**Slow science and new geometries. Experiments, simulations and calculations of three-arm 'star polyphile' liquid crystals**

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Self-assembly of amphiphiles can be interpreted as a problem of surface geometry, since the 2-phase boundary is an interface. We are interested in extending those ideas and results to star polyphiles, with three (or more) mutually immiscible arms connected to a common centre. Here, the structures must contain three-phase lines as well as (two-phase) surfaces. Fundamental geometric studies have revealed a vast panorama of possible candidate self-assembled forms.  
Experimental studies - mainly SANS and SAXS - prove that three-arm polyphiles form a number of liquid crystalline mesophases, which appear to be related -- though different -- to simpler hexagonal and bicontinuous cubic mesophases found in amphiphilic lyotropes. We are now close to understanding some of the more complex liquid crystals formed in these polyphiles mesophases. And complex they are. One ideal structure consists of interwoven 3D polar labyrinths, 2D (mesh-like) fluorocarbon domains forming a 3D lattice (see image below) and 1D hydrocarbon domains (helical rods). The research is challenging, but ultimately scientifically rich. Progress in this new area would have been impossible without a combined research effort on many fronts, from chemical synthesis, to non-euclidean geometry.
Mesostructured polymer surfactant films

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Solutions of polyelectrolytes with surfactants show a variety of adsorption behaviours at interfaces. We have exploited this phenomenon to create solid films of polyelectrolyte and surfactant at the air-solution surface, from solutions where the polyelectrolyte is uncharged. In these systems, a self-supporting film containing highly ordered 2D or 3D surfactant mesostructures in a hydrogel matrix several hundred nanometres thick forms spontaneously at the solution surface. Our initial investigations focused on films made from polyethylenimine and cationic surfactants and we continue to study film formation mechanisms in this system. We have also grown films using a number of other surfactants and polymers, allowing detailed control of film mesostructure and thickness. More recently we have also prepared films with similar structures by spray coating of the same aqueous solutions. To improve the film biocompatibility, we have grown films using zwitterionic surfactants which are less toxic than cationic surfactants. The encapsulation and release of hydrophobic species has been studied as a function of crosslinking and mesostructure in these hydrogel films. We are continuing to develop these membranes for potential applications in controlled release and as a responsive support for sensor species. Here, I will describe our studies on these films, in particular our work to understand film formation, control the nanostructure in the films, and improve encapsulation and biocompatibility of our materials.
The orientation of water at the hydrophobic/hydrophilic interface and its pH dependence

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Water is the defining liquid of life. The physico-chemical properties of water at hydrophobic or hydrophilic sites determine how interfaces are made and proteins are folded. A molecular level understanding of this behavior is rich in complexity and therefore not fully understood. One particular issue concerns the orientation of water at the hydrophobic/water interface and its relation to the presence of charge. We have investigated charge and water orientation by performing sum frequency scattering and electrokinetic mobility measurements on nanoscopic oil droplets in water. We find that the interfacial water orientation in an ion free droplet solution at pH 7 is indistinguishable from a droplet solution with negative surfactants on the interface. Furthermore, the oil/water response does not depend on pH.
A new setup for force measurement of the pinning of liquid droplets

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For understanding the dynamic wetting behavior of a solid by a liquid, the study of moving contact lines is of fundamental interest, since the contact line is the barrier separating wetted area from not wetted area. On non-ideal surfaces, the contact line pins to the solid surface. In the static case this pinning results in contact angle hysteresis. In the dynamic case it results in an unsteady movement of the contact line [1]. Experimental setups for the investigation of moving contact lines involve either the dipping of solid surfaces into a reservoir of liquid (e.g. Wilhelmy plate) or putting single droplets of liquid on solid surfaces. A standard method to investigate the behavior of moving droplets is the tilted plate method. Using this method, the investigation of the contact angle hysteresis as well as the moving behavior of a droplet on a surface is possible [2]. However, the tilted plate method allows neither direct control over the velocity of the moving droplet nor a direct measurement of the force acting on the droplet.

We present a novel setup for direct measurement of the force needed to move a droplet on a solid surface while measuring the dynamic contact angles. We place a flat solid sample on a disc. On the sample we put a drop of liquid and stick a rectangular capillary into the droplet. The disc is then rotated, moving the surface underneath the droplet. The droplet pins to the capillary and is dragged over the surface. The capillary bends due to the force needed to move the droplet. The capillary deflection is measured by a laser deflection system. Using Hooke’s law, the force acting on the capillary is measured. In particular, the setup is able to resolve forces due to pinning of the contact-line of a water drop. For example we were able to investigate the dependence of the pinning force on the position where the contact line pins to the surface by moving a water droplet over a well-defined periodic test-structure.

References
Shapes and motion of a dissolving dichloromethane drop on a surfactant solution

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Surface tension or buoyancy driven instabilities are at the origin of shape modifications and autonomous motion of drops. We have, in a recent study[1], showed a remarkable succession of dynamical regimes during the dissolution of a dichloromethane (CH₂Cl₂, 25 µL) drop deposited on aqueous solutions of cetyltrimethylammonium bromide (CTAB). The transition between the different regimes is controlled by the surfactant concentration. At low concentration, the drop moves on a back and forth or circular trajectory or, gives rise to steady or periodical spreading events. At intermediate concentrations, the drop transforms into an elongated structure with two (or more) sharp tips and enters into rotation. At higher concentrations, polygonal shapes give rise to a succession of polygons as hexagons, pentagons and squares. Coupled to these shape-forming processes is the emission of smaller droplets that undergo further division or disappear. To our knowledge, this system is the first example of such a sequence of highly ordered patterns induced by coupled hydrodynamic instabilities. The resulting structures show very efficient motility, internal agitation and dispersion properties.

Anomalous spreading with Marangoni flow on agar gel surfaces

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Wetting dynamics of liquids on gel surfaces is important for understanding mass transfer phenomena at biological interfaces because spreading processes contribute to effective nutritional absorption through the small-intestinal wall or to sensitization of the tongue surface. A well-established law that describes the spreading of nonvolatile liquids on flat and clean surfaces is $\theta_D = t^{-0.3}$, which is called Tanner's law. Here, $\theta_D$ and $t$ are the contact angle at three-phase contact lines and the dynamic spreading process time, respectively. On the other hand, to acquire a deep understanding of the spreading dynamics, it is crucial to not only observe the spreading but also to find experimental and theoretical strategies to control it. We have experimentally observed anomalous spreading of aqueous alcohol solutions on flat and rough fractal agar gel surfaces. On flat agar gel surfaces, extremely fast spreading [$\theta_D = t^{-0.92}$] that differs from Tanner's law was observed when the liquid contained over 9 wt % of 1-propanol in which strong Marangoni flow was observed as a fluctuation on the liquid surface. However, on fractal gel surfaces, different spreading dynamics [$\theta_D = t^{-0.58}$] were observed, although Marangoni flow still occurred. We found the surface dependent spreading can be discussed in terms of competition between Marangoni flow and the pinning effect due to surface roughness.

Designer microcapsules made using microfluidics

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Filled microcapsules made from double emulsion templates in microfluidic devices are attractive delivery systems for a variety of applications. In the microfluidic approach, water/oil/water double emulsions are formed in two subsequent dripping events, after which the monomeric oil phase is consolidated by photopolymerization, forming a capsule with an aqueous core. This allows facile tailoring of the microcapsules through a large number of variables, which in turn makes these systems more challenging to predict. To elucidate these dependencies, we start from previous theoretical predictions for the size of double emulsions and present quantitative design maps that correlate parameters such as fluid flow rates and device geometry with the size and shell thickness of monodisperse polymer-based capsules produced in microcapillary devices. Using polymers with selected glass transition temperatures as the shell material, we show through single capsule compression testing that hollow capsules can be prepared with tunable mechanical properties ranging from elastomeric to brittle. Weibull statistical analysis of the load at rupture of brittle capsules is also provided to evaluate the variability of the microfluidic route and assist the design of capsules in applications involving mechanically triggered release. The permeability of the capsules can also be tailored through the choice of polymer and addition of cross-linkers or plasticizers. Finally, we can manipulate the microstructure of the capsule shell by using silica nanoparticles with different surface treatments in the middle phase of the double emulsion templates. This can range from homogeneous composite shells to layered structures, where the particles occupy only the outer and inner surfaces of the shell, allowing for subsequent surface modification of the entire capsule.
Solvents can form micelles without surfactants – the “pre-Ouzo effect“

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In the 1970s mysterious so-called “surfactant-free” microemulsions were proposed, but neither convincing investigations of their structure were published nor was given a plausible explanation of the mechanism [1]. Finally, Katz and Vitale [2] showed a way of producing very fine emulsions with small droplets in a liquid phase without surfactants, dispersing agents of mechanical agitation that are remarkably stable in time. This behaviour is known today as the so-called “Ouzo effect”. It is well investigated now, and it could be demonstrated that the precondition for such “surfactant-free emulsions” is the mixture of two solvents with a solute (can be also a solvent), e.g. anethol, that is highly soluble in one solvent (ethanol), but not in the other one (water). In addition, the two solvents (e.g. water and ethanol) must be miscible, either completely or at least to a large extent. Whereas this two-phase region is widely studied now, we were interested in the monophasic region close to the binodal line, a region that we call here the “pre-Ouzo domain”.

We present a study of the monophasic region of such a surfactant-free ternary system containing water, ethanol and octanol based on dynamic light (DLS), small-angle X-ray (SAXS) and neutron (SANS) scattering. To our surprise, from these experimental results it became evident that well-defined micellar structures and consequently two distinct pseudo- phases exist in these ternary mixtures. To get further information about the partition of ethanol between the pseudo-phases (one is water-rich, the other one rich in octanol) we further considered the partial pressure of ethanol above the liquid medium. From the experimental results we were able to deduce a model for the driving force of this particular micelle formation that does not require the presence of surfactants. In the present contribution we will introduce this new type of association colloids and show how general it is.

References:
Many surfaces in nature are superhydrophobic – for example lotus leaves. Microscopic pockets of air are trapped beneath the water drops. This composite interface leads to an increase in the macroscopic contact angle and a reduced contact angle hysteresis, enabling water drops to roll off easily. However, the addition of an organic liquid such as alcohol or oil, decreases the interfacial tension sufficiently to induce homogeneous wetting of the surface. Drops, initially resting on air pockets (Cassie state), pass the transition to complete wetting (Wenzel state).

Coatings that are simultaneously superhydrophobic and superoleophobic, i.e. superamphiphobic, are rare. For superamphiphobicity, surface roughness, low surface energy and topography are essential. We designed an easily fabricated, transparent, and oil-repelling superamphiphobic coating [1]. A porous layer of candle soot is coated with a 25 nm thick silica shell. The black coating becomes transparent after calcination at 600°C. After silanization the coating is superamphiphobic and remains so even after its top layer is damaged by sand impingement. Water, soap and oil droplets roll off when tilting the surface by less than 5° taking dirt with them. The coating’s superamphiphobicity can be understood from Nosonovsky’s prediction that convex small-scale roughness can provide a sufficient energy barrier against wetting, thus rendering superamphiphobicity possible.

The good repellency of the coating towards different solvents enabled us to investigate drop impact and retraction as a function of impact height. We investigated both, the influence of interfacial tension and viscosity on the impact dynamics and determined a phase diagram separating the different scenarios, complete rebound, pinning and splashing.

Generalizing the link between the interfacial properties and bulk phase behavior of polyelectrolyte / surfactant mixtures

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Polyelectrolyte/surfactant (P/S) mixtures adopt a variety of structures at static air/liquid interfaces, ranging from monolayers, through trilayers to multilayers. These systems have been previously classified into two sorts: type 1 involving strong complex adsorption and thick layers and type 2 forming thin layers while exhibiting a surface tension peak. Recently, we have shown that a surface tension peak for a type 2 system (Pdadmac/SDS) occurs only after unlimited precipitation of surface-active material from the bulk solution reduces the surfactant surface excess. We have also shown that this system can exhibit the type 1 characteristic and interfacial multilayers when appropriately treated. In this work we extend our investigations to a typical type 1 system (NaPSS/DTAB) and show that through tuning the bulk phase behavior it can exhibit the type 2 characteristic of a surface tension peak. We have also investigated the effect salt (ionic strength) and salt quality (NaCl vs. NaBr) on the interfacial properties of NaPSS/DTAB system. Based on the newly available experimental data a new generalized model to describe P/S systems at fluid interfaces with respect to their bulk phase behavior will also be presented.
The effect of slip on fluid flow

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At small length scales, the no-slip hydrodynamic boundary conditions that states the velocity at a solid wall to be zero, is not always valid and slip may occur. Slip may depend on several factors such as wettability, surface roughness and presence of nanobubbles at the surface.

Here we investigate the relation between fluid flow and slip, using computer simulations and experimental flow studies. Most surfaces are not molecularly smooth and their surface heterogeneities may provide patchwise slip. We study the relation between different slip lengths and no slip and how it affects fluid flow in microchannels of varying size.

The computer simulations are made using the Comsol Multiphysics software whereas the experimental flow studies are performed using micron resolution particle image velocimetry, µPIV. In this technique, water is seeded with fluorescent polystyrene particles and the images obtained using two laser pulses within a short time period, are correlated to give the flow velocity. The microchannels used are made of polydimethyl siloxane, PDMS, some of which are further surface-modified to get hydrophilic and hydrophobic surfaces. The surfaces are characterised with different techniques, e.g. ESCA, AFM and contact angle measurements.
Diffusing wave spectroscopy on foams and microbubbles

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The structure of foams and suspensions of microbubbles changes in time due to coalescence, Ostwald ripening, and rearrangements of cells and lamellae. The study of these processes is made difficult by turbidity of the systems. Often, only the part of the systems, which is optically accessible, i.e. close to the glass container wall is observed. This problem is avoided by diffusing wave spectroscopy (DWS). In DWS, the fluctuations of multiply scattered light are analyzed. The spectrum of fluctuations reflects the spectrum of pathways of a photon from light source to detector. These pathways are random walks across the whole volume of the sample. The long pathways probe fast dynamics, whereas the short, more direct pathways fluctuate due to large, and therefore slower dynamics. The correlation functions of the fluctuations are interpreted in terms of the various dynamic processes, which take place inside the bulk of the foam or bubble suspension. In this presentation, the state of the art for DWS on foams will be discussed, and the exploration of the technique for suspensions of microbubbles, stabilized by proteins, will be presented.
Interactions between topographically structured hydrophobic surfaces

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Nanostructured particle coated surfaces with hydrophobized particles arranged in close to hexagonal order were prepared by Langmuir-Blodgett deposition followed by silanization. These surfaces have been used to study interactions between hydrophobic surfaces and a hydrophobic probe using the AFM colloidal probe technique. The different particle coated surfaces exhibit similar water contact angles, independent of particle size, which facilitates studies of how the roughness length scale affects capillary forces (previously often referred to as “hydrophobic interactions”) in aqueous solutions. In a second study we have measured interactions between hydrophobic microstructured surfaces with pore arrays and a hydrophobic colloidal probe. The pore array surfaces were designed to display different pore spacing and different pore depth. Contact angles measured with water and water/ethanol mixtures show that the pore depth does not have any influence on the surface wettability; a result that indicates that the position of the three-phase contact line and not the interactions underneath the droplet determines the contact angle. This is in contrast to the force measurements where the situation at the interface between the surface and the liquid is of crucial importance. The shape of the force curves tells us whether the capillary between the probe and the surfaces was formed on a flat part of the surface or in close proximity to a pore. The different parameters extracted from the force curves, like the interaction distances and the adhesion force, can be explained in relation to the depth of the pores.
Microparticles at fluid interfaces: structure and dynamics of single and binary systems

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Micro- and nanoparticles have attracted much interest as possible substitutes of surfactants and polymers as emulsion and foam stabilizers in food science, cosmetics, and pharmaceuticals. Also they are increasingly used as building blocks of functional materials and for controlled drug delivery. In most of the cases the systems of interest are multiphase, and subject to strong mechanical perturbations (flow, stirring, etc.). Also, quite frequently the particles are trapped at the interface of those multiphase systems, with a trapping energy that may easily be thousands of times the thermal energy. The structure and rheology of the interfacial layers of these systems has been found to have a strong influence on the macroscopic behaviour (structure and rheology) of emulsions and foams. In this work we will briefly present structural details of monolayers of microparticles (1 – 5 µm) at the water/oil interface, both single and binary systems, and we will focus on the dynamics of these monolayers going from the fluid-like to the solid-like state. Using digital video-microscopy we can get the Brownian trajectories of the particles trapped at the interface, from these trajectories different correlation functions and the mean square displacement can be obtained for different states of the monolayer. From these results, for dilute monolayers, the particle self-diffusion coefficient can be extracted and related with the bulk shear viscosities using the appropriate hydrodynamic theories. For the dense states both direct and hydrodynamic interactions must be consider in order to explain the sub-diffusive behaviour. These results will be compared with the ones obtained using optical-tweezers to trap the same particles at the interface.
Imaging of drops on superhydrophobic surfaces in the Cassie and Wenzel states

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Describing wetting of a liquid on a rough or structured surface is a challenge, because of the wide range of involved length scales. Recently, we demonstrated the capability of laser scanning confocal microscopy (LSCM) to image water drops on an array of micropillars in the Wenzel state [1]. LSCM allowed us to quantitatively analyze the local and large-scale drop profile simultaneously. The "microscopic" contact angles close to the substrate varied by up to 30° along the contact line. The drop profile approached its spherical shape only at a length scale one order of magnitude larger than the pillars’ height. The height and position dependence of the contact angles can be understood from the interplay of pinning, complex 3D substrate topography and minimization of the air-water interfaces. Thus, by using LSCM, pinning can be quantitatively studied as a function of the size, shape and chemistry of the substrate asperities.

Next, I applied LSCM to image superhydrophobic surfaces. Stable Cassie drops are obtained by silanization the micropillars with perfluorinated molecules and increasing of height of the pillars. LSCM images directly the air layer between water and the substrate, proving that a drop is indeed in the Cassie state. The dependence of the contact angle on the height is determined, showing that variations decay faster than theoretically expected. The analysis of the curvature of the water-air interface between pillars allows to estimate the stability of the Cassie state. By lowering the liquid surface tension or pillars’ height the Cassie state becomes metastable, allowing to monitor the Cassie-to-Wenzel transition. A first set of dynamic measurements is presented. LSCM opens a novel approach to study wetting dynamics, which is of high scientific and technological interest.

Organizaiton of amphiphilic liquid crystals at the air/water interface

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Amphiphilic liquid crystals do not only show a thermotropic polymorphism but often also form stable Langmuir films at the air/water interface. One characteristic feature that was observed for such systems is a wide plateau in the surface-pressure/area isotherm. This was explained with the formation of a stable triple layer by “roll-over collapse” of a monolayer [1]. We studied p-terphenyl derivates, terminally substituted with two hydrophobic n-alkyl chains and laterally substituted with a hydrophilic trioxyethylene unit [2]. Variation of the n-alkyl chain length gives rise to very different phase behaviour at the air/water interface. Molecules with long terminal n-alkyl chains (C16) crystallize in two steps under compression of a monolayer. This was concluded from Infrared-Reflection-Absorption-Spectroscopy (IRRAS), which was used to monitoring the shift of the CH2 stretching vibrational frequency and grazing incidence X-Ray diffraction (GIXD). Molecules with shorter n-alkyl chains (C10) do not crystallize upon compression. Instead, they show a long plateau in the Langmuir isotherm. We used angle dependent IRRAS to determine the layer thickness and the orientation of the liquid crystal cores at beginning and end of the pseudo-plateau. The electron density profiles were determined by X-ray reflectivity measurements. The results indicate that at the beginning of the plateau the liquid crystals are organized in a monolayer with the liquid crystal cores being oriented parallel to the air/water interface. Upon compression a multilayer is formed. The multilayer consists of one well oriented bottom layer and an unoriented top layer with isotropically distributed terphenyl cores. Thus, the mechanism of the multilayer formation is not a roll-over collapse, but exclusion and lift off of molecules from the water surface which leads to a loss of their orientation.

