on the receptor system HasA-HasR [4] will be shown.

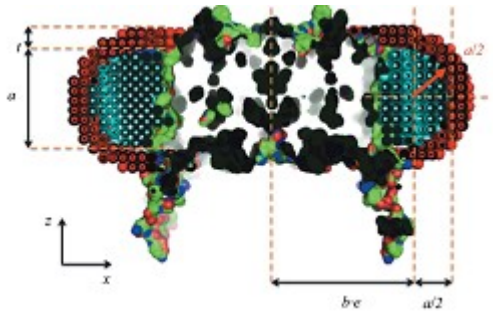

Figure 1: A section of the complex between the full-atom 2b6p structure and its detergent corona optimized from SEC–SAXS experimental data, as modeled in Memprot.

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PL3 Investigating rapid technological coating processes in real-time and in situ

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Modern technologies in organic electronics and photovoltaics (OE/OPV) rely on the understanding of the rapid coating processes involved. This necessitates the observation of the fundamental processes on the nanoscale, from the atom/molecule via the nanocluster to the full-scale domain [1,2,3,4]. Such an understanding enables precise control of the complex nanostructures used in OE and OPV [5]. The coating processes themselves range from vacuum deposition to solvent-based methods, being compatible with technologically relevant roll-2-roll-applications [6]. I will review recent advances in the fundamental understanding of nanostructure formation in the field of rapid coating processes. This includes, but is not limited to, sputter deposition, solution casting, as well as spray deposition. I will give an outlook to future possibilities especially with respect to the miniaturization of devices [7].

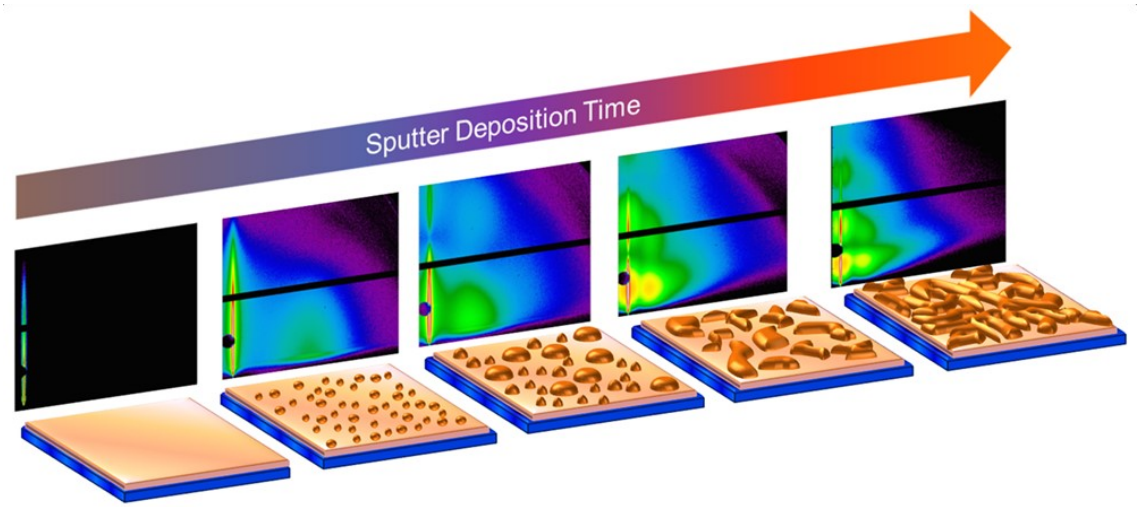

Fig. 1: Nucleation, condensation and growth of a gold layer on top of an organic substrate is observed in real-time during sputter deposition using in-situ µGISAXS [3].

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T13 µGISAXS on curved fluid-fluid interfaces: Following particle rearrangement and ejection upon surface compression

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Nanoparticles with a metal core stabilized by a ligand shell have aroused a lot of interest in recent years because of their unique electrical, magnetic and optical properties, which give rise to many potential applications in nanotechnology [1]. The behaviour of these particles at the interface between two immiscible liquids is also of great interest, as it can alter the stability of emulsions and foams [2], among others.

While micron-sized particles are irreversibly adsorbed at the interface, nanoparticles exhibit much more complex behabior. As the particle size decreases so does the energy of attachment, which eventually becomes comparable to the thermal energy. Thus the behaviour at the interface itself becomes highly dynamic.

Here we probe this behaviour on a highly curved surface upon compression. Using a novel sample cell, we suspended a droplet of oil in water and let a nanoparticle film self-assemble at the interface. Reducing the amount of oil in the droplet causes surface compression and nanoparticle rearrangement [3,4]. We studied this process, including particle ejection from the film, by GISAXS with a microfocused beam on the droplet surface. The sample cell, experimental setup and initial results will be discussed.

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IL7 Application of GISAXS on study of three-dimensional quantum dot lattices

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Grazing incidence small angle x-ray scattering-GISAXS is excellent technique for study of size and arrangement properties of quantum-dot systems. We use it in study of various types of three dimensional quantum dot lattices formed by self-assembled growth and ion-beam assisted nucleation. We successfully use SAXS beamline at Elettra, for more than ten years for GISAXS measurements. Due to the lack of suitable theory for GISAXS interpretation of some specific systems, we have also developed theoretical models for their description and software *GisaxStudio* for data processing. The models enable full structure determination, including lattice type, lattice parameters, the type and degree of disorder in the quantum dot positions and the distributions of the quantum dot sizes.

T14 Performance of the High Pressure SWAXS System Designed for the Austro-SAXS Beamline

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The development of our high pressure system started at the IBN (then IBR) in Graz, Austria in the 1993 and it has been improved since then [1,2]. Primarily it had been supposed that the system would be available for the users of the Austro-SAXS beamline at Elettra which was being designed in parallel at the time. The planned rough main parameters of the system were specified to match the needs of the research on membrane lipids performed at the IBN, extended to studies expected to be performed using the Austro-SAXS beamline in future: The system should cover SAXS and near WAXS angle regions up to circa 30° (2θ), the cell should be a flow-through type, usable up to 300 MPa and 200° C with water as the main pressurizing medium.

To make the cell small, simple and with as low thermal capacity as possible, both, originally Beryllium windows are fixed only by gluing and self-sealed applying Bridgman mushroom piece principle. The output window is fixed directly to the cell body, while the input window to a nose of a high pressure nipple which can be dismounted. Noses of various lengths enable optimizing the sample thickness.

The first tests at a synchrotron source were done at the HASYlab (DESY) in 1995 giving us the first hints in which directions to improve. Eventually, the windows material was changed to diamond with higher price and absorption but obvious advantages including mainly lower background scattering, the possibility to visually inspect the sample, stimulate it by a laser beam or even extend the range of measurements to optical wavelengths. The system was motorized and computerized thus enabling pressurizing and depressurizing jumps and various pressure and temperature regimes e.g. equi-density run. Quite an effort has been made to enable usage of various pressurizing media and to separate them from the samples with reasonable efficiency.

Up to now our system has been used mainly at Elettra and ESRF but the copy of our cell has been used for ten years at the NSLS by a BU group [7] and recently at SLAC. At Elettra, for instance, there have been circa 30 high pressure measuring campaigns during 20 years. Based on the experience from the use of our system, recently a 'pressure-cell-light' version for the use with conventional X-ray sources and a new version of system for the Austrosaxs beamline have been developed successfully.