Lyotropic liquid crystal engineering – ordered nanostructured small molecule amphiphile self-assembly materials by design

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Ordered nanostructured lyotropic liquid crystalline mesophases may form in select mixtures of amphiphile and solvent. Design principles to engineer low energy amphiphile self-assembly materials have advanced to the extent where they can be used to enhance product and process formulations, including drug delivery vehicles, medical imaging contrast agents, and integral membrane protein crystallisation media. In this presentation we will highlight three end-use applications for lyotropic liquid crystalline mesophases. We focus upon the generation of ‘dilutable’ lyotropic liquid crystal phases with two- and three-dimensional geometries from amphiphilic small molecules. We demonstrate that oral delivery of self-assembled nanostructured colloidal particles consisting of 5-fluorouracil (5-FU) lipid prodrugs results in a highly effective, target-activated, chemotherapeutic agent, and offers significantly enhanced efficacy over a commercially available alternative that does not self-assemble. We show that self-assembled nanostructured colloidal particles containing chelating amphiphiles complexed with gadolinium ions have improved efficiency over commercial magnetic resonance imaging contrast agents. We explore the factors governing integral membrane protein crystallisation in lyotropic liquid crystalline mesophases.
Synergistic mixing in ternary surfactant mixtures: a surface and solution study

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The adsorption of surfactant mixtures at interfaces is important in the context of understanding and optimising processes such as lubrication and detergency. Surfactants are typically obtained and used as mixtures in formulations due to the synergistic effects and enhanced performance over those of single surfactant systems, and yet there are many aspects that are still relatively poorly understood. The surface adsorption and solution self-assembly behaviour of complex multicomponent surfactant mixtures at the air/water interface can be investigated using Neutron Reflectivity (NR) and Small Angle Neutron Scattering (SANS). A comprehensive investigation has been carried out into the surface and solution properties of a ternary mixture of three conventional surfactants representative of most current detergent formulations; the non-ionic surfactant octaethylene glycol monododecyl ether, C\(_{12}\)E\(_8\) with anionic surfactants sodium dodecyl benzene sulfonate, LAS and sodium lauryl ether sulfate, SLES. Neutron reflectivity reveals interesting non-ideal surface behaviour in the form of highly dominant adsorption of C\(_{12}\)E\(_8\) and LAS, with very low relative adsorption of SLES. At a SLES solution composition of 0.9 mole fraction, only 0.42 mole fraction of the air/water interface is composed of this surfactant, signifying a greater competition for its incorporation into aggregate phases in solution. All mixtures are in the form of small interacting globular micelles, even at high solution concentrations (50mM), as indicated by SANS. Such complex multicomponent surfactant mixtures have not extensively been explored and understanding the factors which drive adsorption in complex mixtures is of great importance.
Phase and aggregative behavior in binary "water+glycerol" solutions of amphiphiles for stabilization of carbon nanotubes

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Among materials performing optical power limiting (OPL) of laser radiation probably the best ones are suspensions of carbon nanoparticles, in particular, suspensions of single wall carbon nanotubes (SWCNT) stabilized by amphiphiles. Practical OPL materials must be thermally stable, including sub-zero temperatures - so we used a binary solvent "water + glycerol(67 % wt)". The question, however, stands about the behavior of surfactant environment of SWCNTs in such a medium at low temperatures. We studied critical micellar concentrations and Krafft boundaries of sodium dodecylbenzensulfonate (SDBS), sodium dodecylsulfate (SDS) and a blend of SDS and ionic liquid (IL) hexylmethylimidazolium chloride in "water + glycerol" solvent. Micellar surface charges, sizes and composition (in case of mixed micelles) were calculated on the platform of molecular-based model of Gibbs aggregation energy.

The data on amphiphile aggregation were correlated with the spectra of optical absorbance of the suspensions of SWCNTs. The systems were stored for a year with varying temperature from -40 to + 50 °C. All the systems are temporally and thermally stable. The fact that the systems are stable even at the temperatures lower then Krafft values confirms that adsorption of the amphiphiles on SWCNTs plays a significant role alongside aggregation phenomena in stabilizing of suspensions.

All the systems provide relevant OPL properties with respect to laser irradiation in pulse-periodic regimes. Glycerol-containing systems show good resistivity to optical bleaching.

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Equilibrium between miscible pseudo-phases for θ-shaped surfactants

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Theta-shaped (θ) rigid surfactant molecules such as COSAN show complex multi-scale patterns in aqueous solutions as seen by small angle neutron and X-ray scattering over large q-window (qmax/qmin>100). At wide angles a correlation between COSAN (bis(1,2-dicarbollide) cobaltate) is seen, while oligomeric aggregates that correspond to charged micelles scatter in the intermediate q-regime (~0.1nm⁻¹). The low-q part finally exhibit a scattering intensity that is modelled by vesicles made of one or several monolayers of adjacent COSAN.

Vesicles form at lower concentration than micelles (Angewandte Chemie 2011 vol 123 p5410). However, there is a coexistence range of these two aggregates. Therefore, the scattering spectra can be modelled as the sum of two intensities. Exploitation of absolute scale with and without salt allows determining the area per molecule in the two pseudo-phases, as well as equilibrium shape of the aggregates.

Thermodynamic reasons of coexistence of vesicles and micelles are consistent with cryo-TEM and surface tension measurement. Entropic terms like required of Onsager transition favour the enclosure of vesicles, as predicted by Ninham and Marcelja twenty years ago.

Some points of lateral equation of states involved can be derived from the quantitative exploitation of SANS patterns; SAXS is less quantitative since contrast with water is very low. Last but not least, a new and direct method for determining the free energy of transfer between micelles and vesicles seen as miscible pseudo-phases is proposed.
Effect of phase separation on the properties of lipid monolayers

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We used computer simulations to study the effect of phase separation on the properties of lipid monolayers. This is important for understanding lipid-lipid interactions underlying lateral heterogeneity (rafts) in biological membranes, and the role of domains in regulation of surface tension by lung surfactant. Molecular dynamics simulations with the coarse-grained force field MARTINI were employed to achieve large length (100 nm in lateral dimension) and time (tens of microseconds) scales. Lipid mixtures containing saturated and unsaturated lipids and cholesterol were investigated under varying surface tension and temperature. Compositional lipid de-mixing and coexistence of liquid-expanded and liquid-condensed phases, as well as liquid-ordered and liquid-disordered phases was reproduced. Formation of the more ordered phase was induced by lowering the surface tension or temperature. Phase transformations occurred via either nucleation or spinodal decomposition. Using cluster analysis combined with Voronoi tessellation we characterized in detail the structure and dynamics of monolayers with domains. Partial areas of lipids in each phase, phase composition, boundary length, and line tension were characterized as a function of surface tension. At low surface tensions, the monolayers rich in cholesterol manifest spontaneous curvature of domains. Lateral diffusion of lipids is significantly slower in the more ordered phase, as expected. The calculated growth exponents indicate that upon nucleation the growth of a more ordered phase is controlled by Ostwald ripening at early stages, followed by Brownian coagulation.

We also simulated lipid monolayers connected to bilayer reservoirs in water, which are relevant for the function of lung surfactant. The distribution of phases between the monolayers and bilayers was determined, confirming the “modified squeeze-out” mechanism. Nucleation of bilayer folds upon monolayer collapse occurs in the more disordered phase in the case of flat domains, and at the phase boundary in the case of domains with curvature. The latter also facilitate monolayer collapse from the interface.
**Spontaneous oscillation mechanism of liquid membranes of a water/oil/water system using a time-resolved quasi-elastic laser scattering**

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Much attention has been paid to the oil/water interface in order to understand the dynamics of oscillatory interfacial mass transfer phenomena and various bio-oscillations in excitable membranes. The chemical oscillation induced by a Marangoni convection has been extensively studied using a time-resolved quasi-elastic laser scattering (QELS) method, which can measure interfacial tension in a non-contact manner. In this work, a detailed and systematic experimental study on a water/oil/water system has been carried out. The potential, the surface tension and the convection were measured simultaneously and their correlation has been considered.

The oil/water interface was constructed as follows. Oil: Nitrobenzene solution containing 1 mM TBATPB. Water 1: A hollow plastic tube was immersed in the nitrobenzene solution. A mixture of aqueous 0.5 mM CTAB and 0.1 M 1-butanol was added into this tube. Water 2: Water containing 0.1 M sodium chloride or 0.1 M sucrose solution. The potential oscillation across water 1 and water 2 through oil was measured. The interfacial tensions at the water 1/oil (w1) and the water 2/oil (w2) interface were taken by QELS.

The behavior of the potential oscillation strongly depends on the substance adding to water 2 phase. The oscillation of interfacial tension at the w1 interface was not be observed, while the synchronized oscillation of it at the w2 interface was clearly detected. Further, strong convection could be recognized at each potential oscillation. CTAB surfactants diffuse from water 1 to oil phase, subsequently absorb at the w2 interface. As a result, the interfacial tension gradient takes place, giving rise to the Marangoni convection and chemical oscillation. The variation in interfacial tension strongly correlated to the kind of substances contained in water 2 phase, meaning that the absorbed mount of CTAB at the w2 interface depends on it.
Influence of dynamic arm exchange on the crystallization and the glass transition of star polymers

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Associative diblock copolymers are composed of a solvophobic and a solvophilic block. In water, and beyond a critical micelle concentration (CMC), the hydrophobic blocks will self-assemble reversibly to form polymeric micelles while maintaining a dynamic exchange of unimers between micelles. Dense suspensions of micelles jam leading to a liquid-solid transition. In the solid state, micelles can arrange themselves in a crystalline order. If the exchange of unimers between micelles is suppressed, they become no longer dynamic and behave like star polymers. The latter show similar general behavior in dense suspensions as micelles but with significant differences in the rheology and the structure. Notably, the crystal order of star polymers is difficult to achieve. One way to create star polymers is to crosslink the hydrophobic core of polymeric micelles. After cross-linking, the exchange of unimers between micelles is no longer possible. In this way, the effect of arm exchange can be studied for an identical system. In this study, we investigate the influence of the dynamic exchange of unimers on the behavior of micelles and stars. For this purpose we synthesized the amphiphilic diblock copolymer poly(ethylene oxide)-b-poly(methacryloyloxyethyl acrylate) (PEO-b-PMEA) that associates dynamically in water, but whose core can be fixed by photo-crosslinking (UV). In this way, stars are formed that have exactly the same structure as the initial micelles. The effect of eliminating dynamic arm exchange on the rheology and the structure of dense suspensions was investigated. We will show that the reason why polymeric micelles easily form a crystalline order is that they can adapt the number of arms.
Core-shell nanoassemblies with a liquid-crystal-like cores

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Formation of polyelectrolyte–surfactant (PE–S) complexes of poly(ethylene oxide)-block-poly(methacrylic acid), (PEO 705–PMAA 476) and N-dodecylpyridinium chloride, (DPCl) in aqueous solution was studied by static and dynamic light scattering, (SLS, DLS), small-angle neutron scattering, (SANS), small-angle X-ray scattering, (SAXS) and cryogenic transmission electron microscopy, (cryo-TEM). While it was found previously (Macromolecules 1997, 30, 3519) by microcalorimetric titration that in a similar system (PEO 176–PMAA 166) crystallization of aliphatic tails of N-dodecylpyridinium bromide did not occur, in our system it was evidenced by SAXS that upon addition of DPCl to fully ionized PEO 705–PMAA 476 the ordered arrangement of the surfactant occurs in a certain range of PEO 705–PMAA 476 concentrations and surfactant-to-polyelectrolyte charge molar ratio (Z). Four different regions have been identified in the behavior of the PEO 705–PMAA 476/DPCl system: (i) coexistence of loose aggregates of electrostatically bound surfactants to PMAA block with free and almost unperturbed copolymer coils at Z<<1, (ii) formation of aggregates containing ill-defined cores formed by a low density solution of non-ordered DPCl micelles attached to coiled PMAA chains (beads-on-a-string nanoparticles) in the range 0.38 < Z < 1, (iii) formation of compact core-shell nanoparticles with a core formed by densely packed ordered (crystalline) DPCl micelles and PEO shell at 1 < Z < 3.6 and (iv) the region of coexistence of the core-shell nanoparticles with free DPCl micelles (Z>3.6). In the region 0.38 < Z < 1, the nanoparticles with non-ordered cores coexist in a mixture either with a fraction free chains and large swollen nanoparticles decorated by surfactant micelles (at lower Z) or with the core-shell nanoparticles (at higher Z). At all stages, the PE-S complexes were characterized in detail in terms of molar mass, size, shape, and internal structure.

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How small polar molecules protect membrane systems against osmotic stress

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We investigate how small polar molecules, urea and glycerol, can act to protect a phospholipid bilayer system against osmotic stress. The osmotic stress can be caused by a dry environment, freezing, or through exposure to aqueous systems with high osmotic pressure due to solutes like in saline water. A large number of organisms regularly experience osmotic stress and it is a common response to produce small polar molecules intracellularly. We have selected two ternary systems of urea–water–dimyristoylphosphatidyl-choline (DMPC) and glycerol–water–DMPC as model systems to investigate the molecular mechanism behind this protective effect, and we put a special emphasis on applications in skin care products. Using solid-state NMR, DSC, X-ray diffraction and sorption-microbalance measurements we study the phase behavior of the lipid system both exposed to an excess of solvent of varying composition and for systems exposed to water at reduced relative humidities. In this we have arrived at a rather detailed thermodynamic characterization. The basic findings are: i) In excess solvent the thermally induced lipid phase transitions are only marginally dependent on the addition of urea(glycerol). ii) For lipid systems with limited access to solvent the phase behavior is basically determined by the amount of solvent irrespective of the urea(glycerol) content. iii) The presence of urea (glycerol) have the effect to retain the lipid in liquid crystalline phase down to low relative humidities (64% for urea, 75% for glycerol at 27°C), whereas the transition to the gel phase occurs already at a relative humidity of 94% in pure water, demonstrating the protective effect of the polar molecules against osmotic stress. iv) In skin care products urea and glycerol are referred to as a moisturizer, which we find slightly misleading as it replaces the water while keeping the physical properties unaltered.
pH sensitive tubules of bile acid derivative: a new mechanism of tubule opening

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The three-dimensional structures with nanoscopic dimensions that are yielded by the self-assembly of surfactants are of particular interest for their potential nanotechnological applications. Within self-assembly systems those with stimuli-responsive proprieties are worth of attention due to their reversible tunable aggregation. In this work, we demonstrated that a cholic acid derivative, obtained by substituting the OH group in position 3 with a naphtoylamine residue, forms pH sensitive self-assembly tubules in aqueous solutions. The peculiar amphiphilic structure of the bile acids produces very ordered supramolecular arrangements not easily rationalizable on the basis of the conventional geometric rules of surfactant packing. The self-assembly properties of derivative were studied by focusing on the structure of the supramolecular aggregates and their sensitivity to variation of pH. Circular dichroism was used to investigate the molecular packing in the aggregates and their transitions. Transmission electron microscopy, small angle X rays and light scattering were used to determine the aggregate morphologies and their size variation in the transitions. The results point out to a new mechanism of tubules opening where a kind of explosion occurs probably determined by a drastic pH induced variation of their molecular packing.
Interactions between phospholipid mesophases and nanoparticles

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The phase transitions of phospholipids are known to be energetically analogous to the membrane elastic deformation events occurring during membrane fusion 1. Study of these events with nanoparticles (NPs) can lead to observations of how membrane fusion may be affected by NPs, and so how NPs may cross the cell membrane. This study focuses on how the mesophase transitions of pure and mixed thermotropic phospholipids including dipalmitoyl phosphatidylcholine (DPPC), dioleoyl phosphatidylcholine (DOPC) and dioleoyl phosphatidylethanolamine (DOPE) were altered with presence of NPs. Such knowledge will have important implications to the fundamental mechanism of NP crossing cell membranes, with relevance to nanotoxicity imparted by NPs and a simulation by Reynwar2 has previously studied the effect of NPs on membrane curvature and pore formation.

The phase transitions were observed by small angle x-ray scattering (SAXS) both with and without NPs of various core types, surface chemistry and concentrations to establish how the phase boundary position changed, and how the properties of the NP and its concentration affected this. The mesophases were altered with varying either temperature or pressure3 at a fixed volume of water. We observed altered positions of phase boundaries as the NP concentration and surface chemistry were varied.

Of most interest is the generation of pressure-temperature phase diagrams with DOPE and either hydrophobic or hydrophilic silica NPs of 14 nm diameter. Changes in size of mesophase regions and boundary positions were observed.

This talk will discuss the implications of these results in the context of the study of nanotoxicity.

Semiconductor nanowires as a generic materials technology for opto-electronics, energy and life-sciences applications

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With today’s need for ultra-small devices and probes having dimensions down to the 10 nm range, much attention is paid to different self-assembly methods. In this talk I will describe in some detail the materials physics approach for growth of semiconductor nanowires. These are one-dimensional single crystalline needles, or nanorods, which form via locally controlled growth. I will present some of the “hot” issues in the field, dealing with structural properties and the way we can control the formation of atomically sharp, so called, heterostructures between different semiconductor materials. With this background I will illustrate why so much interest is shown towards the nanowire field, with a presentation of nano-electronic (transistors) and nano-photonic (on-chip optics) devices, as well as for energy-relevant application areas for photo-voltaics (solar cells) as well as for solid state lighting (light-emitting diodes, LEDs). Finally, I will present a few of the areas where colleagues in the biology and medical field find interesting uses for our nanowire structures.
High-resolution cryogenic-temperature scanning electron microscopy: a new tool for the study of nanostructured liquid systems

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Cryogenic-temperature transmission electron microscopy (cryo-TEM) is now accepted as an almost standard tool in the study of complex liquids, i.e., liquid systems with aggregates or building blocks on the nanometric scale. Methodologies have been developed to help capture the nanostructure of liquid systems, while preserving their original state at a given concentration and temperature. Cryo-TEM is now widely used to study synthetic, biological, and medical systems. Originally developed for aqueous systems, it has been also applied successfully in the study of non-aqueous systems. However, this methodology cannot be used to study highly viscous systems, or those containing objects larger than several hundreds of nanometers.

Recent developments in high-resolution scanning electron microscopy (HR-SEM) have made it an ideal tool for the study of nanoparticles and colloids in viscous systems or in systems containing large objects (hundreds of nanometers and larger), in which small (nanometric) features are to be imaged. Improved field-emission electron guns, electron optics and detectors have made it possible to image nanoparticles down to a few nanometers. Liquid nanostructured systems can now be studied by cryo-SEM, using much-improved cryogenic specimen holders and transfer systems. In recent years we have developed a novel specimen preparation methodology for cryo-SEM specimens that preserves the original nanostructure of labile complex liquids at specified composition and temperature, quite similarly to what had been done in cryo-TEM.

In my talk I will describe briefly the state-of-the-technology of cryo-SEM, and demonstrate the application of our new methodology in nano- and biotechnology. Among others I will describe applications in the study of microemulsions, liotropic liquid-crystals, mesophase formation, and carbon nanotubes dispersed in super-acids.
Size and conformation relationships in biomacromolecules, aggregates and colloids revealed by asymmetrical flow field-flow fractionation

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Size and structure characterization of large and polydisperse biomacromolecules and aggregates as well as colloidal particles can prove to be a challenging task. Asymmetrical flow field-flow fractionation (AF4) is a powerful separation technique with many applications in this area. An important property of AF4 is the gentle separation conditions such as small shear forces and small internal surface area minimizing degradation and unwanted interaction. When coupled to suitable detectors, size distributions (molar mass and radius) can be obtained and a range of physico-chemical properties can be investigated over the entire size distribution. Furthermore, the method, together with data treatment developed within our research group, also provides information of apparent densities and conformation throughout the size distribution.

Results from several biomacromolecular systems will be discussed in this presentation including both polysaccharides and proteins in solution or dispersions. The results show that relevant properties regarding functionality can be obtained from complex and highly polydisperse mixtures. In turn, this has enabled the comparison model structures for macromolecular conformation as well as structure of colloidal aggregates.

References:
Heterogeneous and anisotropic dynamics of a 2D gel

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We shall report recent results [1] obtained by X-ray Photon Correlation Spectroscopy (XPCS) on a bidimensional (2D) gel formed by a Langmuir monolayer of gold nanoparticles whose morphology is thoroughly characterized by scanning microscopies while the mechanical properties are characterized by Interfacial Shear Rheometry [2]. Its morphology shows a complex structure with features on a hierarchy of different sizes following a Levy distribution. The dynamics observed in the film is anisotropic, as it is confined in the plane of the interface.

Moreover it is highly heterogeneous and super-diffusive on the nanoscale. Heterogeneity is characterized in great detail, as the excellent SNR of our XPCS experiment allows a rare experimental determination of the fourth order time correlation function which is compared to the usual second order correlation functions. Different time scales, associated with fast heterogeneous dynamics inside 2D cages and slower motion of larger parts of the film, can be identified from the comparison of the different correlation functions. Moreover, the fluctuation dynamics measured by XPCS is discussed in view of the models existing in the literature [3] by making use of the accurate characterization of the interfacial mechanical response functions which we have measured by Interfacial Shear Rheology on the same monolayer [4]. Time permitting, preliminary results on the rheology of the same system, observed on the micro scale by Multiple Particle Tracking, shall be presented.

References:

Formation of model membranes supported by nanostructured sensing substrates

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Supported phospholipid bilayers are commonly used as in vitro models of cellular membranes. As membrane models edge further towards the complexity found in nature, more complementary methods are needed to widen our understanding of the interactions at the interface. Membranes formed on sensing platforms may provide quantitative information about interactions between membrane components. Methods for fabrication of reproducible, ordered nanowire arrays, or “nanowire forests”, capable of force measurement have been previously developed (1). We now seek to use the sensing potential of these well-characterized sensing substrates to study the mechanical properties of supported phospholipid bilayers. Our overall aim is to facilitate the formation of single bilayers on the surface of silicon nanowire forests. However, the micro- and nanoscale topography makes the nanowire forest a challenging substrate to work with. Due to differences in surface properties and curvature these forests differ from the planar substrates that are commonly utilized for bilayer supports. In this early stage, we have investigated different methods of formation of complex lipid bilayers on silicon nanowire forests using confocal fluorescence microscopy. We chose silicon nanowires due to the well-described ability of silicon oxide to promote bilayer formation. By varying the lipid composition and deposition techniques, including vesicle fusion and Langmuir-Blodgett Schaeffer methods, we have systematically investigated the formation of bilayer structures. These supported bilayers can be made using lipid compositions that mimic biological membranes.

Role of the cell-division cycle on the uptake of nano-sized materials

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The interaction between nano-sized objects and living systems is currently being extensively investigated. The nanoscale is privileged because many endogenous biological processes operate on the scale of several tens of nanometers. Engineered nanoparticles can thereby engage with biological processes in fundamentally new manners, thereby holding great promise for targeted delivery of drugs, but may also constitute a hazard that living systems have not evolved to manage.

The role of nanoparticle size and surface properties on their cellular uptake is well-studied. Less attention has, however, been given to biological variables, such as the presence of continuous cell division that occurs for most cell systems studied. We show that a range of model nanoparticles do not affect the normal cell-division cycle, and that nanoparticle uptake occurs throughout the cycle. As a cell divides, the internalised nanoparticles are split between the daughter cells. This causes a characteristic ranking during continuous exposure, where cells just about to divide have a higher intracellular load, followed by cells half-way to division, and finally cells that have just divided have the lowest load. The phenomenon is found in a wide range of nanoparticle-cell systems, and we corroborate the finding with a theoretical model.

From a broader perspective, the findings emphasise the importance of considering the evolution of the cells in nanoparticle-cell studies. A concrete example is that nanoparticle accumulation levels saturate during continuous exposure due to cell division. This effect can easily be mistaken for export of the nanoparticles out from cells, obscuring what is, in fact, a permanent nanoparticle load inside cells.

References:
Fabrication of nanoparticle-based periodic structures has attracted considerable attention in material science because new synergistic functions could be derived from such structures. Among organic soft materials, liquid-crystalline (LC) organic dendron is one of the most representatives to form spherical self-assembled aggregates. Such spherical objects spontaneously form 3D self-organized structure. In the present study, we focused our attention on introduction of such self-organization ability into inorganic NPs. One representative strategy to obtain dendrimers is achieved by modification of internal core with hyper branch molecules. Here, if monodispersed gold NPs are used as the internal core, well-defined organic-inorganic hybrid dendrimer would be obtained as a novel-type of functional material. Initially, CO$_2$H-modified gold NPs A1-A3 were prepared as the internal core of the dendrimer. Also, 1st, 2nd, and 3rd generation dendrons G1-G3 with an amino-group at the apex were synthesized. Modification of A1-A3 by G1-G3 was carried out by amidation reaction. From TEM observation, the particle size of A1, A2, and A3 were calculated as 5.9, 6.8, and 6.8 nm, respectively. In the case of G2 modified A2 (G2/A2), G2/A2 formed a hexagonal 2D array with the interparticle distance of 14 nm on a TEM grid. SAXS and DSC measurements showed that G2/A2 exhibited thermotropic LC phases in wide range of temperatures. At 130 °C, G2/A2 formed an unusual hexagonal superlattice structure containing half the number of NPs in the centered column. The G2/A2 structure changed into a simple cubic (SC) LC phase at 170 °C. This result shows that the self-organization ability was introduced into gold NPs, and G2/A2 spontaneously self-organized into the SC LC phase. The effects of dendron generations and modification amounts have also been investigated.
Polyelectrolyte nanocapsules assembled onto liposome templates

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In the last decade significant progress has been made in the fabrication and engineering of nano and microdevices for the delivery of active molecules. Recently, to enhance the active substance circulation in the body, new carriers have been proposed, namely, nano- and micro-capsules where polyelectrolytes are assembled through the Layer-by-Layer technique.

We propose the design of novel capsules assembled onto liposome templates with a diameter smaller than 100 nm. Following the LbL procedure, the polymers alginate and chitosan were alternatively adsorbed on the liposome templates, thus achieving the formation of core shell structures able to be loaded with drugs. Dynamic Light Scattering (DLS) measurements showed that the size of the nanocapsules was of 300 nm diameter after the deposition of eight layers. After assembling of core-shell complexes, in order to achieve the formation of hollow polyelectrolyte structures, the lipid component was removed according to the core removal protocol recently suggested by us [1]. The obtained nanostructures were thereafter characterized by means of DLS, ζ - potential and Scanning Electron Microscopy (SEM). SEM analyses on hollow capsules demonstrated the presence of structures indistinguishable in size and shape from the parent core-shell system. ζ-potential measurements performed to analyze the surface charge of the hollow nanocapsules after the core dissolution indicated a good stability. These nanodevices were demonstrated to be pH [2] and temperature sensitive and thus are potentially useful for molecular encapsulation and triggered release applications.


Electrostatic complexation between PAA coated nanoparticles and different type of positive polyelectrolytes

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Here we present new results dealing with electrostatic complexation between magnetic nanoparticles and oppositely charged polyelectrolytes. Polyacrylic acid coated iron oxide nanoparticles presented remarkable stability against brine solutions or cell culture medium. Because of numerous carboxylate functions, those nanoparticles can be associated with positive polyelectrolytes, that can be either hydrophilic or amphiphilic. We have developed an original synthesis pathway to co-assemble magnetic nanoparticles into spherical or elongated aggregates. Those aggregates conserved the superparamagnetism of the constitutive nanoparticles. We also used amphiphilic PDMS copolymer to compatibilize magnetic nanoparticles with PDMS matrix. This magnetic elastomer can be used in microfluidic systems to capture magnetic materials inside the channel.
A droplet deformation as a mini-rheometer to evaluate interfacial viscoelastic moduli of solid nanoparticles-laden liquid/liquid

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Solide nanoparticules-laden droplet may jam in an anisotropic shape when get deformed, depending on the concentration C and the affinity of the solid particles with the two liquid phases. Therefore we investigated in this study the relaxation of an isolated droplet of diluted aqueous suspensions of modified silica nanoparticles. The deformation of the droplet was monitored using a homemade counter rotating shear device developed at PCI. We used a Newtonian polyisobutene as a continuous phase and we recorder the droplet retraction after a controlled step strain $\gamma$. At low $\gamma$, the droplet jammed into a non spherical shape resembling an ellipsoid of revolution that became more anisotropic when C increases. Thus, by deducing an apparent jamming stress $\sigma_{aj}$ of the deformed droplet given by the gradient of Laplace pressure at its interface, we found that $\sigma_{aj}$ showed a power law dependence on C. In addition, we measured the interfacial viscoelastic moduli of a model planar water/PIB interfaces using a shear stress controlled rheometer equipped with an interfacial geometry (Double Wall Ring) using the same silica nanoparticles. By increasing C, the water/PIB interfaces showed a liquid-solid transition and a power low dependence of the elastic modulus $G''$ with C in the solid state. Remarkably, the interfacial elastic moduli were correlated to the anisotropic shape of the droplet since we found that $G''$ was quasi-proportional to $\sigma_{aj}$ in all the experimental conditions of this study. Interestingly, the droplet behavior undergoing shear deformation could be used as a mini-rheometer to evaluate the viscoelastic moduli of solide particles-laden liquide/liquide interfaces.
Chiral inorganic nanomaterials: chiral construction via self-assembly of amphiphiles and inorganic precursors

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Chiral inorganic nanomaterials was synthesized by cooperative self-assembly of chiral or achiral amphiphiles or DNA and various inorganic precursors. The chiral mesoporous silica exhibits a novel chiral mesostructure with hexagonally ordered chiral channels twisted from the two-dimensional hexagonal p6mm. The helical propeller-like packing of the amphiphiles, which is induced by the asymmetric shapes of chiral or achiral amphiphiles, is considered to be the origin of the chiral mesostructure of CMS. The diversity of the silica precursors enables CMS with designable functional channel surfaces and framework. It was found that the primary chiral structure of the helical propeller-like packing of the chiral amphiphiles can be imprinted by the functional groups of co-structure-directing agents (CSDAs) and mobilized on the channel surface of extracted CMS, which paves a way for supramolecular chiral transcription and recognition. Recently, we have succeed in the synthesis of impeller-like helical DNA-silica complexes by co-structure agent route, and the handedness of helical architectures was reversed by the change of quaternary ammonium:DNA molar ratio, temperature and pH. The rules and mechanisms involved in the synthesis of CMS and related materials provide us with a deeper insight into the nature of chirality, regarding the chiral supramolecular assembling process.

References:
Scanning probe electro-oxidative lithography of OTS monolayers on ITO films: a Scanning Kelvin Probe Microscopy investigation

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Transparent conductive oxides like indium tin oxide (ITO) play a pivotal role in a wide range of innovative applications, such as new generation solar cells. In many of these applications the tailoring of surface properties on the nanometer scale represents a highly desirable target. The local oxidation of self-assembled monolayers (SAMs) using a scanning probe is a promising technique to achieve surface modifications on the nanometer scale.[1] So far, electro-oxidative lithography of SAMs has been reported mainly on Si wafers while there are no previous reports on transparent oxides. Here, we demonstrated for the first time the oxidative lithography of n-octadecyltrichlorosilane (OTS) SAM deposited onto an ITO layer.

The electro-oxidative lithography of OTS-ITO results in a local overoxidation of the substrate, as confirmed by tests onto bare ITO. In addition, the monolayer oxidation causes the conversion of –CH3 groups into hydrophilic functionalities. The latter phenomenon was proven by the site-selective deposition of Ag nanoparticles onto electro-oxidized areas.

The lithography process was compared to that on OTS-Si substrates and systematically investigated by means of Scanning Kelvin Probe Microscopy (SKPM). Surface potential images of OTS-Si and OTS-ITO showed remarkable similarities, while the bare substrates exhibit completely different behavior. Such significant differences, which can be attributed to the presence of the oxidized monolayer, are not observable in the case of topography or lateral force images. Thus, SKPM analysis represents a powerful and unique tool to investigate the mechanism of the monolayer oxidation.

One-step screening process for optimal alignment of (soft) colloidal particles

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The directed assembly of colloidal particles into materials with high packing density, sufficient long-range order and a periodicity on the mesoscale is important for the preparation of photonic, electronic and sensing devices. Challenges in the area of assembly lie in the demand for rapid, scalable and controllable processes [Adv. Mater. 2009, 21, 1897–1905].

Therefore, we developed gradient wrinkle surfaces for one-step screening processes leading to optimal alignment of colloidal particles on nanostructured surfaces.

The periodically structured surfaces are produced by a lithography-free approach based on controlled wrinkling [Soft Matter 2006, 2, 310-323]. A mechanical mismatch between a hard top layer (induced by plasma oxidation of the elastomer) on a soft substrate creates sinusoidal surface wrinkles. By tuning the thickness of the oxidized layer, amplitude and wavelength dimensions of the wrinkles range from several nanometers to one micron. The 2-dimensional wrinkle gradients cover this size range on a single substrate and give way for one-step screenings regarding colloidal assembly.

We use hard and soft colloids – different microgel systems and silica particles – for the combinatorial experiments on the wrinkle gradients. We are able to optimize the assembly regarding the dimensions of the underlying structure and packing density in a one-step process. By comparing the behavior of soft and hard particles (packing density, long-range order, deformation of soft colloids), we draw conclusions on particle-particle and particle-substrate interactions.
Facile synthesis of magnetic silica micro- and nano-rods

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The use of micro and nano-building blocks as fillers to enhance the property of composites is a topic of great interest. The most used ones are spherical nanoparticles, which can be prepared using a variety of methods. Nevertheless, there is a growing interest in the use of anisotropic building blocks, such as nanotubes, platelets, rods, ellipsoids, etc. In this work we propose a new and reliable method for the production of large quantities magnetic silica micro and nano-rods. This method is based on the spinodal decomposition of a silica precursor leading to a bicontinuous phase, the structure of which is controlled by the incorporation of polymer-magnetite nano-spheres with the aid of an external magnetic field. Our system is based on the silica precursor TMOS and the polymer PEG, which undergoes a phase separation following a spinodal decomposition due to the chemical cooling induced by the condensation polymerization of the silica precursor. Polymer-magnetite nanoparticles are incorporated in the silica rich phase due to the strong affinity of the magnetite to the silica precursor. The accumulation of the nanocolloids in the silica rich phase during the spinodal decomposition leads to the formation of long rod-like structures in the presence of an external magnetic field, which form a monolith. At the end of the reaction, the application of strong ultrasonication leads to the disintegration of the monolith and to the recovery of the rods. Different parameter such as rods diameter and length can be tuned by varying reactant concentration, the applied magnetic field and the sonication time. The applied magnetic field has an enormous influence on the length of the produced rods and depending on the kind of field applied, homogeneous or alternating, the length can be varied by more than two orders of magnitude.
Formation of metal oxide nanostructures at liquid/solid and liquid/liquid interfaces in miniemulsion

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The crucial importance of processes occurring at particle surfaces and droplet interfaces is well-known for colloid chemists and, in general, for materials scientists. However, the inorganic crystallization on surfaces and—more dramatically—at interfaces remains still today a complex and challenging topic, with great potential in materials synthesis. Indeed, the formation of inorganic matter at solid-liquid and liquid-liquid interfaces can be used to prepare organic-inorganic hybrid materials and hollow particles [1].

In a first approach, we have prepared polymer/metal oxide hybrids by controlled crystallization of the inorganic component on the surface of functionalized polymer particles. The polymer particles, prepared by miniemulsion polymerization, are decorated at the surface with phosphate, phosphonate or carboxylate groups, which act as nucleation centers for the formation of the metal oxide nanocrystals. The crystallization is driven by the addition of a base to suspensions of the polymer particles containing the corresponding precursor. Depending on the metal oxide deposited, materials with magnetic, optical, and catalytic properties can be prepared.

In a second approach, we have focused on the formation of transition metal oxides and hydroxides at the liquid-liquid interface in miniemulsion droplets. With the exception of some research on hollow silica particles, little attention has been paid so far to the possibility of conducting crystallization at the droplet interface. We have recently shown that hydroxides of group 4 transition metals (titanium, zirconium, hafnium) can be precipitated as amorphous materials and converted to the respective oxides by thermal treatment [2]. In other cases, such as CeO₂ or Fe₂O₃, we have observed the formation of crystalline material at the interface even at room temperature.

Observation of chain structure in nanocomposites

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Model systems of nanocomposites are important for our understanding of the structural and dynamical contributions of the constituents to macroscopic properties, and in particular mechanical reinforcement. We study silica-latex nanocomposites obtained by drying of aqueous colloidal suspensions of polyacrylate nanolatex and silica nanoparticles (20nm). In these systems, control of the precursor solution pH allow obtaining various filler microstructures [1, 2].

Here, we follow the evolution of the polymer structure during nanocomposite formation and annealing using contrast-variation small angle neutron scattering. We used mixtures of hydrogenated and deuterated latex to match the silica signal and directly measure the form factor of a single polymer chain. The progressive disappearance of the latex beads by chain interdiffusion and release in the nanocomposites is analyzed with a model of hairy latex beads and an RPA description of the free chains. Our model [3] reproduces the data nicely over the whole q-range and in absolute units, with very few free parameters. In silica-free matrices and nanocomposites of low (7%v) and high (20%v) silica content, the annealing procedure results in a molecular dispersion of chains. However, the interdiffusion of individual chain is delayed in presence of 20% of silica, reaching molecular dispersion only at the strongest annealing. Observations of similar delay have been made in our previous study on an incompatible H/D latex system [2]. For nanocomposites, evidencing by SANS that the silica filler influences the chain reorganization may be the clue for a molecular understanding of the reinforcement effect.

To summarize, we have been able to properly measure the chain conformation at different silica volume fractions in a given state of aggregation (typically ten silica particles per aggregate, percolating at high concentration). It is found that the radius of gyration is not disturbed by the silica loading under these conditions [3]. This result opens the way for a systematic study of the chain structure in complex environments. [1] Oberdisse, Soft Matter 2006 [2] Tatou, Macromol. 2011 [3] Genix, Macromol. 2012
Design of surfaces with mechanical nano-heterogeneities for a better control of cell-material interactions

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Growing cells are sensitive to the chemical composition, the topography and the mechanical properties of their substrate surface. Chemical composition and topography of surfaces used for interaction with biological matter become more and more controlled, either at a supra- or at a sub-cellular level. However, control of the mechanical properties of a surface at the sub-cellular scale remains challenging. The aim of this study is to prepare surfaces presenting mechanical cues at the nanometer scale and to evaluate cell behavior in contact with these surfaces. The adopted strategy consists in coating a substrate showing rigid topographical nanostructures by a thin layer of an elastomer. This layer ensures the homogenous chemistry and topography of the surface, while the rigid structures underneath provide mechanical contrast.

The combination of colloidal lithography and layer-by-layer assembly allows creating a defined topography over wide areas. Glass surfaces are first covered by positively charged polyallylamine which is then put in contact with a solution of 500 nm silica colloids. Particle deposition is regulated by electrostatic interactions and therefore results in a relatively ordered pattern. To obtain a homogenous chemistry and topography, thin films of poly(dimethylsiloxane) (PDMS) are spin-coated on top of the silica colloids. Limited roughness but significant mechanical heterogeneities were measured using atomic force microscopy by acquiring topographical images and mapping force-distance curves, respectively. MC3T3-E1 preosteoblast cells were cultivated for 3 days on substrates containing or not colloids. A significant increase of cell density and cell area was found in presence of colloids under the PDMS layer. Complementary studies have shown that cell adhesion and cell motility were as well affected. These results demonstrate that cells are sensitive to mechanical heterogeneities at a sub-cellular level.
Nanostructured materials for superhydrophobic and superhydrophilic surfaces

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Applications for superhydrophobic and superhydrophilic surfaces have started to immerse as the research on this topic has been active for the past decade. The applications include for example self-cleaning and anti-fogging coatings for windows. In this abstract silicon and hybrid polymer is nanostructured to produce superhydrophobic and superhydrophilic surfaces. These surfaces can be utilized in e.g. microfluidic devices to control liquid spreading or as surface assisted laser desorption ionization plates for biomolecule analyzes.