The task of the recent presentation is to describe and discuss some of the special and specific properties of our high pressure instrument and to highlight some of the most interesting measurements which were accomplished using it, e.g. [3,4,5,6].

Credits: Peter Laggner, Michael Rappolt, Karin Pressl and Sigrid Bernstorff.

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T15 Biomembrane Complexity at the Sub-Nanometer Level

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Ever since the seventies of the past century, scattering techniques have contributed significantly to our understanding of the physical chemistry pertaining to lipid-only membranes. Such knowledge is either an asset for biotechnological applications (drug delivery, sensors, etc.), or provides important insight into the physiological role of biological membranes. Research in our laboratory is focussed in particular on the latter aspect aiming to unravel the diverse coupling mechanisms between membrane lipids and proteins. Our model systems display various degrees of complexity, including domains of various sizes or transbilayer lipid asymmetry. Applying a diverse set of small-angle x-ray and neutron scattering experiments we have successfully unravelled the sub-nanoscopic properties of such systems including transbilayer coupling in asymmetric membranes, domain elasticity or protein partitioning into coexisting lipid domains. I will these highlight recent advances.

T16 Interaction of Charged Dendrimers with Model Lipid Membrane: a SAXS Study

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Dendrimers represent promising smart polymeric architectures for a wide range of biotechnology and bio-medical applications. Due to their peculiar molecular arrangement, characterized by a highly ramified structure, dendrimers are suitable systems for the development of molecular level synthetic prototypes for the control of organization and dynamical properties on the colloid size scales, as well as a versatile platform to investigate self-assembly processes in complex nanomaterials [1]. In spite of the wide range of applications of dendrimer nanocarriers, a major problem is related with their disruptive effect toward bio-membranes, which bring out some cytotoxicity issues connected with their employment in bio-medical applications [1,2].

The self-assembly processes of mixtures of charged polyamidoamine (PAMAM) dendrimers and dipalmitoylphosphatidylcholine (DPPC) lipids were investigated by means of Small Angle X-ray Scattering (SAXS), complemented with zeta potential analysis and Raman spectroscopy experiments. The obtained results evidence the sensitive interactions between charged dendrimers and lipid molecules at the surface of the liposome (with an enhancement of the liposome zeta potential), as well as in the hydrophobic region of the bilayers (with a perturbation of the lipids alkyl chains of the liposome). Interestingly DPPC liposomes showed different behaviours during their interaction with (negatively charged) sodium carboxylate terminated [-COO- Na+] or (positively charged) amino terminated [-NH2] dendrimers. Analysis of the SAXS structure factor by means of suitable model for the inter-dendrimers electrostatic potential allow an estimation of an effective charge of PAMAM dendrimers, while only a fraction of this charge (about 1/7) contribute to the liposome zeta-potential increase with increasing amount of PAMAM dendrimers. The findings of our investigation help to rationalize the effect of the nanoparticles electrostatic interactions in solution during drug delivery processes [2, 3].

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T17 *In-situ* small angle X-ray scattering reveals formation of highly organised nanostructures during digestion of milk fat

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The digestion of dietary triglyceride oils into the amphiphilic products fatty acid and monoglyceride plays a vital role in the delivery of the lipid-soluble bioactive molecules such as hydrophobic vitamins, carotenoids and drugs to the circulatory system of the body. These components have been found to self-assemble into a variety of thermodynamical equilibrium structures under biologically relevant conditions of the human intestine. Here, the discovery of highly ordered geometric nanostructures during the digestion of model lipids and milk will be discussed: Transitions from normal emulsion through a variety of differently ordered thermodynamically stable nanostructures were observed using time-resolved small angle X-ray- and neutron scattering combined with contrast variation, and visualized by cryogenic transmission electron microscopy. The response of these thermodynamical equilibrium structures to changes in the pH and composition, as well as the location of components within the self-assembled structures, will be presented. The results will help to understand the process of lipid digestion with a focus on colloidal structure formation and transformation for the delivery of hydrophobic functional molecules and may indicate a compensating mechanism for the maintenance of lipid absorption under compromised lipolysis conditions.

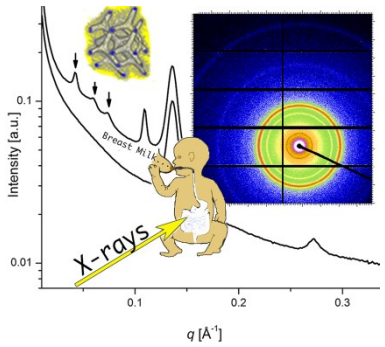

Figure : Experimental set-up used for our SAXS study on the digestion of human breast milk.[1]

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T18 Easy and fast fabrication of a free-jet micromixer for SAXS

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Time resolved Small Angle X-ray Scattering (SAXS) is a powerful tool to study the kinetic of nanoparticles formation, on a structural scale from 1 nm to 1 µm. Stop-flow and continuous mixing devices have been used to follow the nucleation and growth of nanoparticles with an ever increasing time resolution and reduced dead-time (i.e. the first observable point after reaction started).[1-4] Recently, it was demonstrated how a microfabricated micromixer producing a free-jet in air allows for a great improvement in the achievable time resolution (down in the range of 10s of µs per time point), together with a reduction of the dead-time to less than 50 µs, depending on the experimental set-up (e.g. X-ray spot size and speed of the free-jet).[5] That device featured challenging geometrical constrains, i.e. high resolution and high aspect ratio, and required a not-common and costly fabrication strategy based on DXRL for its demonstration. Here we show how an alternative approach based on a moulding technique could make the fabrication of the same device much more convenient, requiring only a high quality master mould in Si, fabricated through standard photo-lithography and dry-etching (fig.1). Hundreds of copies of the final device can then be produced by moulding an UV-curable resin. Sealing is achieved by gluing a flat slab of the same resin. A working device is shown in fig.2.