Nanostructured silicon surface is produced by plasma etching. The formed material is called “black silicon” due to its non-reflective property [1]. The structure on black silicon is then transferred to hybrid polymer by using elastomeric stamp [2]. Both black silicon and nanostructures hybrid polymer were either coated with fluoro polymer to make them superhydrophobic or oxygen plasma treated to make them superhydrophilic. Silicon has been used as a material for microfluidic devices for a long time [3], making the possibility to control its wetting properties interesting. Polymers are however gaining more and more attention and hybrid polymer is proved to be well suited for fabrication of microfluidic components [4]. The contact angles were measured on oxygen plasma treated and Teflon coated black silicon and nanostructured polymer samples by using CAM101 optical tensiometer (Attension, former KSV Instruments). Fluoro polymer coated black silicon contact angle was 170 ° where as that of nanostructured polymer was 160 °. Although the contact angle was little bit lower for nanostructured polymer, the surface can still be considered to be superhydrophobic. The replication process is thus suitable for nanopattern transfer. Same nanostructured surfaces can be made superhydrophilic by instead of fluoro polymer coating treat them with oxygen plasma. The contact angle for both oxygen plasma treated black silicon and hybrid polymer were close to zero.

Nanostructured polymeric hydrogels containing surfactant aggregates for drug delivery

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Polymeric hydrogels are constituted by cross-linked hydrophilic polymers swollen by an aqueous medium. From a macroscopic-functional viewpoint, they behave as viscoelastic materials, while from a microscopic-structural viewpoint they can be considered as polymeric aqueous solutions. This unique combination of properties, together with the low toxicity and high biocompatibility, have promoted hydrogels’ application as biomaterials. Currently, hydrogels are massively used for a variety of biomedical applications including drug delivery, tissue engineering, wound dressing. Since many pharmacological actives are hydrophobic or amphiphilic molecules, we have included surfactants in the hydrogel formulation. In the resulting nanostructured materials, the polymer network could be thought as a macroscopic reservoir of surfactant aggregates that, in turn, act as microscopic reservoir of actives. In this contribution we present the results of our studies on hydrogel formed by UV-photo-crosslinked poly(vinyl pyrrolidone) (PVP), chemically crosslinked chitosan or hyaluronic acid, and poly(vinyl alcohol) (PVA) obtained by the freeze/thaw method. In all of them, we have included single-tailed surfactants (e.g., sodium decylsulfates, alkyltrimethylammonium bromides and the biocompatible alkylglycosides) or double-tailed lipids (e.g., glycerophosphocholines). The resulting materials have been characterized by a combination of experimental techniques, including rheology, small-angle neutron scattering (SANS), electron paramagnetic resonance (EPR), and pulsed gradient spin–echo nuclear magnetic resonance (PGSE-NMR). Polymer-surfactant interactions influence both the morphology of the surfactant aggregates and the visco-elastic behavior of the polymer network. Drug release tests have confirmed that our new biomaterials are suitable for the delivery of a variety of actives, and that the release kinetics can be modulated by a fine tuning of the hydrogel nanostructure.
The fundamental role of hydrogen bonding in polymer adhesion

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First reported in the late 1990’s in the work of Chatellier et al.[1], force plateau in AFM polymer chain stretching experiment has attracted an ever increasing attention from researchers. Particularly in the field of Single Molecule Force Spectroscopy (SMFS), where single polymer molecule is used to probe interfacial phenomena, force plateau is central to the investigation as its magnitude (both in depth and length) reveals much information on the underlying interfacial interaction.

Interestingly, the reported depth of force plateau in many different types of experimental systems only spans a relatively narrow range of value, typically around 90 pN. Common in many of these systems but largely ignored from consideration is the existence of chemical moieties capable of hydrogen bond formation. Despite being routinely implied in the adhesion process, the specific role of hydrogen bonding is, to the best of our knowledge, yet to be a subject of a focused scrutiny.

Here we present our investigation of the fundamental role of hydrogen bonding in polymer adhesion, with particular emphasis on the adhesion at the solid-liquid interface. We have developed an experimental protocol that allows us to use a small ensemble of different types of polymer molecule, in conjunction with different choices of surface and solvency condition, to elucidate the nature and role of hydrogen bonding in adhesion process at the molecular level.

References:

Structure and properties of emulsion-based porous materials

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Polymer foams are cellular materials commonly used for safety applications in many industrial sectors (aeronautic, passive safety gears…). In all applications, their light weight and their mechanical resistance are of great interest. To even improve their performances, the link between their structural parameters (cell sizes, density…) and their mechanical behaviour should be better understood.

It is now well known that such foams may be obtained by polymerizing the continuous phase of highly concentrated emulsions. The advantage of such an emulsion-based strategy is that it becomes possible to take advantage of the good control over emulsion parameters (drop size, dispersed phase volume fraction, continuous phase composition) to elaborate model foams with cells and pores narrow size distributions.

We produced cellular material based on the formulation of model emulsions whose drop size and composition may be continuously tuned. The obtained solid foams are characterized by narrow cells and pores size distributions in direct relation with the emulsion structure. The mechanical properties are examined, by varying independently the cell size and the foam density, and compared to theoretical predictions. Surprisingly, at constant density, Young’s modulus depends on the cell size. This observation likely results from the heterogeneous nature of the solid material constituting the cell walls and we propose a mean field approach that allows describing the experimental data. We discuss the possible origin of the heterogeneity and suggest that the presence of an excess of surfactant close to the interface results in a softer polymer layer near the surface and a harder layer in the bulk.

Structural forces and viscosity of polyelectrolytes and colloidal particles under confinement: AFM as a tool to study rheology

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Solutions of flexible and rigid polyelectrolytes (i.e. NaPSS and DNA) in aqueous medium are investigated with respect to two small scale dynamic parameters. First, the dynamics of oscillatory structural forces in confined geometry were studied by colloidal probe AFM. As the theory suggests, ordering and structural forces are suppressed with increasing polyelectrolyte chain rigidity, presumably due to a longer chain reorientation time [1].

Second parameter is the newly introduced micro-viscosity, measured by an AFM microcantilever [2]. This parameter corresponds to the viscosity in nm scale volume as determined by the range of the microcantilever's Brownian motion (0.01-0.5 nm). While the solutions of flexible polyelectrolyte chains have nearly the same micro-viscosity as the bulk, rigid chains have significantly lower ones as compared to the bulk viscosity. Possible reasons for this significant difference are addressed.

On the other hand, suspensions of colloidal silica particles present structural forces as well, due to an induced ordering under physical confinement. The dynamics of these oscillatory structural forces will be related to the viscosity of the suspensions at various scales of volume. This is possible by controlling the vibration range of an AFM microcantilever by external acoustic or magnetic excitation [3].

DNA condensation and segregation in model bacterial cells

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Bacterial cells are very different from eukaryotic cells; they present no nuclear membrane that confines the DNA and there is no compelling evidence for the existence of histone-like proteins that condense and organize the genome. Microscopic observations suggest that the chromosomes occur in the cell as a compact nucleoid that is phase separated from the cytoplasm. In fact, it was found that nucleoids “in vivo” are embedded in a highly concentrated macromolecule solution (~340 mg/mL), which is expected to favor the compaction of the DNA due to molecular crowding effects [1]. In this respect, it has been shown that cell lysis is accompanied by DNA decompaction [2].

This has inspired a series of Monte Carlo simulations, that uses simple coarse-grain models for the bacterial cell and simulate DNA condensation under crowding conditions. DNA is described as a sequence of hard-spheres connected by harmonic springs, and the soluble crowding proteins simply by hard-spheres, all enclosed in a spherical cell. In this work we have varied the number and charge of the crowding proteins as well as the architecture of the DNA.

We have found that the increase in the number of proteins showed no significant condensation of the polymer chain, within the studied parameters. However, inducing self-association in a few monomers, and concomitant loop formation, led to an increase in DNA condensation induced by the presence of the crowding proteins. Interestingly, highly coupled protein systems were found to lead to the displacement of the DNA from the center to the cell walls.

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The influence of organic salts on the complexation behavior of polymer and sodium dodecyl sulfate

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Combination of polymers and surfactants presents peculiar synergistic behaviors and thus is of importance in colloidal applications. In many situations, salts including inorganic and organic are used together with polymers and surfactants in the technological formulations. The effects of inorganic salt on the polymer-surfactant interactions have been well investigated. Until now, however, the influences of organic salts on the complexation of surfactants on the polymer chains still remain mostly unexplored. In this study, we unravel in aqueous solutions how the nonionic polymers, poly(N-vinyl pyrrolidone) and poly(N-vinly formamide), interact with the SDS, which are conjugated with tetraammonium alkyl bromides or imidazolium ionic liquids. In the presence of the organic salts which are strongly hydrophobic, some unusual association behaviors between polymer and SDS were observed by pyrene solubilization experiments. The occurrence of polymer-surfactant interactions is found to be determined by the degree of affinity between SDS and the salts and also by the molar ratio of SDS to the salts. Therefore, obviously the charges and hydrophobicity on the surfaces of the micelle-like SDS-salt conjugate aggregates are the key factors to promote or to suppress nucleation of the SDS molecules along the polymer chains. The self-diffusion coefficients of polymer, SDS and salt molecules were measured by PFG-NMR. In the case of tetraammonium butyl bromides (Bu4NBr), the cations move almost simultaneously with the SDS molecules as the ratio of [SDS]/[Bu4NBr] is more than one, i.e. Bu4N⁺ is bound tightly with the anionic SDS molecule. In addition, using 2D NOESY NMR, we could conclude that the polymer chain penetrates into the Bu4N⁺-SDS conjugate aggregate. Therefore, as [SDS]/[Bu4NBr] > 1, a ternary complex composed of polymer, SDS and Bu4N⁺ is formed in the aqueous solution. Furthermore, a detailed complexation model, including space-time dynamics, among the polymers, SDS and the salts is proposed in terms of the results from pyrene fluorescence coupled with 2D NOESY and PFG-NMR.
Theoretical studies of polyelectrolyte adsorption under pure electrosorption conditions: non-monotonic response to salt addition

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The main question addressed in this work is: under pure electrosorption conditions, i.e. disregarding non-electrostatic effects, how does the net adsorption of a polyelectrolyte at an oppositely charged surface respond to the addition of simple salt? Previous simulations and mean-field calculations have suggested that the polymers will desorb. However, we will demonstrate that an increased adsorption also is possible, even for pure electrosorption, at low and intermediate levels of salt. As this is a correlation-driven effect, mean field approaches will fail to capture it. Using simulations, one will in general need to simulate large systems, and relatively long polymers. Also important is the presence of a proper bulk solution, with a finite and well-defined polyelectrolyte concentration. We have performed a theoretical study of polyelectrolyte adsorption. The work focuses on effects from ionic screening and polymer length. Specifically, the adsorption at a weakly charged colloidal particle, with a diameter of 200 nm, is monitored for various salt concentrations, in the presence of highly charged chains. These simulations are complemented by predictions from classical polymer density functional theory, utilizing a recently developed correlation-correction. The agreement with corresponding simulations is semi-quantitative, and since the calculations run many orders of magnitude faster than the simulations, longer and more realistic polymers could be studied with this approach. However, switching off the correlation-correction leads to a mean-field theory, which fails to even qualitatively reproduce the simulated response to screening. We are currently performing experimental studies to investigate some of the theoretical predictions, for instance that the adsorption depends rather strongly on chain length at intermediate levels of salt.
On the internal interfaces of polyelectrolyte multilayers

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The internal interfaces of polyelectrolyte multilayers are investigated with neutron reflectivity. The films are made from poly(diallyldimethylammonium) (PDADMA) and poly(styrenesulfonate) (PSS) or deuterated PSS-d. Each film consists of a protonated and a deuterated block, built from m protonated and n deuterated polycation/polyanion layer pairs. The position of the interface between the blocks is varied systematically. The internal roughness is smallest next to the film/air interface and increases with the number of layer pairs away from the film/air interface until a metastable state is reached. Both the equilibrium internal roughness and the interdiffusion constant increase with the salt concentration in the deposition solution, which is attributed to a reduction of ionic monomer/monomer pairs. On increase of the PDADMA molecular weight, the average thickness of a PDADMA/PSS layer pair increases. The internal equilibrium roughness and the interdiffusion constant grow, too. It is suggested that increased mobility of the chains is due to residual stresses, during polyelectrolyte multilayer formation the chains are not fully equilibrated. Post-preparation immersion in 1 M NaCl salt solutions does not change the internal roughness if the multilayer is prepared from low salt solution and high molecular weight PDADMA. However, if the film is prepared from 0.1 M NaCl solution, sufficiently long immersion in 1 M NaCl solution increases the internal roughness beyond the largest value observed directly after multilayer preparation. Complete intermixing of the constituent polyelectrolytes is observed only when the multilayer is prepared from 0.1 M NaCl and low molecular weight PDADMA.
Kinetics of the collapse transition of poly(2-oxazoline)s in aqueous solution

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At their cloud point, thermo-responsive polymers react strongly to a temperature stimulus, e.g. with a discontinuous change in conformation and solubility. Poly(2-oxazoline)s (POx) are very versatile and attractive polymers of this class, as their cloud point can be tuned in a wide range by including hydrophobic moieties. Along with their excellent biocompatibility, this makes them attractive in medical applications, e.g. as a drug delivery agents.

To characterize the nature of the phase transition for various amounts of hydrophobic moieties, we have investigated aqueous solutions of poly(2-isopropyl-2-oxazoline)₅₀ (PiPrOx₅₀), and gradient copolymers P[iPrOx₄₈NOx₂]grad and P[iPrOx₄₆NOx₄]grad which contain hydrophobic (2-nonyl-2-oxazoline) monomers (NOx). Using small angle neutron scattering (SANS), we have identified an intermediate regime just above the cloud point for the gradient copolymers [1]. In this intermediate regime, small precursor aggregates, formed already below the cloud point coexist with few large aggregates. The latter, dominate only at higher temperatures.

To investigate the stability of these small precursor aggregates, we performed temperature jumps from below the cloud point into the intermediate regime and followed the structural changes using time-resolved SANS. We could confirm that for both P[iPrOxNOx]grad samples, the precursor aggregates form within ~1 min and are indeed stable, and no large aggregates form during the time of observation (~45 min). The surface of the large aggregates evolves from a surface fractal over a smooth surface to a surface concentration gradient. Quenches to temperatures far above the cloud point revealed that both the kinetics of aggregate formation and the final conformation of the single chains strongly depend on the target temperature of the jump.

Surface induced conformational changes of adsorbed polyamidoamine dendrimers

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Due to their highly branched and well-defined globular structure, polyamidoamine (PAMAM) dendrimers have been considered as good candidates for achieving stimuli-responsive drug delivery systems and gene vectors [1-3]. Despite several experimental efforts, it was shown that PAMAM dendrimer swelling in aqueous solutions is minimal [4, 5].

In this contribution, by combining quartz crystal microbalance, reflectometry and atomic force microscopy, we show that PAMAM dendrimers adsorbed on silica experience substantial conformational changes when exposed to different electrolyte solutions. In particular, PAMAM dendrimers swell in solutions of high ionic strength and/or low pH. Conversely, molecular shrinking is observed when they are stimulated with solutions of low ionic strength and/or high pH. Furthermore, dendrimer swelling is found to be ion specific. Specifically, cations influence the swelling to a larger extent than anions and, among the cations studied, magnesium induces the biggest effect while lithium produces the smallest. Dendrimer swelling/shrinking behavior can be qualitative rationalized in term of electrostatic attraction between the adsorbed macromolecules and the substrate. At low salt level and/or high pH value, dendrimers are flattened by the electrostatic attraction between the positive amine groups and the silanol groups on the surface. At high salt level and/or low pH value, the attraction is weakened by screening or surface uncharging and the adsorbed molecules assume a more swollen configuration.

Self-assembly of β-Casein in dilute solution. Effect of electrostatic interactions

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β-casein belongs to the group of intrinsically disordered proteins and consists of 209 amino acid residues, including five phosphorylated serines. The molecular mass of the constituent chain is approx. 24 kD and the protein has an amphiphilic character. The main part of the hydrophobic residues is located in the C-terminus, while the N-terminus is rich in polar and negatively charged amino residues. Because it is unstructured, β-casein can be considered as a linear protein polymer and therefore the charge distribution, i.e., both sign and magnitude, varies with pH. The isoelectric point is around pH 4.6 where it precipitates out of solution.

β-casein self-associate in aqueous solution and form micelles. The driving force for the initial association of β-casein is hydrophobic interaction, whereas electrostatic interactions and steric hindrance oppose the assembly process. Hydrophobic and electrostatic interactions are the two main interactions in aqueous β-casein solutions, and to be able to evaluate their contribution to the association process, it is a requisite to define a system as pure as possible, i.e., keep the different molecular species to a minimum.

The fundamental question we have addressed is if it is possible to control the association by electrostatic interactions and if electrostatic phenomena as screening and correlations effects are possible to capture. The applied techniques are membrane osmometry, mass spectroscopy, and liquid chromatography to address the osmotic pressure/interactions, the stability of the proteins, and the size of the assemblies.
Modeling the complexation of dendrimer as ion penetrable sphere with DNA

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Poly (amidoamine) (PAMAM) dendrimers, being protonated under physiological conditions, have great potential as nonviral vectors for gene transfection. Many experimental and simulation studies have been performed to study the effect of dendrimer size, charge, and salt concentration on the structure and transfection efficiency of condensed DNA aggregates. In this study a theoretical model describing a linear polyelectrolyte (LPE) and ion-penetrable spheres has been developed and applied to investigate the interaction between linearized DNA and positively charged dendrimer of different generations. Throughout the study, first we emphasized on the effect of the medium's environments on the complexation of LPE chain with one dendrimer, namely the concentration of 1:1 salt solution, dielectric permittivity of the solvent, and pH conditions. Other factors have been investigated such as size and charge of the dendrimers, degree of polymerization of the LPE chain, and it's rigidity. Then we investigated the effect of the salt concentration on the interaction between linearized DNA plasmids (4331 bp) and positively charged dendrimers of generations 1, 2, 4, 6 and 8, previously studied experimentally. It is found that in the first case of complexation of LPE chain with one dendrimer, the wrapping degree of the chain around the dendrimer increases by increasing dendrimer's charge, Bjerum length, length of the LPE chain, and salt concentration. Also, charge inversion of dendrimer is obtained, and the value of the inverted charge increases by increasing the above mentioned parameters. While the complex shows more wrapping degrees, and less inverted charge as the pH of the solution decreases. In the case of complexation of DNA plasmids with dendrimer of different generations, the wrapping length of the LPE chain depends on dendrimer generation. With small generations, the optimal wrapping length of LPE chain around dendrimer increases by increasing the salt concentration, while , the complexation is insensitive to ionic strength with large generations.
Interaction mechanisms of indomethacin and biocompatible graft copolymer in aqueous solutions

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Nanoparticles from thermally-responsive biocompatible graft PNIPAM-g-PEO copolymers can incorporate indomethacin at room temperature and release the drug under controlled conditions at 37 °C. The aim of the present investigation is to clarify the mechanisms of inclusion and discharge processes. Extensive studies are performed on dynamic, equilibrium and rheological properties of the adsorption layers of aqueous polymer solutions at the air/solution interface. The investigated system contain also various ethanol quantity. Copolymers with different degrees of grafting are probed. The pool of experimental data comprise studies on bulk solution properties, determined by dynamic light scattering and TEM. Additional information is acquired from measurements of surface forces by the microinterferometric foam-film techniques, equipped with Scheludko-Exerowa cell.

The obtained results provide valuable input about indomethacin entrapment and release mechanisms by the PNIPAM-g-PEO nanoparticles. These data supply important knowledge about fine-tuning of drug uptake and release by core-shell nanoparticles. The investigations give grounds for certain generalizations about the optimization of the nanoparticles design and their more efficient functionalization in view of both the targeted and similar NSAID systems.

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Surface properties of a PNIPAAm-PAA copolymer remain constant as the film is heated past the LCST

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Force-distance curves acquired using atomic force spectroscopy were used to characterise thin hydrogel films prepared from a thermoresponsive copolymer of N-isopropylacrylamide and acrylic acid. The contact region of the curves provided information about the mechanical properties of the film, while the non-contact region provided information regarding relative changes in the surface charge density. As the films were heated in water from 26 to 42 °C, the mechanical response indicated that the films had collapsed. This is associated with heating the films past the lower critical solution temperature, or LCST. The bulk of the polymer becomes less soluble in water above the LCST, but the surface properties seem to remain constant, as indicated by the non-contact region of the force-distance curves. This may be due to the segregation of the acrylic acid-rich polymer segments near the gel surface, which is in agreement with reports for analogous systems. The characteristics of the aqueous interface are of interest when such hydrogel films are used in biosensor or other applications, since this will affect the interactions with the surrounding environment.
Spreading and evaporation of sessile droplets of liquid: complete and partial wetting, role of kinetic effects

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In the case of complete wetting, when equilibrium contact angle for liquid on a solid substrate is zero, the spreading/evaporation of a droplet can be subdivided into two stages: spreading (contact area increases) and evaporation (contact area decreases). A theoretical model of simultaneous spreading and evaporation of liquid droplets over hydrophobic surfaces in case of complete wetting is presented. It is validated against experimental data available in literature. In the case of partial wetting, due to contact angle hysteresis, the whole process can be subdivided into four stages: (0) spreading: quick process, when contact angle decreases until it reached its static advancing value; (i) evaporation with constant contact area, when contact angle decreases from static advancing down to static receding value; (ii) evaporation with constant (static receding) contact angle when the contact area decreases; (iii) evaporation with both contact angle and contact area decrease with time. A theoretical model is presented for stages (i) and (ii) and validated against experimental data on evaporation of pure liquids, aqueous surfactant solutions and nanofluids. A good agreement is observed in all those cases. Numerical simulations of evaporating of small size (micrometer and below) sessile aqueous droplets placed on a heat conductive substrate are carried out. It is shown that kinetic effects significantly influence the evaporation only if droplet sizes is less than one micrometre.

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Surface shear rheology of hydrophobin adsorption layers: laws of viscoelastic behavior with applications to foam stability

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The long-term stabilization of foams by proteins for food applications is related to the ability of proteins to form dense and mechanically strong adsorption layers that cover the bubbles in the foams. The hydrophobins represent a class of proteins that form adsorption layers of extraordinary high shear elasticity and mechanical strength, much higher than that of the common milk and egg proteins [1-3]. Our investigation of hydrophobin layers revealed that their rheological behavior obeys a compound rheological model, which represents a combination of the Maxwell and Herschel-Bulkley laws [3,4]. It is remarkable that the combined law is obeyed not only in the simplest regime of constant shear rate (angle ramp), but also in the regime of oscillatory shear strain. The surface shear elasticity and viscosity are determined as functions of the shear rate by processing the data for the storage and loss moduli, $G'$ and $G''$. At greater strain amplitudes, the spectrum of the stress contains not only the first Fourier mode, but also the third one. The method is extended to this non-linear regime, where the rheological parameters are determined by theoretical fit of the experimental Lissajous plot. The developed approach to the rheological characterization of interfacial layers allows optimization and control of the performance of mixed protein adsorption layers with applications in food foams [4].

The Drop and Bubble Micro Manipulator (DBMM) as a tool for the quantitative analysis of the interaction between drops and bubbles as basic elements in foams and emulsions

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The question about the stability of foams or emulsions is directly linked to the coalescence of two drops or two bubbles or to the characteristic behaviour of the corresponding liquid films. To investigate the direct interaction between drops or bubble there is the recently developed DBMM which provides a set of new quantitative and qualitative experiments to study the interaction between two drops or two bubbles or even between a single drop and a single bubble in a liquid medium [1] (see figure below). The DBMM provides a fine dosing system and capillaries with different diameters suitable for producing drops/bubbles of exact size and according to a given protocol. The available software can determine the radii of the liquid menisci at both sides and records the corresponding capillary pressure. A video with the standard sampling rate of 25fps can be recorded. With the installation of a high speed video camera (up to few thousand fps) it is possible to record and quantify also the characteristics of very fast processes like the coalescence [2]. In this work we study the stability of bubbles and drops in aqueous solutions of b-lactoglobulin (b-LG) against coalescence. A threshold protein concentration is determined as a function of adsorption time (lifetime of single drops or bubbles) for which the drops or bubbles, respectively, are stable. The pH and ionic strength influence the threshold concentration and also the addition of different types of surfactants is changing the stabilizing action of the protein.

References:


Structured conducting organic nanofilms via ionic self-assembly

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Conducting organic materials offer low-cost, large-area production for a range of flexible electronic and optoelectronic devices. Of great importance when developing such systems is to understand and control to the self-assembly behavior and structures formed when confined to thin films in order to maximize the conductive properties. Poly(aniline) has demonstrated great potential within this field but its application is limited due to its poor solubility in most common organic solvents and inherent disordered structural nature which limits the observed conductivity. In order to overcome these problems short oligomeric derivatives provide a route to more processible materials with tunable molecular architectures. Through coordination with acid-surfactants, the “ionic self-assembly” approach has been shown as a promising route to well-defined supramolecular structures in the bulk phase.

For application in electronic devices the active organic layer is often required to be around 100 nm in thickness; within this regime energetic contributions at the air-film and substrate-film interfaces can drastically influence the self-assembly and the resulting functionality from that of the bulk material. As such, the aim of this project is to develop a fundamental understanding of the structure-function relationship of oligo(aniline)-surfactant complexes when prepared in sub-µm thick films using surface-sensitive X-ray scattering techniques and conductive atomic force microscopy. Through exploiting ionic and other non-covalent interactions highly ordered nanofilms have been generated. Lamellar morphologies have been observed, which under certain conditions demonstrate a high degree of order and orientation with respect to the underlying substrate. The exact structural nature of these films has shown a dependence on the oligomer molecular architecture, film thickness and interfacial roughness.(1)

Polymer chain diffusion in interpenetrated microgels with upper critical solution temperature

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Poly (acrylic-acrylamide) interpenetrated microgels present continuous phase transition from collapsed to swollen state around 42 °C. This upper critical solution temperature (UCST) has prompted scientists to consider these systems as candidates for its use in biological applications such as smart drug delivery systems. The swelling of the polymer matrix would permit the release of the drug previously entrapped within the microgels.

In these systems the increment of the temperature can break inter-chain interactions, mainly hydrogen bonds, which reduce the elastic tension that stabilizes the microgel, favoring the polymer swelling. The microgel molecular dynamics at the UCST can be investigated using Incoherent Elastic (IENS) and Quasielastic Neutron Scattering (IQNS) [1-2]. From the analysis of the IQNS data we obtained that the diffusion coefficient of the polymer segments depends on the composition of the interpenetrated matrix. Thus, at room temperature, microgels with a polymer composition of 50% of each component present a diffusion coefficient $1 \times 10^{-12} \text{ m}^2/\text{s}$, while for the microgels formed by only one component the diffusion coefficient is $5 \times 10^{-10} \text{ m}^2/\text{s}$. This huge difference in the diffusion coefficient is conspicuously reduced when temperature increases, and we attribute this effect to the breaking of the inter-chain interaction. By means of FTIR-ATR analysis we have identified the groups that are involved in this phenomenon and we propose a model that relates the breaking of the polyacrylic-polyacrylamide interactions with the macroscopic behavior of the microgel.

References:
Hydration force in the presence of hydrotropes as the origin of stability of surfactant-free micelles?

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We consider poorly miscible but not immiscible liquids like water and octanol (or hexanol) in the presence of relatively low amounts of hydrotropes such as ethanol: a large single phase domain is observed in ternary systems. In this domain limited by a Winsor I regime, weak but observable light scattering is produced. The microstructure comes from two immiscible pseudo-phases along the nomenclature introduced by Tanford 1 in dynamic equilibrium, but also miscible at macroscopic level, i.e. the “super-aggregation” phenomena proposed by Ninham 2 twenty years ago, with some significant but no definite experimental “proof”.

In ternary systems, water-ethanol- fatty acid or alcohol, the presence of droplets of well-defined size with known composition is a hard fact. Attractive mechanisms leading to coalescence of surfactant and polymer-free nanodroplets are universal: Van der Waals forces and depletion via the associative dynamical networks in the water rich force are there.

What can make these droplets stable in the absence of charge or steric repulsion via an absorbed on the interface? The surface layer of the droplets is saturated with hydroxyl groups, as shown by SANS measurements. We propose here as the main stabilizing repulsive mechanism the hydration force. In the presence of solutes in the aqueous pseudo-phase, the hydration force is modified 3, but still generated by an enthalpy-entropy balance along the Marcelja-Radic mechanism 4.

In a dispersion of droplets, the hydration force favours small droplets and opposes to attraction mechanism. This phenomenon should be general and the quantification, with first general expression proposed herein, will allow new routes to formulation for surfactant-free solubilisation.

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The impact of ionic solvation energy, cavity energy and water structure on surface forces

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The structure of water in an interfacial region differs from bulk due to surface-induced water ordering. The difference is seen both in the local water density and in the water polarisability, particularly orientational polarisability as water dipoles are oriented towards the interface. The impact of this phenomenon in continuum models is to replace the dielectric constant of the medium with a position-dependent spatial dielectric function. The relationship that this kind of dielectric function bears with the electrostatic potential is known in the Poisson equation. But an additional effect of the spatially dependent dielectric function on ionic solvation energies (including a change in the Born energy of the ion) has not been widely recognised. The dielectric constant in an interfacial region has been reported to fall to values around 5, far from the bulk value of 78. The corresponding spatially-dependent ionic solvation energy therefore introduces a strongly repulsive ion-surface interaction which must be included as an additional "nonelectrostatic potential" in the Boltzmann factor, determining ion concentrations in a Poisson-Boltzmann model. Consequently a strongly repulsive surface force, a primary hydration force, is obtained with a range corresponding to the range of the surface-induced water ordering, usually the thickness of several water layers.

We explore and illustrate the effect of a spatially-dependent dielectric function and ionic solvation energy in a Poisson-Boltzmann model, applying spatial dependence suggested by computer simulation. We underline the importance of nonelectrostatic effects by including the cavity energy (the energy cost of removing water from the empty cavity of the ion) and the quantum mechanical dispersion component of the ionic solvation energy alongside the traditional electrostatic Born component. The dispersion component includes quadrupole and octupole contributions, which augment dipole polarisabilities.
Effects of very large hydrophobic ions on lipid assemblies

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The effects of small ions on colloidal systems are usually understood through double layer theory. Irrespectively of the models used to describe the double layer, the possible effects of the ions on the surface structure are usually ignored. In the case of soft-matter assemblies, based on surfactants or polymers, and very large "hydrophobic" ions there is a competition between surfactants and ions for interfacial sites, which may lead to dramatic structural modifications of the interfacial structure. In other words, in such systems, the boundary conditions at the surface matter more than the double layer that invariably forms. In the present work we have examined the effects of tetraphenylborate (TPB) anions on DPPC Langmuir monolayers and dodecyl phosphocholine (DPC) aqueous micelles. In both cases the strongly surface-active anions perturb greatly the structures of the assemblies. In monolayers we have found through a range of methods (IRRAS, GIXD, BAM), that the ions in fact push the lipids aside and reside on the surface, while affecting the properties of the lipid phases in unexpected ways (appearance of a unique untilted LC phase of DPPC). DPC micelles on the other hand are greatly perturbed. Mixed micelles of DPC and TPB appear to form, containing fewer lipid molecules as the TPB concentration increases. These two examples demonstrate a different aspect of ion-lipid interaction, which is probably closer to the interaction of lipids with proteins and other macromolecules.
Hydration force around cellulose crystalline nanorods and the water uptake of wood from atmosphere

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Wood consists of parallel, hollow, cylindrical cells. The so-called “wood material”, i.e. the materials the cell walls are made of, is a complex, highly anisotropic and hierarchically organized nanocomposite. It is characterized by stiff cellulose nanofibers parallel to each others embedded in a matrix of a much softer, less anisotropic, macromolecules (hemicelluloses and lignin). The matrix is hygroscopic and swells with increasing relative humidity providing a source of internal stress while the cellulose fibrils, winding with a spiral angle (the microfibril angle, MFA) around the central lumen, passively extend and reorient following the stress applied to them. Depending on the MFA, wood fibers exhibit a wide range behaviour and mechanical properties. This behaviour of wood fibres upon swelling is a major technological problem, but it essential to plants to actuate movements and to react to external forces. The swelling of the wood polymers is normally described as arising from the H-bond interactions of water molecules with the hydrophilic poly-saccharides and the sorption isotherms interpreted with modified BET, Dent, HH or GAB models. Although those expressions fit the sorption data excellently, they do not take into account neither the structure of the composite at the nanometric scale nor its hierarchical arrangement. Moreover, the fitting parameters do not directly provide thermodynamic values. In this work we explore the possibility that, at the molecular level, the force driving water sorption in wood is mainly the hydration force. Considering a hexagonal arrangement of the wood material’s building blocks at the two molecular levels (cellulose nanocrystals and single matrix polysaccharide chains) and taking into account the current wood structural models, we attempt an energy balance between hydration, Van der Waals and H-bond energies between the wood constituents. From the energy balance, the osmotic pressure is computed and the theoretical sorption isotherm calculated. The results of the model, compared with experimental sorption data and with small angle X-rays scattering data, seem to capture the main features of the wood swelling by water.
Determining the charge and aggregation number of ionic surfactant micelles from the stepwise thinning of foam films

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The oscillatory structural surface force causes stepwise thinning (stratification) of free liquid films containing spherical colloidal particles, including surfactant micelles. If the particles are charged, the height of the step is considerably greater than the micelle diameter. At a given micelle charge and ionic strength, it is possible to predict the step height. Here, we are addressing the inverse problem, viz. to determine the aggregation number, N, and charge, Z, of ionic surfactant micelles from the experimental curves of stratifying foam films. The effect of counterion binding is taken into account. In particular, N is determined from the height of the step [1], whereas Z is determined from the final film thickness that is established after the last stepwise transition. The method is applied to determine N and Z for micelles of ionic surfactants such as sodium dodecylsulfate (SDS), cetyltrimethylammonium bromide (CTAB), cetylpyridinium chloride (CPC), sodium laurethsulfates (SLES) and potassium myristate. The results are in good agreement with data for N and Z obtained by other methods. The method has the following advantages: (i) N and Z are determined simultaneously, from the same set of experimental data; (ii) N and Z are determined at each given surfactant concentration, i.e. they are not average values for a range of concentrations and (iii) N and Z can be determined even for turbid solutions, like those of carboxylates, where the micelles coexist with crystallites and the light-scattering methods are inapplicable, but the films stratify. The method is applicable to all surfactant concentrations at which the foam films stratify and the ionic micelles are spherical.

Caseins self-assembly

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The molecular association of individual caseins to form the milk casein micelles is still an unresolved phenomenon. Beta- and kappa-caseins are key players in maintaining the stability of these complex aggregates. Beta-casein (24 kDa, 209 amino acids), the most abundant casein, contains a large, hydrophobic C-terminal domain and a polar, negatively charged N-terminal domain at neutral pH. Kappa-casein (~19 kDa, 169 amino acids), as opposed to beta-casein, has a predominantly hydrophilic C-terminal block and a hydrophobic N-terminal end. Consequently, both proteins self-organize into detergent-like micelles. Kappa-casein further assembles into amyloid-like fibrils, structures that are associated with numerous degenerative diseases like Alzheimer, Parkinson and type-II diabetes. Although kappa-casein is not considered a disease-related protein, fibrillization of this protein, and inhibition of this process may assist in studying the fibril formation phenomena in these diseases. In the last few years we studied micellization and thermodynamic characteristics of these two proteins as a function of temperature, pH, and ionic strength [1-5], and interactions between the two, by numerous techniques including isothermal titration calorimetry (ITC) and cryogenic-transmission electron microscopy (cryo-TEM). Here we focus on our recent findings with kappa-casein that presents several parallel pathways of self-association at low concentrations, and mixtures of kappa- and beta-caseins. The combination of calorimetry and cryo-TEM clearly demonstrate that beta-casein can decrease and even prevent kappa-casein fibrillization.

References:

Synthesis of magnetic nanoparticles by the oil-in-water microemulsion reaction method and their incorporation in nanocomposites

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The objective is to investigate the potential of the O/W microemulsion reaction method for the synthesis of magnetic nanoparticles and explore their incorporation into magnetic nanocomposites. The method implies the use of organometallic precursors, dissolved in nanometer-scale oil droplets (stabilized by surfactant), and dispersed in the continuous aqueous phase; the precipitating agents are added directly to the microemulsion as aqueous solutions without compromising microemulsion stability. It was reported for the first time as proof of concept in 2009[1]; it was demonstrated that small nanoparticles, good crystallinity and high specific surface area (SSA) could be obtained. In this contribution, magnetic nanoparticles (MNPs, Fe2O3, as well as mixed ferrites Fe2Mn0.5Zn0.5O4 and Fe2Ni0.5Co0.5O4, have been prepared by this method, using a nonionic microemulsion system (hexane/Synperonic 91/6/water). Small nanoparticles (from 3 to 10 nm depending on the reaction conditions), with high specific surface area (up to 300 m²/g) were obtained. High resolution transmission electron microscopy and X-Ray Diffraction studies demonstrates that maghemite structure (for Fe2O3) and spinel structure (for mixed ferrites) were obtained under mild conditions. The magnetic characterization was consistent with superparamagnetic behavior in most cases. Furthermore, the synthesis method was adapted for in-situ functionalization of the nanoparticles with fatty acids; preliminary results show that this functionalization is promising for dispersion of the particles in organic solvents, which is useful for nanocomposites preparation. On the other hand, bare magnetic nanoparticles could be coated with biopolymers: a protein-magnetic nanoparticle composite was prepared, which was very active for the purification of the protein, due to the high SSA of the material. The results demonstrate that MNPs synthesized by the o/w microemulsion reaction method have a great potential for their incorporation into nano-biosystems, either through in-situ functionalization or by direct incorporation. [1] M. Sanchez-Dominguez, M. Boutonnet and C. Solans, J Nanopart Res, 11 (2009), 1823
WEDNESDAY
Generic and specific roles of soft interlayer in modulating interactions at biological and material interfaces

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Numerous interfacial contacts in biological systems are mediated via soft interlayers containing various carbohydrates, such as extracellular matrix and glycocalyx. They are not only acting as a “protector” or “filler” to retain the structural integrity but also are recognized by specific proteins and complementary carbohydrates. Considering the complex interplays of generic and specific interactions, quantitative understanding of the physics and chemistry of soft interfaces is an interdisciplinary scientific challenge.

In the main part of my talk, I will introduce some strategies to design well defined model systems by functionalization of solid/liquid and liquid/air interfaces. Here, the combination of various physical techniques, such as specular and off-specular X-ray and neutron scattering and grazing incidence X-ray fluorescence, enables one to unravel the roles of "soft" biopolymers (carbohydrates) in fine-adjustment of interfacial interactions.

In the next part, I select some examples to show how soft interfaces, new physical techniques, and statistical analysis can be used to understand some principles in human diseases and development. Finally, I would like to point out some potential applications of soft functional interfaces, since they can bridge soft, biological matter and hard materials.
New strategies to improve the self-assembly properties of DNA-coated colloids

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DNA-coated colloids have great potential for the design of complex self-assembling materials, but so far the achievements have been modest. We present experiments and computer simulations on novel DNA-functionalized systems that have improved self-assembly properties. We show that the multi-bond character of the DNA-mediated interactions plays an important role and we then apply this knowledge in two different ways. In one system, we provide the DNA sticky ends on the surface of micrometer-sized colloids with lateral mobility, whereas another system exploits the competition between different types of sticky ends to widen the normally very narrow temperature window for equilibrium self-assembly.
Mimicking protein channels with colloids in microfluidic channels

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Transport through quasi-one-dimensional (1D) structures as pores and ion channels is ubiquitous in biological and artificial systems.[1,2] In fact the diffusive behavior of Brownian particles depends on the interaction between them and the environment around them: when the space available for the particles is restricted entropic barriers arise that have strong impact on the particle diffusive behaviour.[3] The corresponding Brownian dynamics of particles confined in narrow channels with varying cross-section have been analytically investigated and confirmed by numerical simulations.[4] On the other hand it has been demonstrated that particle free diffusion may be greatly enhanced if both the free space or a 1D system is permeated by ad hoc potential energy landscapes.[5,6]

We propose a novel model system based on sub-micrometer colloids, microfluidics and holographic optical tweezers to investigate the influence of both quasi 1D confinement and potential energy landscapes on the particle diffusive behaviour. Specifically we introduce a new approach for the fabrication of microfluidic chips with arrays of sub-micrometer channels (with minimum feature size down to 600 nm) of tunable cross section and aspect ratio[7] and we analyse the trajectories of single particles translocating and interacting in such channels. By coupling a custom-made holographic optical tweezers set-up we manipulate several particles simultaneously and independently inside each channel and furthermore we are able to tailor arrays of optical line traps along the channel length thus introducing a variety of potential energy landscapes.
Nano-vesicles with tunable architecture formed by biocompatible star-shaped block-copolymer

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An amphiphilic star-shaped block copolymer, based on well known biocompatible components was synthesized using branched poly(ε-caprolactone) as hydrophobic core and branched poly(ethylenglycol) as hydrophilic corona. The composition of this molecule based on two well differentiated blocks conferred anisotropy to the whole system that acted as driving force for self-assembling the molecules in aqueous media. Depending on the concentration of the polymer it was possible to obtain different architectures. The TEM micrographs permitted to follow the evolution of the system from single vesicles toward necklace entanglements. In this contribution, we propose and discuss a mechanism to explain the evolution of the morphology of the assembled nanoparticles as a function of the concentration of the block copolymer.