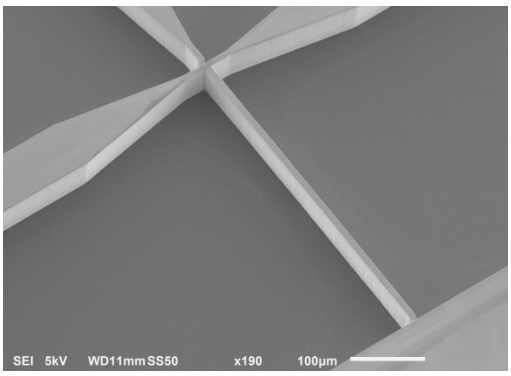

Fig.1 – SEM picture of the produced Si mould

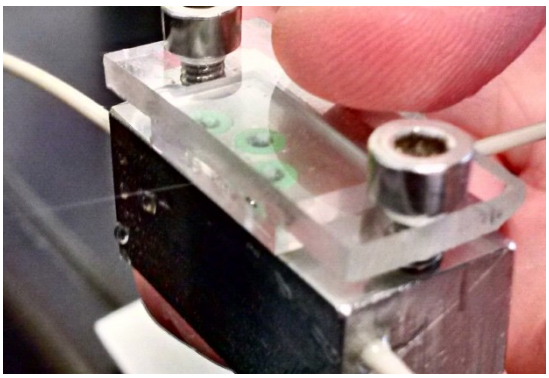

Fig.2 – Picture of a working device producing the free-jet

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Poster presentations

	Presenter		Title
p1	Carducci	Federica	Tunability of Anisotropic Properties of Guan(os)ine enriched GMP Wires
p2	Digiacomo	Luca	Structure of liposomes in biological media: a synchrotron SAXS study
p3	Ghazal	Aghiad	Direct Monitoring of Calcium-Triggered Phase Transitions in Cubosomes Using SAXS Combined with Microfluidics
p4	Kornmuller	Karin	Supramolecular self-assembled peptide double helices revealed by Synchrotron SAXS
p5	Wolf	Marcell	Effective interactions in protein-salt solutions approaching liquid-liquid phase separation
p6	Hodzic	Aden	Monitoring of Pentoxifylline Thermal Behavior by Novel Simultaneous Laboratory Small and Wide X-Ray Scattering (SWAXS) and Differential Scanning Calorimetry (DSC)
p7	Juraić	Krunoslav	Simultaneous grazing incidence small and wide angle X-ray scattering on titania nanotube arrays
p8	Koczwara	Christian	Ion electrosorption in hierarchically ordered carbon studied by in-situ small angle X-ray scattering
p9	Naumenko	Denys	Laser-induced aggregation of gold nanoparticles: Multi-technique analysis for SERS applications
p10	Rigodanza	Francesco	Perylene Bisimides: new synthesis and applications to bring an old material into a new era
p11	Syrgiannis	Zois	Ruthenium based photosensitizer/catalyst supramolecular architectures in light driven water oxidation
p12	Tawfilas	Massimo	Mimetization of TiO2 Nanocrystals into polymer matrix through grafting surface modification
p13	Zhigunov	Alexander	Aggregation behaviour of boron clusters inside polymeric nanoparticles in aqueous solution
p14	Haider	Richard	Development of a 3D Mixing Device for Small Angle X-Ray Scattering Measurements
p15	Hill	Christian	Optofluidic Force Induction: Platform-technology for particle characterization and manipulation
p16	Marmiroli	Benedetta	Synchrotron SAXS study of the interaction of silica nanoparticles with lysozyme using a free jet micromixer
p17	Burian	Max	Towards a pump-probe x-ray scattering setup at the Austrian SAXS beamline
p18	Chemelli	Angela	Small angle X-ray and dynamic light scattering investigations of nanosheets
p19	Kriechbaum	Manfred	20 years High-Pressure Cell for the SAXS-Beamline at ELETTRA
p20	Sartori	Barbara	Tuning the structure of silica mesoporous materials by precursors composition: solvents effect studied in situ with SAXS
p21	Zidansek	Aleksander	Small-angle X-ray scattering studies of confined smectic liquid crystals
p22	Bernstorff	Sigrid	Formation and properties of Cu nanoparticles
p23	Rath	Thomas	Time resolved GISAXS and GIWAXS investigations of precursor based formation routes towards metal sulfide nanocrystals

p24	Karlušić	Marko	Observation of ion tracks on GaN and TiO2 surfaces by AFM and GISAXS
p25	Naughton	Kyle .L.	Self-Assembly of the Cephalopod Structural Protein Reflectin

p1 Tunability of Anisotropic Properties of Guan(os)ine enriched GMP Wires

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Metal-ion induced guanosine-based G-quadruplexes have been first reported in the early 1960s[1] and their structural properties and ability to assemble in supramolecular structures were fully analysed[2,3]. Aqueous solutions of guanosine compounds can form gels through G-quadruplex self-assembly: stable hydrogels at neutral pH over a wide temperature range were in particular found in binary mixtures of 5'-guanosine monophosphate (GMP), potassium salt, and guanosine (G). These hydrogels could be used in a wide range of biotechnological and clinical applications as cellular immobilization, drug delivery, biomolecules separation if the gelation properties are controlled and convenient[4].

In the case of GMP, SAXS experiments in diluted solutions demonstrated that quadruplexes are relatively short (the quadruplex length is typically around a few hundred Å), but the length is strongly dependent on the type and the counter-ion concentration and temperature (in particular, alkali metal ions induce helix elongation, while temperature induces helix fragmentation and shortening). By SAXS experiments, we recently demonstrated that G-enriched GMP-quadruplex gels show anisotropic property: butterfly patterns, strongly dependent on composition and temperature, indicate that anisotropic quadruplex orientation in the gels can be easily modulated.

These results open new opportunities in gaining new perspectives on applications in guanosine derived self-assembling systems.

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p2 Structure of liposomes in biological media: a synchrotron SAXS study

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The ability to shape matter at a nanometric scale leads to the development of systems able to encapsulate and deliver drugs for therapeutic purposes, including treatments of cancers and genetic diseases. The efficiency of these nanoparticles (NPs) relies on the interactions with the living matter, especially those arising from the first contacts to biological media, i.e. blood and plasma. Indeed, under in vivo conditions, NPs get covered by a protein layer, which is commonly referred to as protein corona (PC). Recent advances in bio-nanotechnology have improved the characterization of the NP-PC complexes, mainly in terms of size, surface charge and protein composition [1]. However, a detailed information about the internal structure of these systems is still missing and may be crucial to categorize dynamical processes at cellular and subcellular level, e.g. their cytoplasmatic trafficking. In this regard, current researches have pointed out that the efficiency of liposome/DNA complexes and lipid-based NPs is affected by their intracellular dynamics and the underlying bio-interactions. Liposomes are nanometer-scale vehicles, which are designed to contain drugs, nucleic acids and therapeutic agents. Their self closed lipid-based structure protects their cargo from the degradation and the similarity of their external surface to biological membranes provides unique opportunities for the delivery of molecules into cells. A deep knowledge of their inner structure has been achieved through synchrotron Small Angle X-ray Scattering experiments. Here, we aim to extend this perspective by including the presence of the PC, in order to evaluate the alterations induced by biological media. To this end, we processed synchrotron SAXS experimental data through global fitting analyses [2][3]. Our findings support the hypothesis that biological media may modify the internal structure of liposomes, thus affecting the entire nanoparticle as well as its surface. These results may have profound impacts for the design of nanoparticles for drug and gene delivery.

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p3 Direct Monitoring of Calcium-Triggered Phase Transitions in Cubosomes Using SAXS Combined with Microfluidics

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We present a simple microfluidic device that can be combined with synchrotron SAXS for monitoring the dynamic structural transitions of negatively charged lipidic cubosome nanoparticles on exposure to buffer containing calcium ions[1,2]. The microfluidic device is a thiol-ene based system equipped with 125 µm thick polystyrene windows, which are suitable for X-ray experiments[3]. The device was prepared by soft lithography using elastomeric molds followed by a simple UV-initiated curing step to polymerize the chip material and simultaneously seal the device with the polystyrene windows. The microfluidic device was successfully used to explore the dynamics of the structural transitions of phytantriol/dioleoylphosphatidylglycerol (PHYT/DOPG)-based cubosomes on exposure to buffer containing calcium ions. The resulting SAXS data were resolved in the time frame between 0.27 and 1.35 s and a calcium-triggered structural transition from an internal inverted type cubic phase of the symmetry *Pn3m* to an internal inverted type cubic phase of the symmetry *Im3m* was detected. The combination of microfluidics with X-ray techniques opens the doors to the investigation of early dynamic structural transitions, which is not possible with conventional techniques such as glass flow cells. The combination of microfluidics with X-ray techniques can be used for investigating protein unfolding, monitoring the formation of nanoparticles in real-time, and for other biomedical and pharmaceutical investigations.