The proposed system presented good solubilizing properties that were used to disperse in water, molecules with low solubility, such as chlorine-carbazoles. Furthermore, these molecules might serve as model to investigate the properties of the system as drug nano-carrier [1-4].

References:
Single-molecule observations of protein-protein associations at the solid-liquid interface

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Protein adsorption at interfaces is a widely studied and very complex phenomenon. Single-molecule techniques uniquely allow us to separate protein surface dynamics (e.g. protein adsorption from desorption vs. net adsorption) and identify and characterize multiple protein populations and dynamic modes (e.g. oligomeric states, multiple diffusive modes vs. average protein behavior). Recent work using single-molecule total internal reflection fluorescence microscopy (TIRFM) at low protein concentrations, such that protein-protein interactions are insignificant, has shown protein desorption and diffusion to be indeed highly heterogeneous. For example, multiple residence time populations were found to correspond to protein oligomeric state where 99.9% of individual protein monomers reside on the surface for <1s while larger oligomers (trimers and tetramers) reside for up to hundreds of seconds. These findings suggest that protein-protein interactions, rather than individual protein-surface interactions, greatly influence protein layer formations and protein surface dynamics observed at high concentrations. By combining TIRFM and intermolecular fluorescence resonance energy transfer, the dynamics of protein-protein associations can be directly observed and quantified at the solid-liquid interface. Using this technique, protein-protein association times are found to be heterogeneous and fast with the majority (80-90%) of protein-protein associations lasting for <1s. Similarly, the distribution of RET efficiencies (indicating the extent of protein-protein contact) is heterogeneous. Ultimately, by separating different association populations and dynamics, we can gain a greater fundamental understanding of protein-protein interactions that lead to protein aggregation and protein layer formation.
Disentangling critical slowing down and dynamical arrest in lens protein solutions

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The occurrence of an arrest transition in concentrated colloidal suspensions and its dependence upon the interaction potential is a currently intensively discussed and very important field of soft matter research. However, while we do understand dynamical arrest and glass/gel formation in one-component colloidal suspensions at high densities quite well by now, very limited information is available for the interplay of dynamical arrest and phase separation/critical phenomena close to the critical point for short range attractive particles. Here we report on an experimental investigation of the interplay between critical phenomena and dynamical arrest in concentrated solutions of the lens protein γB-crystallin. This globular protein has been identified as one that closely mimics the phase behaviour of colloids with short-range attractive potentials and exhibits a metastable liquid-liquid phase separation. We use a combination of 3D static and dynamic light scattering, small-angle X-ray scattering, neutron spin echo measurements and pulsed-gradient spin echo NMR to study the structural properties as well as collective and self diffusion of the protein at all relevant length and time scales along different isochores (i.e. performing temperature variations) at concentrations close to and above the critical concentration c_0. We indeed find very different divergence of the characteristic decay time of the intermediate scattering function along the critical isochore and along isochores at higher concentrations, with a clear signature of critical slowing down along the former, and dynamical arrest caused by the presence of an attractive glass transition along the latter. We discuss our results in view of our current understanding of the phase behavior of particles with short range attractions, and make links to the role that γB-crystallin plays in cataract formation and presbyopia, two common lens diseases that are ultimately linked to the attractive interactions of this protein.
Effects of additives on the phase behaviour of protein solutions

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Understanding protein phase behaviour and in particular protein crystallization is important for biology and biotechnology, e.g., in the context of diffraction studies or the occurrence of proteins in tissues. We studied the phase behaviour of lysozyme, in particular in the presence of additives, such as glycerol, DMSO or guanidine hydrochloride [1]. Crystallization as well as liquid-liquid phase separation were investigated using direct observation, optical microscopy and static light scattering for different protein concentrations and temperatures. Upon addition of glycerol and DMSO, the crystallization boundary and the binodal shift to lower temperatures, while the gap between them increases. Based on second virial coefficients, both additives are found to render the protein interaction potential more repulsive (in the range of additive concentration studied). Furthermore, if the second virial coefficient is used instead of temperature, the binodal phase boundaries fall on top of each other.

Peptides and proteins in polyelectrolyte microgels

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Microgels offer opportunities as carriers for protein and peptide drugs, since they offer protection from chemical and biological degradation, since release can be sustained, controlled, and triggered, and since bioavailability can be improved. For microgels to reach their full potential in this context, however, peptide/protein incorporation into, and distribution within, microgels, as well as its effects on the microgel network, must be controlled. In an effort to generate new knowledge on these complex processes, studies were performed on various aspects of peptide/protein incorporation into oppositely charged microgels. These studies involved a range of methodologies, but with particular emphasis placed on micromanipulator-assisted microscopy and confocal microscopy. Using these and other techniques, aspects such as peptide length in relation to microgel mesh size, peptide/microgel charge contrast, pH and ionic strength, as well as the chemical nature of similarly charged peptides, were investigated. Also effects of the detailed peptide structure were investigated, e.g., for antimicrobial peptides, as were spherical model proteins. The investigations demonstrate that incorporation of peptides/proteins into oppositely charged microgels, as well as their subsequent release, are complex processes, where shell formation, transport limitations, and non-equilibrium effects significantly influence the performance of such microgels as carriers of peptide and protein drugs.
Nano-wear studies of adsorbed proteinaceous layers

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Proteinaceous layers can confer diverse properties to surfaces such as molecular binding, biocompatibility, or simply act as protective/lubricant barriers. Here we present a novel method [1,2] to study these systems based on their response to a variable-load scratch performed with an Atomic Force Microscope (AFM) operated in the Friction Force Spectroscopy (FFS) mode. Specifically, this method provides the load-dependence of both the topography of the layer at the nm range, and the friction between tip and the layer at the nN range. This allows characterizing the resistance of the layers to external sources of mechanical damage through the forces needed to break and to remove them. Moreover, we also show how the method can be used to study the lateral diffusion of proteins on surfaces, and even to gain insight into the structure of the layers. Results are presented for model layers of soft globular proteins (those formed by BSA molecules), for model layers of amphiphilic disordered proteins (those formed by β-casein molecules), and for the more complex salivary films [3,4].

References:
Protein motion in bicontinuous microemulsions as seen by quasielastic neutron scattering and by FCS

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The enzyme diisopropyl fluorophosphatase (DFPase) from the squid Loligo vulgaris is of relevance due to its ability to catalyze the hydrolysis of highly toxic organophosphates. In the present work, the enzyme structure in solution (native state) is studied by different scattering methods. The results are compared to hydrodynamic model calculations. Bicontinuous microemulsions (bME) made of sugar surfactants are discussed as reaction media for the DFPase. The bME remains stable in the presence of the enzyme, which is revealed by scattering experiments. It is shown that the DFPase still has a high activity in the bME [1]. The collective and the local motion of the surfactant interface in the bME is studied by means of neutron spin-echo. Moreover, it is tried to study the enzyme motion decoupled from the microemulsion by contrast variation. In addition to the neutron scattering experiments also experiments on the diffusion of green fluorescent protein + (GFP+) in a bicontinuous microemulsion will be presented using fluorescence correlation spectroscopy (FCS).

The diffusion coefficient GFP+ decreases with decreasing size of the water domains of the microemulsion.

Natural bactericidal surfaces: mechanical rupture of *Pseudomonas aeruginosa* cells by cicada wings

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Numerous insects have evolved to possess superhydrophobic surfaces, which are thought to limit bacterial contamination through a self-cleaning action. While investigating the adhesion of *Pseudomonas aeruginosa* on the wings of a species of cicada (*Psaltoda claripennis*), we discovered that the wing surfaces were actually lethal to the cells. Electron microscopy showed nanopillars on the surface penetrating the cells, which was confirmed to be lethal through viability experiments. The effect occurred very quickly, with individual bacterial cells killed within approximately three minutes. The bactericidal effect is primarily based on the physical surface structure; significant alteration of chemistry through gold coating of the wing failed to inhibit cell death. The cicada wings were able to maintain a clean surface by continuous cleansing through bactericidal action, rather than repelling bacterial cells. Surfaces that inhibit bacterial contamination via their physical structure represent a novel area of research for the development of antibacterial surfaces.
Comparison of two methods detecting lysozyme adsorption to oil-water interface in the presence of surfactants

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The purpose was to compare the formation of the adsorbed lysozyme layer at the oil-water interface with two different methods and to scrutinize the possibilities of avoiding film-formation by addition of model surfactants. Interfacial tension measurements were carried out using pendant drop (KRÜSS, Germany). An aqueous droplet of 70 µL was formed with a needle (diameter 1.83 mm) in a glass cuvette containing the oil-phase. Film formation was evaluated by withdrawal of the aqueous phase after 10 minutes emersion in the oil phase. Rheological properties were measured by use of a TA AR-G2 rheometer equipped with a double wall ring (DWR) geometry. The system consists of a ring and a Delrin® trough with a circular channel (interfacial areal=1882.6 mm²). Oscillatory shear measurements were conducted at constant frequency of 0.1 Hz, temperature of 25°C and the strain was set to 1%.

The adsorption of lysozyme to the oil-water interface results in the formation of a flexible protein film. This formation can be prevented by addition of surfactants, in a manner that is dependent on the concentration and the type of surfactant. According to the rheological method the more hydrophilic surfactants are more effective in hindering lysozyme adsorption to oil-water interfaces whereas the hydrophobic surfactants seem to be more effective according to the interfacial tension measurements. According to the rheological method the larger surfactants are more persistent in preventing film formation whereas the smaller eventually give place for the lysozyme on the interface.

The two methods can be used to detect the interfacial adsorption of lysozyme and can be used to evaluate the performance of model surfactants in hindering film formation. This will aid in processing of any delivery systems for proteins where the protein will be introduced to oil-water interfaces that could affect the stability of the protein.
Design of the self-repairable materials using colloidal particles

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We suggest an approach for the design of materials with self-repairable switchable and ultrahydrophobic properties. The approach is based on use of colloidal particles located on a surface of hydrophobic oil which melting point slightly higher than the ambient temperature. We demonstrate that colloidal particles, which freely float on the surface of fusible waxy solids, can be used for the design of switchable surfaces. Due to surface forces, the colloidal particles segregate at the wax surface and change its wetting properties. Since the degree of particle immersion in the wax depends on the environment (hydrophilic or hydrophobic), the roughness, as well as the hydrophilicity/hydrophobicity of composited particle-wax surface, can be switched. Moreover, the obtained morphology can be “frozen” by cooling down below oil melting point [1]. Notably, since the colloidal particles are homogenously distributed in the wax bulk, removal of the topmost particle-wax layer does not result in disappear of switchable properties. The particles segregate again at newly formed surface and switchable properties are recovered. The similar approach is used for the design of materials with self-repairable ultrahydrophobic properties. The materials are based on perfluorinated decane with incorporated colloidal particles. Due to the highly pronounced tendency of this perfluorinated decane to crystallize, the formation of blends with rough fractal surfaces was observed. In order to prove their self-repairing ability, we mechanically damaged them by scratching, which removed most of the particles from the surface. Melting of the damaged blend resulted in reorganization of the particles at the wax-air interface, restoring the initial structure and thus the ultrahydrophobic behaviour [2].


Synergetic effect on multilayer formation of fluorocarbon compounds at the hexane/water interface studied by interfacial tensiometry and synchrotron X-ray reflection

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The synergetic effect of fluorocarbon compounds on novel multilayer formation at the hexane/water interface was investigated from the viewpoint of intermolecular interaction and mixing of molecules in the adsorbed film. The two kinds of mixed systems were employed; 1H,1H,2H,2H-perfluorododecanol (FC12OH) - 1H-perfluorodecane (HFC10) (system A) and 1-icosanol (C20OH) - HFC10 (system B). The interfacial tension $\gamma$ of the hexane solution against water was measured as a function of total concentration $m$ and the composition of HFC10 in the mixture $X_2$ at 298.15K under atmospheric pressure. XR was performed at BL37XU in SPring-8 as a function of scattering vector $Q_z$. In both systems, the $\gamma$ vs. $m$ curves except for pure HFC10 system have a break at low concentration due to the gaseous - condensed monolayer (system A) and the expanded - condensed monolayer transition (system B). Remarkable difference was that the curves of system A shows another break at high concentration close to the solubility limit. The total interfacial density above this break point was around 7 ~ 11 $\mu$mol/m$^2$, suggesting the spontaneous molecular piling to form multilayer. The phase diagrams of adsorption in the condensed monolayer indicated that the film composition of HFC10 is negative in the system B but definitely positive in the system A. This clearly shows that HFC10 molecules are miscible with FC12OH but immiscible with C20OH in the condensed monolayer. Thus, it is likely that the mixing of HFC10 with FC12OH in the condensed monolayer induces multilayer formation. The Fresnel divided X-ray reflectivity $R/R_F$ vs. $Q_z$ plot in the condensed monolayer of system A was fitted by one-slab model with uniform electron density and thickness. The electron density profile was almost same as that of pure FC12OH system. The plot in the multilayer, on the other hand, was fitted well by two-slab model with different electron densities and thicknesses. The electron density profile showed that the multilayer consists of two layers; one of which has slightly higher electron density than bulk hexane phase and piles on the lower layer with almost same electron density as condensed FC12OH monolayer.
Measuring the boundary slip of water on smooth surfaces by total internal reflection fluorescence cross-correlation spectroscopy

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Liquid flow in a confined geometry can be accurately described only if its behaviour at the interface between the fluid and the solid is thoroughly understood. While for many years the so called no-slip boundary conditions (velocity equal to zero at the interface) were applied to describe macroscopic flows, recently it has been recognized that these conditions may not always apply, especially when channels with micro- and nano-sizes are considered. Nevertheless, the existence and the extend of the boundary slip and its dependence on surface properties and the shear rate are still very much debated, partially due to the lack of highly sensitive techniques that can measure very small slips [1].

Recently, we developed a new method for direct studies of flows in the close proximity of a solid surface based on measuring the velocity of small fluorescent tracers flowing with the liquid by total internal reflection fluorescence cross-correlation spectroscopy [2]. Furthermore we employed Brownian-Dynamics techniques to simulate the tracers’ motion through the observation volumes and generate a “numerical” cross-correlation curves that can be consequently used to fit the corresponding experimental data and extract information on the boundary slip length with accuracy better than 10 nm [3]. Here we apply this method to measure the boundary slip of water flowing at smooth hydrophilic and hydrophobic surfaces.

The use of thin, tailored Layer-by-Layer (LbL) films to improve adhesion

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The use of LbL treatment of cellulose fibres with oppositely charged polyelectrolytes to improve the mechanical properties of fibrous networks has been known for some time (1). It has been shown that both the amount of adsorbed polyelectrolytes and the ordering of the polyelectrolytes in the adsorbed layers have a profound influence on how much the mechanical properties of the networks can be improved. However, so far there is a limited knowledge about how the selection of components of the LbLs can be used to tailor the properties, i.e. thickness, Youngs modulus, softening behavior etc, of the formed layers and how this in turn will affect the adhesion between treated surfaces. In the present work a careful selection of polyelectrolytes and nanoparticles, including Nano Fibrillated Cellulose (NFC), was made to prepare nanometer thin, tailored LbLs on model surfaces and to determine the wet/dry adhesion between these surfaces. The properties of the adsorbed layers were carefully characterized using SPAR (Stagnation Point Adsorption Reflectometry) and DPI (Dual Polarization Interferometry) to determine the adsorbed amount and the water content of the adsorbed layers. SIEBIMM (Strain Induced Elastic Buckling Instability for Mechanical Measurements) was then used to determine the elastic properties of the dry films. The wet adhesion between LbL-treated surfaces was determined using colloidal probe AFM and the dry adhesion was evaluated using a Micro Adhesion Measurement Apparatus (MAMA) based on the Johnson Kendall and Roberts (JKR) theory. The results show that a migration of the polyelectrolytes across the interface between the surfaces are needed to create a high wet adhesion and in order to maintain this in the dry state it is necessary to create a polymer alloy, with optimized mechanical properties, of the materials in the LbL structure.

References:

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Pickering emulsions stabilized by soft and thermoresponsive microgels

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Pickering emulsions are surfactant-free metastable dispersions of two non-miscible fluids stabilized by colloidal particles. Such emulsions have been extensively studied over the last two decades using mineral or organic solid particles. More recently, stimuli-responsive microgels have received much attention as stabilizers because of their ability to impart responsiveness to the resulting emulsions. These particles are made of weakly cross-linked polymer swollen by a solvent. Because of their softness, the interfacial behavior of microgels is fundamentally different from that of solid particles. The aim of the present study is to provide insights into the origin of emulsion stabilization by this class of soft colloids.

We used thermoresponsive microgels made of p(N-isopropylacrylamide) as model particles. They were swollen by water below the so-called volume phase transition temperature (VPTT) and shrank above it, due to a change in the polymer/solvent affinity. We varied continuously their softness, either in situ by changing the temperature or during the synthesis by varying the crosslinker concentration, and showed that it is a key parameter in controlling emulsion stability [1]. The microgels deformed at interface and formed 2D connected networks that hindered coalescence and provided stability of emulsions. Whenever the microgel deformability was lost, either by increasing the cross-linking density or by warming the samples above the VPTT, their stabilization efficiency was considerably reduced and could result in the emulsion phase separation.

Emulsion end-use properties (flocculation and stability against coalescence) could be controlled by adapting the emulsification conditions. Depending on whether the microgels adsorbed in their expanded or collapsed state, the interfacial monolayer could be trapped in a very dense and rigid configuration, providing resistance to bridging flocculation and to coalescence [2].


Ionic liquid nanotribology: stiction suppression and surface induced shear thinning

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The interfacial ordering and capacity for self assembly in Ionic liquids is of enormous current interest. The friction and adhesion between pairs of materials (silica, alumina and polytetrafluoroethylene) have been studied and interpreted in terms of the long-ranged interactions present. In ambient laboratory air, the interactions are dominated by van der Waals attraction and strong adhesion leading to significant frictional forces. In the presence of the ionic liquid (IL) ethylammonium nitrate (EAN) the van der Waals interaction is suppressed and the attractive/adhesive interactions which lead to “stiction” are removed, resulting in an at least a 10-fold reduction in the friction force at large applied loads. The friction coefficient for each system was determined; coefficients obtained in air were significantly larger than those obtained in the presence of EAN (which ranged between 0.1-0.25), and variation in the friction coefficients between systems was correlated with changes in surface roughness. As the viscosity of ILs can be relatively high, which has implications for the lubricating properties, the hydrodynamic forces between the surfaces have therefore also been studied. The linear increase in repulsive force with speed, expected from hydrodynamic interactions, is clearly observed, and these forces further inhibit the potential for stiction. Remarkably, the viscosity extracted from the data is dramatically reduced compared to the bulk value, indicative of a surface ordering effect which significantly reduces viscous losses.
Designing composite microgels with varying shapes and functionalities: on the road towards supracolloidal self-assembly

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Designing functionalized colloids with varying shapes and functionalities has attracted a growing interest, particularly as it opens the route for new self-assembly routes. Composite core-shell microgels are an ideal platform as they combine the intrinsic properties of the core and the shell. In our research we use a thermosensitive poly(N-isopropylacrylamide) or poly(N-isopropylmethacrylamide) network as the shell. In this way, not only the size and therefore the effective volume fraction can be efficiently controlled but also the nature of the interactions from long-ranged repulsive to short-ranged attractive. When using mixtures of oppositely charged particles, this provides us with exquisite control of the self-assembly processes and to cover a variety of structures such as clusters, gels and crystals.