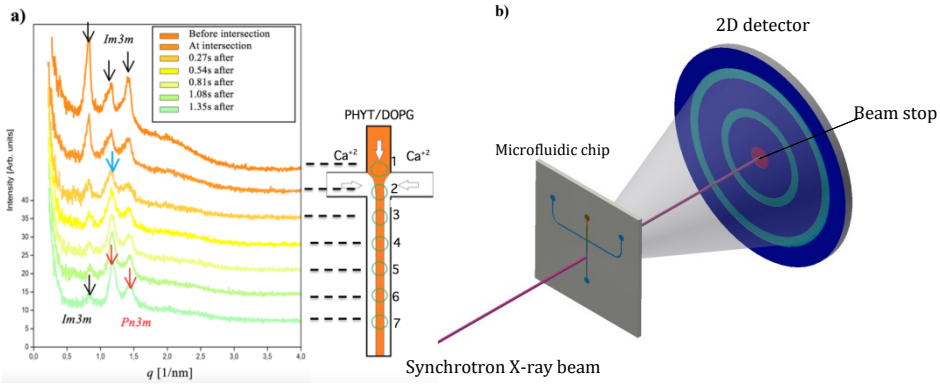

Figure 1: Time resolved SAXS experiments displaying the dynamic structural transitions in the interiors of PHYT/DOPG nanoparticles, a) hydrodynamic focusing chip with green circles indicating the positions at which SAXS was measured b) the combination of microfluidic with SAXS.

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p4 Supramolecular self-assembled peptide double helices revealed by Synchrotron SAXS

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A very active area of research aims to use peptide-based nanostructured materials as biomimetic artificial matrices in nanomedicine [1]. Self-assembling amphiphilic designer peptides are a promising class of molecules for these purposes. To fully exploit their potential, we aim to acquire a deep understanding of the peptides' individual, as well as their collective morphologies, the underlying dynamic assembly mechanisms and how these peptide materials act at the interface of synthetic and biological membranes. By combining Synchrotron small angle X-ray scattering (SAXS), transmission electron microscopy (TEM), and infrared (IR) spectroscopy we studied the concentration-dependent self-assembly of GAAVILRR, an 8-residue amphiphilic designer peptide. Above a critical aggregation concentration it forms a variety of structural intermediates and finally develops to unique supramolecular double helices. They are characterized by lengths of several hundreds of nanometers and uniform diameters of about 24 nm. SAXS and IR spectroscopy allowed a detailed look at the internal organization of the structures: we propose a 3-layered model, where monomers are interdigitated and tightly packed, held together by multiple weak noncovalent bonds. The double helices are intertwined to a network, with gel-like properties. These double helices remained structurally unaltered for several months [2]. To probe the interaction of peptide-assemblies with artificial membrane mimics, we performed differential scanning calorimetry (DSC), TEM and SAXS. Even at high concentrations, the membrane integrity remained unaffected by the peptides' presence. Based on our results, this peptide has a high potential to meet the needs of a next-generation biomaterial in future medical and technological applications.

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p5 Effective interactions in protein-salt solutions approaching liquid-liquid phase separation

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Effective interactions of proteins in aqueous solution play a crucial role in understanding protein crystallization and many physiological diseases related to protein association [1]. Experimental results on the phase behavior of protein solutions show a variety of phenomena [2-4] including metastable liquid-liquid phase separation (LLPS) and reentrant condensation in the presence of multivalent counter ions [5]. The metastable LLPS is related to a short-ranged isotropic attraction or attractive patches.
We present an experimental study combined with a theoretical discussion of the effective interactions in protein solutions approaching a liquid–liquid phase separation (LLPS) induced by addition of multivalent metal ions. We have determined the salt and protein partitioning in the two coexisting phases, which provides the isothermal binodal of the LLPS in the (cp, cs) plane (Fig. 1a). The reduced second virial coefficient, B_2/B_2^{HS} (Fig. 1b), is used to describe the interaction and discussed with theoretical predictions for colloidal systems.

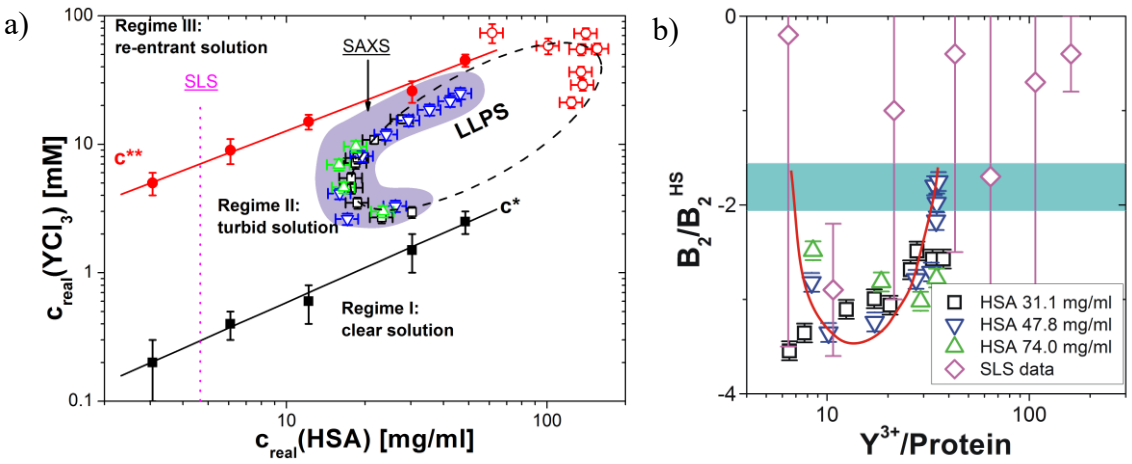

Figure 1: Phase diagram of the studied system in the (cs, cp) plane (a) with the corresponding reduced second virial coefficient B_2/B_2^{HS} b)

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p6 Monitoring of Pentoxifylline Thermal Behavior by Novel Simultaneous Laboratory Small and Wide X-Ray Scattering (SWAXS) and Differential Scanning Calorimetry (DSC)

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The thermal and structural evolutions associated to active pharmaceutical ingredient (API) purity are monitored using a laboratory instrument (S3-MicroCaliX) allowing simultaneous time-resolved X-ray scattering at both wide and small angles (SWAXS) as a function of temperature. This is performed simultaneously with differential scanning calorimetric (DSC) that is carried out in the same apparatus at scanning rate of 2 K/min on the same sample in the range from 20° to 200 °C. We have studied simultaneous thermal and structural properties of pentoxifylline, as an active pharmaceutical ingredient (API), for its purity quality control. We have found a satisfying API purity, due to obtained melting temperature and enthalpy values, which are in a well agreement with literature. We have also found that the combination of these techniques allows the thermal monitoring of scanning rates of 2 K/min, continuously without the need for static thermal equilibration, particularly for X-ray spectra. Hence, DSC and SWAXS allowing better identification of the structural thermal events recorded by following of the phase transitions simultaneously. This interpretation is much better possible when X-ray scattering at small and wide angles is coupled with DSC from the same sample. Hence, as a laboratory tool, the method presents a reproducible thermal and crystallographic API purity quality control of non-complex samples, as crucial information for pharmaceutical technology [1].

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p7 Simultaneous grazing incidence small and wide angle X-ray scattering on titania nanotube arrays

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Titanium dioxide (TiO_2) is one of the most intensively investigated compounds in material science due to its essential properties. It is a wide band gap semiconductor having band-edge positions appropriate for solar cell applications and for hydrogen generation by water splitting. It is also known as a non-toxic, environment-friendly, corrosion-resistant material. Nano-forms of TiO_2 such as nanoparticles, nanorods, nanowires and, in particular, nanotubes can be used as photoelectrode in dye-sensitized solar cells. The conversion efficiency of solar cells can be significantly improved by using oriented nanorod-like materials on top of transparent conductive oxide.

In this work, we examined vertically aligned TiO_2 nanotube array thin films as possible photoanode material. TiO_2 nanotube arrays usually are prepared by anodization of titanium metal foil. Instead of thick Ti foil, in this work we used Ti thin films prepared by DC magnetron sputtering as a substrate for the anodization process. SnO and ZnO-coated glass were used as a substrate for magnetron sputtering. For anodization, we used a conventional two electrode system with Pt foil as counter electrode and electrolyte containing ethylene glycol, ammonium fluoride and distilled water. The anodization time and applied voltage were adjusted to maximise the nanotubes length and to produce nanotubes with a diameter of 30-80nm. After anodization, the TiO_2 nanotube arrays were calcined in an oven at 500ºC [1].

We performed a simultaneous grazing incidence small and wide angle X-ray scattering (GISWAXS) experiment for the structural analysis of the obtained TiO_2 nanotube array samples before and after the calcination process. We will present how structural properties of the TiO_2 nanotube array thin film samples are influenced by the anodization parameters.

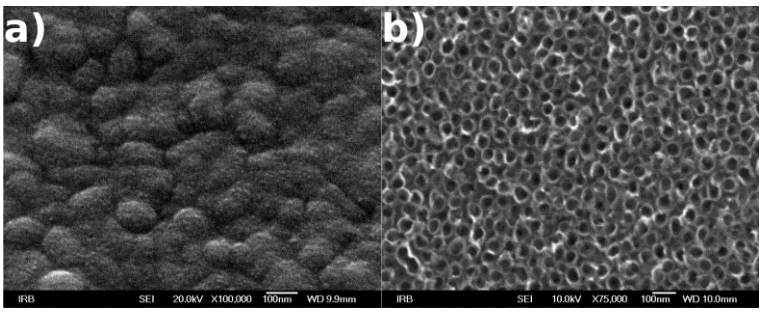

Figure 1: SEM image of Ti substrate (a) and TiO_2 nanotubes array (b) obtained by anodization.

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p8 Ion electrosorption in hierarchically ordered carbon studied by in-situ small angle X-ray scattering

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Supercapacitors are devices used for the efficient storage of energy revealing high power densities and extremely long cycle lifetimes. If two electrodes are immersed in a liquid electrolyte and a voltage is applied, an electrical double-layer will form at the electrode-electrolyte interface causing the capacitive behaviour. A number of in-situ techniques have been established within the last years aiming to gain a basic understanding of ion transport and the charge storage mechanism within the nanometer-sized confinement of porous carbon electrodes. [1] Recently we have shown that in-situ SAXS is a suitable technique to study both, global ion fluxes and local ion rearrangements within activated carbon electrodes during charging. [2] To gain in-depth information on the ion electrosorption process or associated phenomena like pore swelling we are currently following two approaches. Besides the development of an enhanced data-analysis strategy to treat disordered nanoporous carbons, we investigate model carbons with narrow size distribution tailored for the demands of our in-situ scattering experiment.
We have performed in-situ small-angle x-ray scattering (SAXS) at the Austrian SAXS beamline at ELETTRA to track ions in aqueous electrolytes within hierarchically structured carbon (HSC) electrodes (macro-, meso- and microporous) during charging and discharging. The investigated HSCs pore structure is a negative replication of a corresponding silica template. To add additional micropores an activation process with carbon dioxide is necessary. The SAXS curve shows a sharp peak at q = 0.7 nm^{-1} which confirms the existence of an ordered pore structure enabling the investigation of lattice strains (pore swelling) of the carbon electrode during adsorption and desorption of ions. Peak shifts following the applied cell voltage suggest that the carbon pore structure swells and shrinks during charging and discharging. However the separation between structural changes of the carbon pore structure and voltage induced ion rearrangements remains difficult. The use of different electrolytes containing ions with different electron numbers represents a contrast variation approach which turned out as a suitable way to systematically analyse such in-situ SAXS experiments. [2]

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p9 Laser-induced aggregation of gold nanoparticles: Multi-technique analysis for SERS applications

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Surface-enhanced Raman scattering (SERS) has already demonstrated its significant potential in analytical science [1]. Thus, current efforts are focused on the development of affordable and reproducible SERS substrates, which exhibit high enhancement factors and uniform responses. A large number of strategies was adopted to produce effective SERS substrates; however, most of substrates are tuned for a use of single excitation wavelength and consequently can only be applied for a limited number of analytes. Hence, SERS substrates that demonstrate broadband plasmonic properties represent a more flexible analytical tool for multi-wavelength or tunable light sources, especially for biological applications. In the current study, we demonstrate that direct laser writing (DLW), which activates a photoreactive moiety and immobilizes functionalized gold nanoparticles on chemically modified glass substrates [2], can be used to produce SERS substrates of various sizes and geometries [3]. Using multi-technique approach that includes Scanning Electron Microscopy, Atomic Force Microscopy, UV-Vis and Raman spectroscopy, and Small Angle X-ray Scattering (SAXS), we show that by tuning the DLW parameters a broad plasmonic response is possible to obtain that is due to the formation of rod-like gold nanoparticles aggregates at higher DLW powers as revealed by both SAXS and optical extinction measurements. Finally, two Raman reporters, a small synthetic benzotriazole azo organic dye and a larger biological molecule, hemin, are tested at three fixed excitation wavelengths in the visible range (473 nm, 532 nm and 660 nm). SERS enhancement factors show a weak dependence on the wavelength used and the molecules investigated; moreover, the possibility of creating arbitrary shaped and uniform structures is demonstrated. The reported results show that DLW is an excellent technique to engineer microstructured and broadband SERS substrates.

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p10 Perylene Bisimides: new synthesis and applications to bring an old material into a new era

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Perylenebisimides (PBIs) are robust and photostable dyes, with outstanding optical and electronic properties. As a consequence, these compounds have been widely studied as pigments, fluorescence sensors, n-semiconductors in organic electronics, photovoltaics and bases for copolymers or oligomers, with important role in single junction devices.

We focused on designing a photocatalytic system mimicking the PSII oxygen evolving center. Our approach was based on the unprecedented combination of metal-free perylene bisimides as photosensitizers with totally inorganic polyoxometalates (POMs) as catalysts. In particular, N,N'-bis(2trimethylammonium)-ethylene)perylene-3,4,9,10-tetracarboxylic acid bisimide, PBI^{2+}, photosensitizer, one of the stronger photo-generated oxidant in its excited state, which nicely complements $[Ru_4(\mu\text{-}O)_4(\mu\text{-}OH)_2(H_2O)_4(\gamma\text{-}SiW_{10}O_{36})_2]^{10-}$ (Ru_4POM), the forerunner of its class as water oxidation catalyst. This is a game-changing strategy with respect to all of the established methods based on the usual Ru(II)polypyridine photosensitizers and/or conceived through the covalent design of photocatalytic electron donor-acceptor conjugates.