In addition, when a polystyrene core is employed, novel model systems with tailored shape could be obtained by post-processing the original spherical particles into ellipsoidal, faceted or hollow hemispheroidal particles. Thus, nanoengineering core-shell allows us to combine non-spherical complex shapes with control on interparticle interactions through the thermosensitive shell.

Furthermore, the polystyrene core can be replaced by a maghemite spindle with paramagnetic properties to design composite microgels with directed interactions that can be controlled using an external magnetic field.

These complex and tunable composite particles open up new and exciting applications from improved ferrofluids to nanoreactors and bioinspired self-assembly where the particles act as colloidal molecules.
Tuning suspension rheology using capillary forces

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When a small amount (less than 1%) of a second immiscible liquid is added to the continuous phase of a suspension, the rheological properties of the mixture are dramatically altered from a fluid-like to a gel-like state. The yield stress and viscosity increase by several orders of magnitude as the volume of the second fluid increases. This transition is attributed to the capillary forces of the two fluids on the solid particles, and in an analogy to wet granular materials, two distinct states are defined: the "pendular state" where the secondary fluid preferentially wets the particles; and the "capillary state" where the secondary fluid wets the particles less well than the primary fluid [1].

Since the capillary forces control the formation of the particle network, the strength of this network and hence the flow behavior of the suspensions can be widely tuned through the variation of the capillary force and the number of capillary bridges. Accordingly, the yield stress of the capillary suspensions scales with the inverse radius and the surface tension of the added liquid, which e.g. generally decreases with increasing temperature. The strength of these suspensions can be further adjusted through the addition of surfactants. Several nonionic surfactants with a range of HLB values, when added to the bulk fluid, all show that the yield stress decreases with increasing surfactant concentration much more than would be predicted through the reduction of the interfacial tension alone. Microscopic images reveal that with the addition of nonionic surfactants, the number of capillary bridges is reduced, decreasing the yield stress by several orders of magnitude. The addition of an ionic surfactant added to the disperse, aqueous phase is also able to decrease the yield stress by one order of magnitude.

Dynamics of colloids in random potential energy landscapes – effects of dimensionality and particle concentration

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Potential energy landscapes provide a theoretical framework which has proven to be very powerful, e.g., for understanding the phenomena of protein folding or the dynamics of glass-forming systems. We experimentally studied the dynamics of individual as well as interacting colloidal particles in random potential energy landscapes [1, 2], which were optically generated by laser light fields. The particle dynamics were extracted using video microscopy. They were studied over many orders of magnitude in time and characterised using, e.g., the mean squared displacement, the van Hove function and the non-Gaussian parameter. In 1 D and 2 D, the dynamics are initially diffusive and then, at intermediate times, show an extended sub-diffusive regime before diffusive motion is recovered at long times. Local maxima can easily be circumvented in 2 D, and thus the long-time diffusive regime is reached at shorter times.


Non intrusive measurement of emulsion viscoelastic properties by passive microrheology

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This work presents a new technique of the passive microrheology for the study of the microstructure properties of soft materials like emulsions. Our technology uses Multi Speckle DWS (MS-DWS) set-up in backscattering with a video camera. It allows to measure the mean displacement of the microstructure particles in a spatial range between 0.1 and 100 nm and a time scale between $10^{-3}$ and $10^5$ seconds. Different parameters can be measured or obtained directly from the Mean Square Displacement (MSD) curve like a fluidity index, an Elasticity Index, a Macroscopic Viscosity Index, a relaxation time, a MSD slope. This technique allows to monitor the evolution of the microstructure, the restructuration after shearing, the variation of the viscoelastic properties versus temperature, pH, the physical stability of emulsion or suspension... This work focuses on the measurement of viscoelastic properties evolution of emulsions to follow their stability, the evolution of their properties depending on the volume fraction and thickener concentration. The results will show the advantages of using a non intrusive method to detect nascent destabilisation of the microstructure before rheology or visual method.
Hydrodynamic Rayleigh-Taylor-like instabilities in sedimenting colloidal mixtures

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Experimental studies have shown that the sedimentation of an initially inhomogeneous distribution of colloids can result in the formation of Rayleigh-Taylor-like hydrodynamic instabilities [1]. By using a coarse-grained hybrid molecular dynamics and stochastic rotation dynamics simulation technique we are able to study the sedimentation of binary colloidal mixtures confined to a slit at the particle level [2]. This technique allows us to take into account both the Brownian motion and the hydrodynamic interactions between colloidal particles in suspensions, both of which are important for describing experimental setups correctly. By varying both the properties of the species forming the binary mixture and its composition we examine how specific choices of mixture parameters influence the system behavior.

We show that the structural properties of the swirls that form as a consequence of the instability depend greatly on the relative magnitudes of the particle Peclet numbers, and much less on the composition of the mixture. Calculating the spatial colloid velocity correlation functions allows us to follow the time evolution of the instability, and the time dependence of the characteristic correlation length. As the instability develops the correlation length increases and its magnitude indicates the size of the dense colloidal droplets that form. We also calculate the growth rates of the unstable modes both directly from our simulation data, and also using a theoretical approach, and we show that the instability behavior depends only on the macroscopic properties of the complex fluid and not on the specific distribution of colloids within the mixture.

Lyotropic smectic B phase formed in suspensions of charged colloidal platelets

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We present the first observation of a smectic B (SmB) phase in a system of charged colloidal gibbsite platelets suspended in dimethyl sulfoxide (DMSO).[1] The use of DMSO, a polar aprotic solvent, leads to a long range of the electrostatic Coulomb repulsion between platelets. We believe this to be responsible for the formation of the layered liquid crystalline phase consisting of hexagonally ordered particles, that is, the SmB phase. We present high resolution X-ray scattering experiments for different concentrations in the SmB phase additionally supporting the phase characterization. The identification of the formation of a SmB phase that is not only governed by the shape and concentration of the colloidal particle but also by their anisotropic interactions is of fundamental interest. Such interplay between parameters in the formation of liquid crystalline phases in colloidal system gives tools to manipulate these systems. In addition, this finding challenges theory and simulation to predict this phase.

Controlled release from functional colloidosomes

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Encapsulation and controlled release of materials from microcapsules are of great interest in biomedicine, food and pharmaceutical science. Capsules made from closely packed colloids in the shell, usually referred to as colloidosomes, are very attractive for these applications. Compared to conventional approaches, colloidosomes fabricated using double emulsion templates made in microfluidic devices allow for highly flexible, efficient loading and capsule formation in a one step process. In this work, we show that the range of materials used to form the shell can be significantly widened by appropriately adjusting the surface chemistry of the colloidal particles and of the oil-water interfaces. We investigate more closely the role of the surface active species needed to stabilize both double emulsion interfaces. Also, we demonstrate colloidosome systems that allow for pH-controlled release of molecules. To produce colloidosomes, water-in-oil-in-water double emulsions are formed in a glass microfluidic device using a particle-loaded toluene suspension as intermediate oil phase. The surface chemistry of the particles has been adjusted using surfactant or silane treatment where needed. Since toluene is slightly soluble in water, it can be completely evaporated from the intermediate oil layer, leading to well-controlled colloidosomes. Lowering the interfacial tension and proper steric stabilisation of the double interfaces has been identified as crucial to obtain stable colloidosomes. We found that polyvinyl alcohol is a particularly good surfactant for this system due to synergeric interactions with the particles from the oil suspension. We were able to produce mechanically stable, photocatalytic, potentially piezoelectric, magnetic and high surface area colloidosomes. By encapsulating a second type of cargo-loaded nanoparticles inside the colloidosomes, controlled multiple release can be achieved without compromising the integrity of the colloidosome shell. A change in the pH of the external media inverts the charge of the encapsulated particles and initiates the release of the surface-adsorbed cargo.
Smart core-shell Janus particles with chemical ‘patchiness’ and controllable Janus balance

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The concept of design smart functional materials with controllable interfacial and surface properties as well as understanding interactions between single building blocks is very important for development of novel sustainable systems with advanced properties. Therefore, smart core-shell particles with anisotropic shape and chemical ‘patchiness’ (Janus particles) are promising building blocks for engineering the assembly of particular targeted structures. Moreover, a particularly interesting group of Janus particles comprises the ones specifically responding to change of environmental conditions. Apparently, grafting of two polymers with different sensitivities on opposite sides can further diversify responsive properties of functional Janus colloids.

In this paper, we report a new versatile concept for the design of smart core-shell Janus particles with chemical ‘patchiness’ and controllable Janus balance as well as investigation their self-assembly.

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Electro-colloidal lithography: a new surface patterning technique

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Patterned surfaces have attracted great attention for applications in microelectronics, optoelectronic devices, as well as biological and chemical sensors. Many lithography methods for patterning surfaces in the sub-micrometer range were developed, including photolithography, electron beam lithography, X-ray and “dip-pen” lithography.[1] Each of these technologies has advantages and disadvantages regarding resolution, structural ordering, time and cost. As an alternative, soft lithography such as colloidal lithography has become one of the most robust and versatile route to fabricate ordered microstructures.[2]. Colloidal lithography consists in combining classical nanofabrication techniques with colloidal particles assemblies (2D crystals) used as masks or templates. It is now recognized as a facile, inexpensive, and repeatable lithography technique. However, colloidal lithography still requires the use of expensive or/and hardly handling equipment required in the nanofabrication techniques e.g. for particles etching or metal evaporation.

We will describe an innovative technique for surface patterning simply based on colloids and electrical fields [3-5], the so-called electro-colloidal lithography. Two types of designed surfaces will be presented:
- polymer surface containing non close compact hexagonal arrays of conductive holes
- metal surface containing non close compact hexagonal arrays of metal nanorings.

We will show that a fine tune of surface morphology could be reached by electro-colloidal lithography and will present some applications of the described surfaces.

Microfluidic methods to assess emulsion stability

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Assessment and control of emulsion stability is of high importance in many industrial applications. Access to quantitative information about the coalescence kinetics of emulsions is crucial for the design of many processes in e.g. the petroleum or food industry.

The coalescence dynamics of dense emulsions in pressure-driven flows or under enhanced gravity on the level of individual droplets have not been subject of much investigation until now, which is mostly due to the lack of in-situ techniques with a sufficient time resolution. In this work we present the results of a microfluidic approach to assess coalescence kinetics in oil-in-water emulsions under two experimental conditions: 1.) laminar shear and 2.) compressive forces induced by centrifugation.

For the study of shear-induced collisions we use custom-designed microfluidic circuits. Monodisperse oil droplets dispersed in water, which were formed at a microfluidic T-junction, were injected into a wide channel where droplets are accumulated and undergo collisions. The droplet interactions were followed with a microscope and a high-speed camera. We evaluate the coalescence rate of the emulsion and the distribution of individual coalescence times of colliding droplet pairs. The kinetic parameters were mapped as a function of the droplet size, fluid flow rates, electrolyte and surfactant concentration, and viscosity ratio, and interpreted in terms of colloidal and hydrodynamic forces.

To study the effect of enhanced gravity on coalescence, we use an experimental setup which allows continuous in-situ microscopic monitoring of a monolayer of emulsion droplets in a centrifuged oil-in-water emulsion stabilized by an ionic surfactant. We evaluate the growth of pure oil phase which is formed over time and the evolution of droplet size distribution in the compressed layer. With the experimental setup, the timescales of drop/pure-oil and drop-drop coalescence were directly measured.

The proposed methods can serve as useful tools for in-situ measurements to assess the stability of dense emulsions on the level of individual droplets in pressure-driven flows and under enhanced gravity.
Measuring nanoscale interactions of colloidal quantum dots

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The interparticle interactions are a key property for the self-assembly and colloidal stability of nanoparticles in liquid media. However, quantitative experimental information about the interactions is scarce, because of the shortage of suitable techniques with nanoscale resolution. Our approach is quantitative cryogenic transmission electron microscopy (cryo-TEM). Snapshots of nanoparticle positions are obtained after cryogenic quenching of thin films of liquid dispersion. The precise particle positions allow calculation of the radial distribution function, cluster size distribution, and contact interaction. This approach was validated using magnetic nanoparticles, whose contact interactions are known from the start.[1,2]

Here, we study PbSe semiconductor particles of 5 to 10 nm, so-called quantum dots.[3] They are of interest for the development of novel materials with tunable opto-electronic properties, superlattices of nanoparticles obtained by self-assembly at the liquid/air interface,[4] the temperature dependence of the structures indicates that interparticle interactions play a major role.[5,6] We reveal that the interaction potential of PbSe quantum dots has an anisotropic component, ascribed to the presence of an electric dipole moment. The entropic component of the nanoparticle interaction free energy is as expected from theory, and the interaction enthalpy increases with particle size.[7] 3D nanoparticle positions obtained by tomographic reconstruction reveal whether or not the nanoparticles are adsorbed at the liquid/air interface, depending on the chemical composition of the liquid.

Theory of colloidal gelation: the link between microphysics and rheology

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Gelation in colloidal systems, that is, the sudden rise of viscosity at the sol-gel transition, is poorly understood so far. We propose a model that combines the microscopic interactions and clustering kinetics with a viscoelastic description of the macroscopic response based on the concept of rigidity transition at the cluster level, associated with the growing length-scale of cooperatively rearranging clusters. The theory allows us to account for finite bond energies and the consequent finite dissociation rate of colloidal bonds. This is realized in a suspension of thermosensitive colloids where the interparticle binding energy can be brought from zero up to a final value 12kT [1] at a constant rate in the same sample subjected to rheological measurement. Our predictions, quantitatively confirmed by the experiments, provide evidence of a sharp fluid-solid transition, provided that the final attraction energy reached in the process is larger than a critical value. This theory opens up the way to the rational design of materials switchable between fluid and solid in a fully controlled way.

Guided self-assembly of microgels: from particle arrays to anisotropic nanostructures

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Our work aims at the production of microgel strings, rods and meshes via crosslinking of well-defined particle arrays. Therefore wrinkled substrates are prepared by oxidization of stretched Polydimethylsiloxane (PDMS). Subsequently, microgels of N-vinyl caprolactam/Acetoacetoxy ethylmethacrylate (VCL/AAEM) and N-isopropylacrylamide/N-vinyl-caprolactam (NIPAAm/VCL) are spin-coated onto these highly ordered surfaces thereby forming self-assembled structures within the wrinkle grooves. We consequently transfer those pre-aligned particles onto flat silicon wafers to create periodic microgel arrays with well defined spacings covering large surface areas up to 2 x 2 cm, as confirmed by GISAXS measurements. UV irradiation and redispersion of the assembled particles yields microgel chains with variable lengths up to 100 µm and widths of 500-2000 nm which are tunable by altering the wavelength of the PDMS wrinkles. Due to the thermo-responsiveness of VCL/AAEM colloids anisotropic contraction can be induced, which leads to possible applications as sensors and actuators. By using light or electrosensitive microgels and considering their biocompatibility even more applications, such as synthetic muscle fibers, appear feasible.
Surface roughness guided self-assembly of colloidal micelles

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Self-assembly of colloidal particles into larger structures bears potential for creating materials with unprecedented properties, such as materials with full photonic band gaps in the visible spectrum. Colloidal particles with site-specific directional interactions, so called "patchy particles," are promising candidates for bottom-up assembly routes towards such complex structures with rationally designed properties. Here we present an experimental realization of patchy colloidal particles based on surface roughness specific depletion interactions. Smooth patches on rough colloids are shown to be exclusively attractive due to their different overlap volumes. We discuss in detail the case of colloids with one patch that serves as a model for molecular surfactants both with respect to their geometry and their interactions. These one-patch particles assemble into clusters that resemble surfactant micelles. We term these clusters "colloidal micelles." Similarities as well as differences between the colloidal model system and molecular surfactants are discussed and quantified by employing computational and theoretical models.
Magnetic effects on DNA-mediated nanoparticles clusterisation

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Magnetic composites are of great interest for their potential applications in the targeting of biomolecules and in the treatment of human diseases. These systems include two types of materials, a magnetic material needed for the targeting and a biopolymer that possess a biocompatibility. Magnetic nanoparticles represent a set of unique building blocks whose size and composition are tunable to meet the requirements for such applications. Among these, cobalt ferrite nanoparticles are of particular interest as a consequence of their ease of synthesis via colloidal methods, remarkable chemical stability, excellent mechanical strength, high magnetocrystalline anisotropy, and moderate saturation magnetization. In this work we synthesized core-shell magnetic NPs that comprise a magnetic core (CoFe2O4) and a shell that serves as a platform for the surface functionalization of the NPs with DNA molecules. The magnetic core must be pre-coated with a shell to ensure their stability, biodegradability, and non-toxicity in the physiological medium. In this regard Au coating over magnetic nanoparticles is a very attractive composite system: with Au coating, the magnetic nanoparticles can be efficiently stabilized in corrosive biological conditions and readily functionalized through the well developed Au-S chemistry; the coating also renders the magnetic nanoparticles with plasmonic properties. The Au layers are then functionalized with thiolated DNA molecules via a covalent bond to the surface of the NPs. The as-synthesized multivalent CoFe2O4-Au particles, with many strands of DNA attached on their surface, can be polymerized to build extended materials where we can program the formation of hierarchical architectures and lots of crystal structures. By combining the properties of the multifunctional magnetic nanoparticles with the DNA assembling properties, it is therefore possible to create new types of materials able to form crystals with controllable and tunable physical and magnetic properties, that can be designed based on the placement of the particles, their periodicity and arrangement in three-dimensional space, controlled by the DNA interconnects.
Control of peptide self assembly into fibrils by crowders, osmolytes, and cosolutes

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It is widely recognized that peptide assembly into fibrils (or amyloids) sensitively responds to conditions set by myriad cellular solutes. These cosolutes include two important classes: macromolecular crowders and compatible osmolytes. In effort to resolve the effect by which these cosolutes control peptide self-assembly, we have recently found that addition of macromolecular PEG only slightly affects fibril formation of a model peptide in vitro. Polyol osmolytes, in contrast, lengthen the lag time for aggregation, and lead to larger fibril mass at equilibrium. Model calculations suggest that all cosolutes similarly lengthen the time required for nucleation, possibly due to their excluded volume effect. However, PEGs may in addition promote fibril fragmentation, leading to lag times that are overall almost unvaried. Moreover, polyols effectively slow the monomer-fibril detachment rates, thereby favoring additional fibril formation. Preliminary results from coarse-grained molecular dynamics allow us to follow the first steps of protein assembly and nucleation. Input from experimental solvation thermodynamics of a model peptide are used to calibrate our simulations, and to test the effect of cosolutes on peptide assembly into proto-fibrils. Our analysis provides first hints that cosolutes act not only by changing association or dissociation rates of peptide self assembly, but potentially also by directing the formation of fibrils of varied morphologies with different mechanical properties. Importantly, hydration interactions are suggested as an integral component that directs this process. Although additional experiments are needed to unambiguously resolve the action of excluded cosolutes on amyloid formation, it is becoming clear that these compounds are important to consider in the search for ways to modulate fibril formation.
Surface modification of silica nano-colloids for pickering emulsions

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In Pickering emulsions oil droplets are stabilized by adsorption of solid particles at the oil-water interface. These particles are required to be small, easily available and of course to have suitable surface properties like contact angle and stability. Silica nano-colloids which have a wide range of applications in science and industry are easily available and are therefore a good candidate. However, their surface is too hydrophilic. It had been shown that surface hydrophobicity can be increased by adsorption of cationic surfactant (CTAB) on these negatively charged colloids but this has the disadvantage of charge compensation which decreases their stability.

Recently, we could show that it is also possible to adsorb anionic surfactant (SDS) or non-ionic surfactant (C_{12}E_{10}) on the negatively charged silica particles. This adsorption does not only lead to a more hydrophobized surface but also increases the surface charge and so maintains the stability.

In this contribution we show that such modified silica particles can be successfully applied for the production of Pickering emulsions. In our most recent developments we could create a surfactant free modification by adsorption of oleic acid. We will report on stability and applicability of such modified silica nano-colloids for the creation of Pickering emulsions.