The novel hybrid material, fully characterized by SAXS, AFM, TEM and UV-Vis analyses, performed oxygen evolution with well-behaved kinetics, long operation time and good conversion yield. The fascinating potential of organic PBIs for solar energy storage was herein demonstrated in water, under visible light irradiation and within an exceptionally broad pH window featuring the unprecedented combination with a totally inorganic and very robust molecular catalyst.

p11 Ruthenium based photosensitizer/catalyst supramolecular architectures in light driven water oxidation

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Light driven water oxidation is a key step in artificial photosynthesis, aimed at splitting water into hydrogen and oxygen with sunlight.[1] In such process, the interactions between a photosensitizer (PS) and a water oxidation catalyst (WOC) play a crucial role in the rates of photoinduced electron transfers, determining the overall quantum efficiency of the system. In this work, by means of Small Angle X-ray Scattering (SAXS) we investigate the nature of the aggregates between ruthenium polypyridine photo- sensitizers (Rubpy and Ru 4 dend) and a tetraruthenium polyoxometalate (Ru_4POM) water oxidation catalyst.[2] Aggregate scattering is confirmed by the strong intensity-increase in the low-q regime, whereas the power law-fit of this region show slopes between -3 and -4, suggesting globular and porous aggregates. Intermolecular PS/WOC distances lower than 3 nm support the observed fast photoinduced electron transfers (<120 ps), however the proximity of the two components in the hybrids is also responsible for fast charge recombination. Approaches for inhibiting such undesired process are discussed.[3]

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p12 Mimetization of TiO_2 Nanocrystals into polymer matrix through grafting surface modification

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Surface ligand engineering is an effective tool for producing polymer nanocomposites, a tantalizing kind of materials that express the properties of inorganic matter within an organic matrix. The introduction of a thin polymeric layer enables a good dispersion of the inorganic nanoparticles (NPs) in solvents and matrixes in which bare particles instead aggregate and precipitate. The surface modification is needed in order to tune the nanofiller morphology during synthesis or introduce additional functionalities to the NPs, providing new properties for a wide number of applications [1]. By grafting polymer chains on the surface, we are able to create a thin layer of mixing that makes the particles adhere and blend with the matrix at the microscopic level. The main parameters that control the quality of the dispersion and the performance of the final material are the molecular weight of the grafted chains and of the polymer matrix (N, P) together with the grafting density (σ), i.e. the number of chain per unit area of the nanoparticle [2]. Small-angle X-ray scattering analysis (SAXS) is particularly suited to determine the conformation of the grafted chains which in turn determines the mixing with the matrix. In our contribution we explored the grafting of polystyrene with different molecular weight onto spherical anatase NPs. The polymers are synthesized with RAFT (Reversible Addition-Fragmentation chain Transfer) synthesis with two RAFT agents (chain transfer agent CTA) ended with a carboxylic acid and a phosphoric acid [3]. The NPs synthetized by a solvothermal method [4] and they are characterized through electron microscopy and WAXS. The polymers are investigated with Nuclear Magnetic Resonance (NMR) and vibrational spectroscopy. Termogravimetric analysis (TGA) is used to quantify the amount of polymer grafted on the NPs. SAXS analyses are performed in static and dynamic conditions with different solvents in order to study the behaviour of the polymer chains and define the conformation of the grafted chains on the NPs surface.

Fig.1: Representation of the grafting to approach that bring to a brush regime (left) and a random coil (right) polymer

conformation on the NPs surface.

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p13 Aggregation behaviour of boron clusters inside polymeric nanoparticles in aqueous solution

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Compounds based on boron clusters could be used for the treatment of a broad variety of biological targets including the inhibition of HIV protease, and others. Possible arrangements and aggregation behaviour of these clusters are important for understanding of its potential role in medical applications. In this study, we have followed the behaviour of anionic boron hydrides and its association with block copolymers. We have observed precipitation when metallacarborane [3-cobalt bis(1,2-dicarbollide)]$^-$ anion, CoD^-, were added into salted solution of poly(ethylene oxide) (PEO). On the other hand there were only weak interactions of CoD^- anions with several others hydrophobic polymers. Thus it was possible to create block copolymer and control the complexation behavior of the composite by varying blocks lengths, architecture of copolymer and ratio. It was shown that interaction of poly(ethylene oxide)-block-poly(methacrylic acid) with CoD^- in salted solution (H_2O with 0.154 M NaCl) leads to a spontaneous formation of core-shell nanoparticles [1]. Complex of NaCoD with star-like architecture copolymer based on poly(ethylene oxide) and poly(2-methyloxazoline) in aqueous media forms micelles, the size of which does not change in a wide range of CoD^-/copolymer ratio.

While analysing aggregation behaviour of boron clusters in aqueous solution, interparticle distances were measured for a range of concentrations by means of small angle x-ray scattering (SAXS). For this step of our study, we have chosen boron clusters B12 ($[B12H12]^{2-}$) with Na cations and clusters CB11 ($[CB11H12]^{1-}$) with K cations. Based on results obtained from concentration dependence we propose a model which comprises two levels of aggregation. Observed correlation peak at q = 1.2 $Å^{-1}$ is in agreement with the model proposed by Terrence J. Udovic et al. based on crystallographic results [2]. In SAXS region we observe another level of association, which is seen as a structure peak at q = 0.36 $Å^{-1}$ for the concentrations 0.6M and higher. This peak is not observed for lower concentrations and for higher concentrations it shifts towards lower angles. Similar behaviour have been observed by W. Hausler et al [3] for Apoferritin in aqueous solution. The aggregation is a result of concurrent electrostatic repulsion and minimizing of energy due to B12 hydrophobicity.

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p14 Development of a 3D Mixing Device for Small Angle X-Ray Scattering Measurements

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Using Micromixers it is possible to measure, time resolved and in-operando, fast chemical reactions, like nanoparticle formation [1], protein folding [2] or interactions of proteins with nanoparticles [3]. While only using only very small sample volumes, hydrodynamic focusing allows microfluidic devices to achieve very short mixing times and high time resolution.

A micro mixing device employing two dimensional sheath flow focusing has already been designed, fabricated with Deep X-Ray Lithography and tested by our group [4,5]. The free jet was used to reduce the background signal of the mixer walls and avoid the eventual clogging of the channels due to formation and/or aggregation of nanoparticles after the mixing. However the liquid jet itself also gives a SAXS background [6], which limits the quality of measurements made with this device, especially for reactions with weak scattering signal.

To overcome these problems we are currently developing a device, which shall use a sheath flow in three dimensions, keeping the reactant from the mixer walls by enveloping it on all sides, thus avoiding nanoparticle aggregation and clogging issues. Furthermore this allows measurements to be conducted already at the beginning of the mixing, enabling observations from partial to complete mixing.

The current studies focus on finding the optimal mixer geometry to achieve this goal. Also the production process using laser lithography is currently under investigation and being tested at the SAXS beamline at the Elettra-Sincrotrone radiation facility in Trieste.

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p15 Optofluidic Force Induction: Platform-technology for particle characterization and manipulation

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Nanoparticles, especially in the biotechnological and pharmaceutical fields, possess significant potential for future applications. However, undefined and heterogenic particle populations demand for analytic tools and advanced manipulation equipment for a focused and controlled application. Since the discovery was made that photons can be used to manipulate particles in the nano- to microscopic size regime,[1] research efforts have focused on different application and innovations using this approach. Our studies focus on advancements in optical force chromatography (OFC)[2] to achieve separation of heterogeneous mixtures in a liquid medium by the application of optical forces, counteracting well-defined fluidic drag forces (Fig. 1).[3][4] Combining the potentials of modern diffractive optics[5] with highly defined microfluidic flow-setups, new and fine-tuned membrane-like modes for characterizing, separating, sorting, concentrating and filtering of nano-structured particles and materials are achievable. Through these optical techniques a tuneable working space can be established. This approach increases sensitivity and throughput and simultaneously reduces particle-aggregation and optical binding. The system can be operated in sizing mode (Fig. 2, left), filter mode or trapping mode (Fig. 2, right) and operates label-free, contact-free and non-invasively as continuous flow or batch setup.

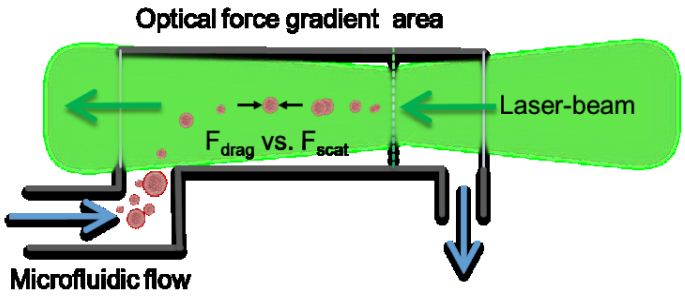

Fig. 1 Schematic presentation of the basic OFC principle

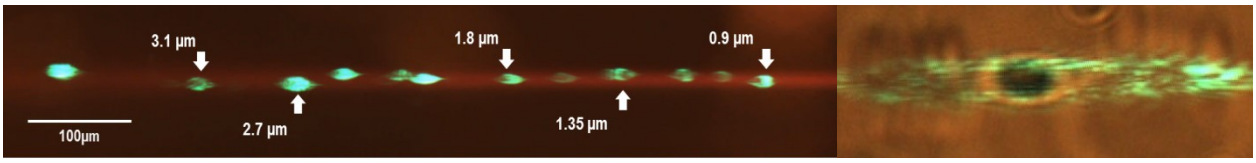

Figure 2 Left: Sizing and filtering of multilamellar phospholipid (DPPC) vesicles; system-parameter result in corresponding filter pore size of 0.9 µm. Right: Cell deformation in trap-mode. Deformation force = 5.2 pN

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p16 Synchrotron SAXS study of the interaction of silica nanoparticles with lysozyme using a free jet micromixer

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The aggregation of nanoparticles caused by their interaction with proteins can provoke bioadverse consequences [1]. For this reason the understanding of the interaction mechanism is essential both in the biomedical and biotechnological field, for safe use of nanoparticles. Up to now, few studies have been reported about protein mediated particle aggregation. The adsorption of lysozime at silica nanoparticles within a wide pH range has already been investigated, showing both heteroaggregation and flocculation [2]. Preliminary kinetic studies conducted at the Austrian SAXS beamline @ Elettra-Sincrotrone Trieste, have evidenced that the reaction occurs in a time scale smaller than 10 ms, therefore it cannot be examined with conventional instruments like the stop-flow. In order to reach a smaller time scale, we have employed microfluidics. In the last years, we have combined rapid mixing by means of microfluidics, a free liquid microjet in air and synchrotron SAXS technique, to measure fast reactions below the millisecond time range. The micromixer is based on hydrodynamic focusing and has been fabricated using Deep X-Ray lithography technique at the DXRL beamline at Elettra-Sincrotrone Trieste [3]. The presence of a free jet overcomes the background given by the existence of channels walls, making the device suitable for many techniques. The micro free-jet produces a signal in the SAXS measurement that has to be taken into account. We have therefore investigated the jet behaviour by SAXS showing that it can be used as a signal background during subsequent investigation of the fast reaction [5]. In this communication we present the first results of the early stage studies of the interaction of silica nanoparticles with lysozyme by using the free jet micromixer in combination with SAXS. We will describe the fabrication process, the measurement issues, and we will demonstrate the suitability of the technique for this kind of studies.

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p17 Towards a pump-probe x-ray scattering setup at the Austrian SAXS beamline

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Through the advent of free electron lasers as well as ultrafast lab-based laser systems, highly time resolved methods have risen to be essential tools to study the interaction between light and condensed matter.[1] However, both of the named techniques lack the ability to directly track structural changes on the atomic scale, immediately after irradiation. The pulsed nature of synchrotron light, on the other hand, opens up a window at exactly these time- and length-scales: filming sub-nanometer structural changes of liquid- and solid-state systems with picosecond time resolution.[2]

We are implementing such an optical-pump hard-x-ray-probe setup at the Austrian SAXS beamline at the Elettra. A custom radio-frequency circuit that is in phase with the storage-ring-cavity synchronizes all necessary devices and delivers the gating pulses required for x-ray bunch discrimination at the detector. Further, a femtosecond laser will be installed to deliver high-power pulses in the VIS-IR range to initiate light-induced phenomena in liquid and solid samples. We will present the detailed setup with its specifications and provide an overview of the current challenges we are facing.

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p18 Small angle X-ray and dynamic light scattering investigations of nanosheets

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Exfoliation or delamination of layered materials has gained a lot of interest due to the improved properties of nanosheets compared to the pristine bulk material. Their sizes and especially their thicknesses are of great importance and dictate their properties. In contrast to microscopic methods, light and X-ray scattering can be directly applied to dispersions of nanosheets. The need of pretreatment is circumvented and thus accompanying changes in the samples can be prevented. Small angle X-ray scattering is applied to measure thicknesses and information about their lateral sizes is gained by means of dynamic light scattering. Additionally to the redundant pretreatment, results obtained from these measurements are an average of all particles in the scattering volume and thus provide and overall picture of the sample. The proper overview of sizes and thicknesses of all particles included is important for subsequent application and consequently, scattering methods complement microscopic measurements for the characterization of nanosheets.

p19 20 years High-Pressure Cell for the SAXS-Beamline at ELETTRA.

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Based on the experience of the 20 years ago developed high-pressure SAXS system at the SAXS-beamline at ELETTRA, we have recently improved and redesigned the existing system [1-2]. The new system consists of a novel and compact pressure cell and an automated pressure control system for numerous applications to study nanostructure as a function of pressure with SAXS. The cell itself is machined out of stainless steel with cube dimensions of 3 x 2 x 2 cm and has two disc-shaped diamond windows with a diameter of 4 mm and a thickness of 0.75 mm each, serving as the X-ray entrance and exit windows, respectively. The cell is connected to a motor-driven spindle press using water as the pressure transmitting medium and the system can be operated in automated pressure or temperature scans. Additionally p-jumps triggered by pneumatic pressure valves, separating two reservoirs of different pressures can be applied, following the nanostructural changes by time-resolved SAXS. The sample in the cell can be pressurized up to moderate 3000 bar and above and its angular range encompasses 0° - 20° (2θ). Changing temperature in the cell can be achieved either by a circulating flow of water through copper plates or by Peltier elements between which the cell is sandwiched.

The application range is widely spread from studying phase diagrams of lyotropic or thermotropic liquid crystalline systems, proteins, lipoproteins or polymers and their barotropic phase transitions. The pressure cell can also be utilized in experiments with super-critical CO_2 and in the grazing incidence mode (GISAXS) for oriented and aligned lipid systems on solid supports.

Selected examples of high-pressure SAXS experiments and their results will be shown. In this example, studies with reversible pressure-scans (at constant T) and T-scans (at elevated p) have been performed on various LDL-samples (low-density lipoprotein) differing in the amount of triglycerides.

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p20 Tuning the structure of silica mesoporous materials by precursors composition: solvents effect studied in situ with SAXS

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Mesostructured silica nanoparticles can be synthesized by various methods: among them, the spray-drying of aerosol droplets is an attractive one-step route to obtain well ordered nanoparticles, as it is environmentally benign, is cheap if compared to alternative methods, and allows a good control of the synthesis conditions.

The control of the morphology, particle size, and uniformity is getting more and more important to produce nanoparticles tailored on the forseen applications, such as catalysis, adsorption, optical devices, bio-imaging, drug delivery, and nanomedicine.

During the self-assembly of mesoporous silica, the templating agent drives the condensation of tetraethyl ortosilicate (TEOS) molecules into an organized structure. We designed and built a system to study in-situ with SAXS the self assembly process of mesoporous material condensation in the gas phase. [1]

Changing the precursor stoichiometry is one mean to control the final structure of the nanoparticles. In the present communication we will show an in-situ study in the gas phase of the formation of silica nanoparticles in water/ethanol system: we will show the influence of two different solvents and their respective molar ratio on the mesostructure of silica nanoparticles produced via spray drying.

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p21 Small-angle X-ray scattering studies of confined smectic liquid crystals

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Small-angle X-ray scattering at the Austrian SAXS Beamline at Elettra is an excellent method for the determination of smectic order parameter in liquid-crystalline systems. The alkylcyanobiphenyl (n-CB) homologues series of liquid crystals comprise an excellent model system for studying the smectic ordering in confined geometry, because their bulk properties are well understood. We started these investigations with a temperature dependence measurement of the smectic ordering parameter for octyl-cyanobiphenyl liquid crystal confined to controlled-pore glass matrices [1]. The latter exhibits a high surface area, efficient filling of liquid crystals into cylindrical pores of well-defined diameter, and chemical inertness. The confinement of octylcyanobiphenyl liquid crystal to control porous glasses can be qualitatively reproduced using the Landau-Ginsburg approach, and it also significantly influences the smectic layer spacing [2]. Dimensional crossover and scaling behavior have also been observed in these systems [3]. Possible applications of small-angle X-ray scattering in other liquid-crystalline systems possessing smectic-type of ordering [4] will be also discussed.

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p22 Formation and properties of Cu nanoparticles

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Nanometer sized metallic particles, such as Cu, Ag and Au, posses specific optical properties due to the presence of a plasmon band. This band originates from the collective oscillations of the conduction electrons, the plasmons, and it corresponds to a narrow absorption band in the visible spectral range. The plasmons lead to a modification of the properties of the adjacent dielectric material in which the nanoparticles are included. Controlling the chemical environment of the nanoparticles and tuning their size and shape, one can modify the macroscopic properties of the host matrix in a controllable manner. In our work, the focus was on Cu nanoparticles, synthesized in or on silica, thus forming a composite material.

We produced the samples by high vacuum thermal evaporation of a single Cu layer on top of Si substrate and/or one capped with a thin SiO_2 layer. The substrate temperature was varied during the deposition, and the samples were additionally annealed ex-situ in high vacuum. The nanoparticles morphology and development was studied by simultaneous grazing incidence small and wide angle X-ray scattering (GISWAXS). The results are compared to those obtained by Atomic Force Microscopy and Scanning Electron Microscopy measurements on the samples without capping layer. It is shown that the Cu production is critically dependent on the starting configuration of the layers. Finally, the plasmonic effect was monitored by UV-Vis reflectance spectroscopy, while the oxidation of nanoparticles was further studied by photoluminescence spectroscopy.

p23 Time resolved GISAXS and GIWAXS investigations of precursor based formation routes towards metal sulfide nanocrystals

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Metals sulfide nanocrystals have attracted increasing attention in the last decades because of their optical and electronic properties, which make them interesting materials for numerous applications including photovoltaics, light emitting diodes, bioimaging and photocatalysis. In recent years, we directed our research towards solution-based approaches for the synthesis of nanocrystals in polymeric matrices as well as of nanocrystalline metal sulfide films for application in solar cells. For these preparation routes, we used precursors such as metal xanthates or other metal salts in combination with a sulfur source like thiourea and thioacetamide, which can be converted into metal sulfides by mild thermal treatment (approx. 200°C). The formation process as well as the growth kinetics can significantly influence the properties and quality of the final nanocrystals. Therefore, we explored time resolved grazing incidence small and wide angle X-ray scattering (GISAXS and GIWAXS) techniques to gain information about the formation of the nanocrystals and nanocrystalline thin films. By simultaneous measuring of small and wide angle scattering data, information about structural changes in the films and, at the same time, information about the crystal structure as well as the evolution of the crystallite size could be gathered. In this contribution, we discuss the benefits and instructive outcomes of these measurements on the basis of several examples from our recent research. In the case of the synthesis of copper indium sulfide as well as cadmium sulfide nanocrystals in a matrix of a conjugated polymer, the time resolved GISAXS/GIWAXS measurements revealed knowledge about the reasons for the formation of different nanomorphologies depending on the used precursors [1,2]. In another example, insights on the formation of copper indium sulfide and copper zinc tin sulfide (CZTS) nanocrystal films will be presented [3,4].

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p24 Observation of ion tracks on GaN and TiO_2 surfaces by AFM and GISAXS

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Swift heavy ions have found widespread use in research and technology, for both materials analysis and modification. Having a kinetic energy in the MeV range and above, their usability now spans diverse fields such as hadron therapy, industrial production of track etched membranes and testing of electronic devices against single-event upsets. The impact of the swift heavy ion leads to intense heating of the material due to electron–phonon coupling and often triggers melting in a nanoscale volume along the ion trajectory. Upon rapid resolidification, permanent damage called an ion track is formed. At the surface, various nanostructures can be found on the position of the ion impact by means of atomic force microscopy. In recent years particular attention was given to grazing incidence swift heavy ion irradiation that yields ion tracks on the surface in the form of long chains of nanodots [1-3]. In the present contribution we show that such ion tracks on the surfaces can be also investigated by grazing incidence small angle X-ray scattering. Examples of the observation of surface ion tracks on GaN and TiO_2 surfaces are presented [4,5].

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p25 Self-Assembly of the Cephalopod Structural Protein Reflectin

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Cephalopods are well known for their remarkable camouflage abilities; they can modify their coloration, texture, pattern, and reflectance to blend into the surrounding environment. Such dazzling camouflage abilities are partially enabled by specialized intracellular nanostructures that are composed of unique structural proteins known as reflectins. Recently, we have discovered that films from reflectins possess interesting optical and electrical properties in vitro, such as protonic conductivities that are on par with state-of-the-art artificial materials. However, a definitive structural rationale for some of the films' properties and for their in vivo functionality has not been previously established. As such, we have extensively characterized reflectins both in solution and at solid substrates with scanning electron microscopy (SEM), small-angle x-ray scattering (SAXS), grazing incidence small-angle x-ray scattering (GISAXS), and grazing incidence wide-angle x-ray scattering (GIWAXS). Based on these experiments, we have developed a model of the self-assembly of reflectins into films. Our findings may hold implications for not only better understanding the mechanisms that cephalopods employ to dynamically control their coloration but also for the development of bioinspired proton conducting materials and modular infrared camouflage materials.

Contributors		
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Grenci	G.	IL8, p16
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Hofmann	Eddie	il3
Holy	V.	IL10
Huber	A.	p15
Huerre	Axel	t13
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La Ganga	Giuseppina	p11
Laggner	P.	IL1
Lahajnar	Gojmir	p21
Lebius	Henning	p24
Lechner	Rainer T.	IL5
Lehofer	Bernhard	p19
Leitinger	G.	p4
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