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PLL/Casein and PLAr/Casein Multilayer Films – formation and stability

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Casein represents one of the intrinsically unstructured proteins (IUP), which in their natural state do not adopt stable folded structure. That enables its easy integration into the multilayer films using the sequential adsorption or layer-by-layer (LbL) deposition technique [1]. Therefore, casein together with some biocompatible polycation, can form a membrane, which permeability can be controlled either by its thickness or by the external trigger (pH). Moreover, casein embedded in the multilayer structure preserves its ability to bind calcium ions [2]. We investigated formation of PE multilayer films containing α- and β-casein and their stability when exposed to aqueous environments with various pH and salinity. The negatively charged casein (pH 7.4) was used as a polyanionic layer together with biocompatible polycations poly-L-lysine (PLL) and poly-L-arginine (PLAr). We found that the films with PLL were not stable at strongly acid (pH 2 – due to charge reversal of casein) and strongly basic (pH 11 – due to neutralization of PLL) conditions. At the isoelectric point of casein (pH 4.5), only films containing α-casein are stable. Ellipsometric thickness of the PLAr/casein films was greater than of the PLL/casein films containing the same number of layers. These results were confirmed by the Quartz Crystal Microbalance (QCM-D) experiments. The XPS and FTIR-ATR experiments demonstrated that despite larger thickness of PLAr/casein films they have lower ability to bind calcium ions than PLL/casein ones.

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Plasmon coupling of gold nanoparticles after embedding into p-NIPAM microgel particles

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In the last decades, there was a huge interest in combing organic and inorganic components. Therefore, gold nanoparticles (Au-NPs) have been used for the preparation of hybrid materials.[1] Due to the thermoresponsibility of poly-N-Isopropylacrylamide (p-NIPAM) microgel particles, they are of interest for a lot of applications. In the literature, only a few studies deal with the loading of microgels with preformed nanoparticles, e.g. the usage of Au-NPs to investigate the structure of p-NIPAM microgel particles.[2]

In the present study, the focus is on the interaction of spherical, citrate stabilised Au-NPs with a diameter of 18 nm with chemically cross-linked p-NIPAM microgel particles. To investigate the dependence on the crosslinker content, microgel particles with 0.25%, 5% and 10% N,N’-methylenebisacrylamide (MBA) were synthesized. The received microgel particles were characterized by Dynamic Light Scattering (DLS), Static Light Scattering (SLS) and Atomic Force Microscopy (AFM) while the Au-NP loaded microgels were investigated by means of Transmission Electron Microscopy (TEM) and temperature-dependant UV-VIS-spectroscopy. The sensitivity of the Au-NP Plasmon resonance to changes in the dielectric environment offers to investigate the particle distribution, penetration depth of the Au-NPs and structural changes during microgel collapse. Additionally, the optical properties of the polymer particles can be controlled by the crosslinker density.


Biomembranes and the study of nanoparticle translocation

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We present different model cell mimics that aim at investigating either specific or non-specific processes occurring at the cell surface. In particular, the use of PAMAM based dendrimers in drug delivery has been proposed to occur via a combination of passive and active transport. We investigate how the cell mimetic used influence the interpretation of the mechanism of interaction between the nanoparticle and the cell membrane. On the other hand, specific processes usually occur via membrane bound proteins. We focus as on creating biomembranes where membrane proteins are not functional but close conditions to the original biological environment, so as we can follow those processes involving these type of membranes.
Mechanisms of fibrinogen adsorption on colloid particles

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Under most conditions proteins such as enzymes and immunoproteins show a strong tendency to adsorb at various surfaces. Examples include the immobilization of enzymes on solid matrices in biocatalysis and of immunoglobulins in clinical diagnostics [1,2]. In our study adsorption of human and bovine fibrinogen on polystyrene latex particles was studied using microelectrophoresis, dynamic light scattering and AFM methods. Measurements were carried out for a pH range of 3.5-11 and an ionic strength range of 10^-3 - 0.15M, regulated by sodium chloride. Firstly, the physicochemical properties of fibrinogen and latex particles were characterized for these ranges of pH and ionic strength. It was shown that fibrinogen molecules exhibited amphoteric characteristics, being positively charged for pH< 5.8 (isoelectric point), and negatively charged for higher values. On the other hand, polystyrene latex and mica did not show any isolectric point, remaining negatively charged in these ranges of pH and ionic strength. Afterwards, the electrophoretic mobility of polystyrene latex particles, modified by different amounts of fibrinogen, were studied respectively. It was revealed that for the broad pH range, fibrinogen adsorption on polystyrene latex was irreversible. Only for pH > 9.7 did fibrinogen adsorption not occur. This data was interpreted in terms of the theoretical models postulating a side-on and end-on adsorption of fibrinogen molecules. It was also suggested that anomalous adsorption of fibrinogen at pH=7.4, where fibrinogen and latex particles are negatively charged, can be explained in terms of the heterogeneous charge distribution on fibrinogen molecules [2]. An important aspect of this work is devoted to developing an efficient procedure for preparing covered latex particles with a controlled fibrinogen monolayer structure and coverage.


Surface-grafted glycoconjugate brushes via surface initiated atom transfer radical polymerization from silicon surfaces

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Surface-grafted glycoconjugate poly(2-O-(N-acetylglucosamine)-hydroxy-ethyl methacrylate), PGEMA, brushes were prepared by surface-initiated atom transfer radical polymerization (SI-ATRP) on the initiator-coated silicon wafers. After that, the N-acetylglucosamine of PGEMA was enzymatically polymerized to oligo-N-acetyllactosamine, oligo-LacNAc, by combining recombinant glycosyltransferases. In this work we used the dipping method for the fabricating multivariant assemblies of surface-grafted glycoconjugate brushes onto flat solid substrates. The obtained PGEMA and oligo-LacNAc blocks possess a systematic and gradual variation of molecular weight as a function of the position on the substrate. X-ray photoelectron spectroscopy (XPS) was used to determine the surface chemical changes of the silicon wafers upon surface-confined ATRP of PGEMA. The density and the film thickness of the grafted glycoconjugate brushes were determined by atomic force microscopy (AFM). Further studies of lectin recognition on the glycoconjugate brushes demonstrated that the unique glycan structures of the oligo-LacNAc could specifically recognize galectins. These bioactive brushes have potential applications such as new adhesive materials, templates for bioanalysis and cell culture materials.
Surfactant-rich biocompatible microemulsions for transdermal administration of methylxanthine drugs: a comparative study

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In recent years microemulsions based on biocompatible and safe materials have received increased interest as vehicles for the solubilization of bioactive molecules. Under the perspective of their potential pharmaceutical and cosmetic applications, new bioinspired microemulsions were prepared and structurally characterized to be used as novel formulations for transdermal administration of theophylline and theobromine. Three different microemulsion systems are reported in this study: a) Isopropyl palmitate/Triton X-100/Water/Propylglycol, b) Mygliol 818/ Triton X-100/Water/ Propylglycol and c) Isopropyl palmitate/Triton X-100/Span 20/Water/ Propylglycol. Pseudo-ternary phase diagrams of these systems, determined at 25°C, are presented. Interfacial properties and dynamic structure of the monolayer of the microemulsions were studied by Electron Paramagnetic Resonance (EPR) spectroscopy employing the nitroxide spin probe 5-doxylstearic acid. The systems were studied along the dilution line 73 (70% surfactant-30% oil) both in the absence and in the presence of bioactive compounds, at 25 °C. Particle size measurements were performed using Dynamic Light Scattering. To evaluate the ability of the proposed microemulsions to serve as carriers of bioactive compounds, such as theophylline and theobromine, for topical administration, in vitro permeation studies were carried out using the Franz type diffusion cells and a synthetic model membrane. The presented results reveal many interesting structural features of three complex microemulsion systems that are crucial in understanding the compositional dependence of the microemulsion structure and the delivery of specific bioactive compounds. The nature of the oil and the surfactant used for the construction of the microemulsions affected the phase behaviour, the size and also the interfacial properties of the free and loaded systems. Nevertheless, constant and controlled delivery of theophylline and theobromine from the drug containing microemulsions was observed for all three systems examined.
Antimicrobial properties of novel nanostructured with silver-tiopronin nanoparticles bone cement

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Bone cement is routinely used during orthopaedic surgeries to fix fractures or devices to bones. Infections caused by microorganisms are a possible complication and a serious risk faced by patients undergoing these procedures. These infections are generally treated with antibiotics and are caused by bacteria colonising the bone cement and surrounding areas. However, antibiotics are quickly losing efficacy in treating infections because of the rise of resistant population of bacteria; therefore, the development of novel antimicrobial approaches, not based on antibiotics, is urgent. To this end, metal nanoparticles, particularly silver, have been used to confer antimicrobial activity to surfaces and materials.

In this work silver nanoparticles were obtained through reduction of AgNO\textsubscript{3} and capped with tiopronine; these nanoparticles were 10 ± 2.5 nm in diameter. These nanoparticles were encapsulated into bone cement at concentrations of 1, 0.5 and 0.1 \% (w/w) and were analysed. Bending and compression tests revealed that the addition of these nanoparticles did not affect mechanical properties; furthermore no increase in cytotoxicity was observed against osteoblasts, compared to samples without the nanoparticles. The antimicrobial activity was established against MRSA and S. aureus measuring the growth rate and lag phase of the surviving bacterial cells after contact between bone cement and cells suspension. The two microorganisms exhibited the same behaviour; the antimicrobial activity depended on the amount of silver-tiopronin nanoparticles added, even the lowest concentration used (0.1 \%) was still capable of reducing the number of bacterial cells attached to the bone cement.

Our work shows that it is possible to encapsulate silver-tiopronin nanoparticles in bone cement in order to exhibit antimicrobial activity without causing any detriment effect on the mechanical and cytotoxic properties.
Recent advances in the preparation of anthracycline-loaded colloidal nanoparticles for cancer chemotherapy

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Anthracyclines are among the most effective anticancer agents used in many current clinical chemotherapy regimens. The association of anthracycline drugs (such as epirubicin, doxorubicin, etc.) with a biocompatible and a bioerosive colloidal carrier provides new possibilities for control of the pharmacokinetics of these cytostatics. The colloidal nanoparticles of poly(alkyl cyanoacrylate) (PACA) possess a great promise as drug carrier systems for delivery of anthracyclines, especially in cases of multi-drug resistant cancers. Such particles are classically prepared by emulsion polymerization, starting from the respective monomer. Our recent studies demonstrate that pure and drug-loaded PACA colloidal particles could be successfully prepared by nanoprecipitation, starting from a pre-synthesized polymer. Nanoparticles prepared from human serum albumin (HSA) represent another promising carrier system for anthracycline delivery, especially in the cases where active drug targeting is required. In this report, we consider the advantages and disadvantages of both types (PACA and HSA) of nanoparticle drug carriers, as well as the advantages and disadvantages of the various methods for their preparation. Here, we also describe the effects of the type of nanoparticles, surfactant type, surfactant concentration and other factors on the cytotoxicity and the drug uptake by cancer cells. Based on these observations, we discuss the possibilities for application of such nanoparticles in cancer chemotherapy.

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Some aspects of conducting polymer synthesis

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Conducting polymers are used for many technological applications. Conducting and non-conducting conjugated polymers might be formed and deposited by chemical \cite{1}, electrochemical \cite{2} and even by enzymatic \cite{3-5} synthesis. Electrochemical deposition is usually used in development of various types of electrochemical sensors and biosensors, including (i) enzymatic sensors \cite{2,4,5}; (ii) immunosensors; (iii) DNA sensors; (iv) molecularly imprinted polymer based sensors \cite{6}. The aim is to overview major achievements of authors in synthesis of conducting polymers including basic environmentally friendly chemical \cite{1}, electrochemical \cite{2} and enzymatic \cite{3,4,5} methods. Useful properties of newly generated polymers including applicability in biosensors will be introduced.

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Fabrication of SPR nanosensor using gold nanoparticles and self-assembled monolayer technique for detection of Cu²⁺ in an aqueous solution

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In this work we investigated the fabrication of surface Plasmon resonance (SPR) nanosensor using gold nanoparticles (AuNPs) chemisorbed onto self assembled monolayer of 10-(3-amino phenoxy) decane-1-thiol on gold substrate. The fabrication process of SPR nanosensor was characterized using different techniques such as infrared reflection-absorption spectra (IRRAS), X-ray photoelectron spectroscopy (XPS), Atomic force microscope (AFM) and Scanning electron microscope (SEM). The fabricated SPR nanosensor was used for detection of Cu²⁺ in an aqueous solution using Surface Plasmon Resonance Refractometer. The results confirm the fabrication of new SPR nanosensor. The fabricated SPR nanosensor showed a good activity toward the detection of Cu²⁺. The detection of Cu²⁺ in an aqueous solution using the fabricated SPR nansensor was enhanced in the presence of gold nanoparticles.
Low temperature sinterable Ag nanoparticles for printed electronics applications

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Printed conductive structures are of great interest in industrial electronic applications (i.e. printed electronics applications) primarily due to their versatility, inexpensive production and compatibility with fast roll-to-roll manufacturing techniques. In this regard, metallic nanoparticle inks have attracted a tremendous interest in recent years. In order to fit the targeted markets such as RFID (radio frequency identification), PV (photovoltaics), OLED (organic light emitting diode) etc., metallic nanoparticles should fulfill a range of stringent demands, where sinterability at low curing temperature (below the glass transition temperature of cheap flexible substrates e.g. PET- polyethylene terephthalate) at short processing time, and very high conductivity of the sintered structures (approaching conductivity of the most conductive metal – silver) being the most challenging ones. In this work we present a dispersion of Silver nanoparticles which sinters at low curing temperatures (<150°C) and at short processing time (<5min). The cured Ag layer is highly conductive, reaching up to 50% conductivity of bulk silver metal. Synthesis of Ag nanoparticles is straightforward, up-scalable, with Ag content in the reaction medium reaching up to 14% wt. The obtained dispersion can readily be formulated into an ink and used in a range of printed electronics applications employing flexible substrates.
Ordering of colloidal particles under confinement

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Characteristic lengths of the structuring of silica nanoparticle suspensions confined between two flat silica surfaces were extracted from the oscillatory force profile of colloidal-probe atomic force microscopy (CP-AFM) measurements and compared with the bulk counterparts as obtained from structural peak of small angle X-ray scattering (SAXS). The wavelength \( \lambda \) and the decay length \( \xi \) extracted from CP-AFM correlated well with the mean particle distance \( 2\pi/q_{\text{max}} \) and correlation length \( 2/\Delta q \), respectively, as obtained from SAXS structural peak. This observation suggests that there is no confinement effect on characteristic lengths themselves that represent the structuring, even though the confinement indeed induces a layered structure of the particles [1,2]. A more quantitative study shows a relation of oscillatory wavelength of silica nanoparticle suspensions with the particle number density as \( \lambda=\rho^{-1/3} \), irrespective of particle size, surface charge of the particles [2, 3] and ionic strength of the solution [4]. This is in contradiction with the results described in literature, which predict a scaling as \( \lambda=2(R+\kappa)^{-1} \) with the particle radius \( R \) and the Debye length \( \kappa \) for confined nanoparticles. The decay length \( \xi \) was found to be controlled both by particle size and ionic strength of the solution. A relation of \( \xi=R^{+1} \) is observed at silica particle concentrations below 10 vol % [2]. CP-AFM measurements of silica nanoparticle suspensions on mica substrate [5], polyelectrolyte-coated rough surface(s) [6], and deformable air-liquid interface [7] are further applied to investigate the effect of confining surface properties on the corresponding structuring. CP-AFM measurements on modified and deformable confining surfaces show an increase in amplitude with increasing surface potential and decreasing surface roughness and deformability. However, the corresponding wavelength and correlation length remain unaffected. In addition, uncharged colloids, non-ionic surfactant micelles, are used in AFM to study the effect of surface charge and rigidity of the colloids. The scaling law of \( \lambda=\rho^{-1/3} \) for oscillatory wavelength and the relation of \( \xi=R^{+1} \) for correlation length break down in case of non-ionic surfactant micelles. With increasing surfactant concentration, both amplitude and decay length increase, while the wavelength of micelles remains constant equal to the micelle diameter [8].

Light Activated Colloids: a new building block for smarter materials

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Self-propelled micro-particles are intrinsically out-of-equilibrium. This renders their physics far richer than that of passive colloids while relaxing some thermodynamical constraints and give rise to the emergence of complex phenomena e.g. collective behavior, swarming…I will present a new form of self-assembly originating from non-equilibrium driving forces. When activated by light, a set of new self-propelled particles spontaneously assemble into living crystals which form, break, explode and reform somewhere else. We will show that this complex dynamics originates in the competition between self-propulsion of the particles attractive interactions induced respectively by osmotic and phoretic effects. Attraction and propulsion triggered on-demand by a light stimulus opens new perspectives in the design and the properties of smarter materials
Colloidal behavior of oxidized tannins

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Physico-chemical interactions involving condensed tannins, together with polysaccharides and proteins, take a primary part in wine stability, clarification and taste. These interactions, despite their importance, are not fully understood yet. This is mainly due to the difficulties encountered in the structural analysis of these compounds. Condensed tannins are flavan-3-ols oligomers and polymers. Native tannins differ by the nature of their constitutive units and by their average degree of polymerization (aDP). This initial complexity increases during winemaking and ageing: due to their chemical reactivity, tannins are not stable once extracted from grapes. They undergo several structural changes (oxidation, recombination, cleavage), leading to the formation of derived tannins. These modifications are important as these new compounds, which represent a large part of wine tannins, are expected to exhibit properties that are different from those of their precursors. Dynamic light scattering, along with small angle X-ray and neutron scattering were used to address the questions of (i) the conformations, molecular dimensions and solubility of tannin oligomers/polymers in ethanol and aqueous solvent/ethanol mixtures, (ii) the changes induced by autoxidation reactions. This information is required to elucidate the mechanisms of their interactions with other biopolymers. Experiments on purified fractions of different aDP and composition indicated that if tannins are mainly soluble in the tested concentration range, some structures within the fractions form colloidal dispersions and/or precipitates. Aggregation and precipitation depend on the ethanol content and on the ionic composition\(^1,2\). Structures prone to aggregate/precipitate were identified as oxidized tannins. Further studies on the structural and solubility changes induced by autoxidation showed that these changes are dependent on both tannin aDP and concentration\(^3,4\).

3 Poncet-Legrand et al. (2010), Biomacromol, 11: 2376.
A novel nanocarrier for an effective dermal drug release

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The present investigation was devoted to the evaluation of a novel nanostructure useful as a platform for the development of nanocarriers able to protect, transport and release sensitive therapeutic agents. Such a nanostructure was tested for its ability in release diclofenac into the deep skin strata. Two penetration enhancers, namely monoolein and lauroylcholine, were combined to formulate an undeformable liposome-like nanostructure endowed of excellent stability (> 6 months) and skin penetration ability, along with short term low cytotoxicity, as evaluated via the MTT test. The physicochemical features, investigated through SAXS, DLS, and Cryo-TEM techniques, revealed that the nanostructure is retained after loading with diclofenac in its acid (hydrophobic) form. The drug release performances were studied using intact new born pig skin. The analysis of the different skin strata indicates that the drug mainly accumulated into the viable epidermis with almost no deposition into the derma. Indeed, the flux of the drug across the skin was incredibly low, with only 0.3% release after 24 h. These results validate the use of this novel formulation for topical drug release when the delivery to the systemic circulation should be avoided.
Magnetic transport, mixing and release of cargo with
tailored nanoliter droplets

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The controlled transport, mixing and release of small amounts of cargo is important in many fields and applications. From the various types of carriers available, droplets in the form of single or double emulsions provide a convenient and flexible platform for cargo encapsulation, transport, mixing and release. Double emulsion droplets are particularly interesting due to their ability to encapsulate reagents that are soluble in both the inner droplet and the outer continuous phase. This has proven to be critical in drug delivery applications and for protection of food ingredients and analytes. Here, we present a method to produce and magnetically manipulate sub-millimeter single and multiple emulsions that offers controlled handling of nL size fluids. Such magnetic-based handling of single droplets has so far been limited to single emulsions that usually exhibit µL volumes and millimeter sizes.

The monodisperse nanoliter sized double and single emulsion droplets were made in microcapillary devices. We added superparamagnetic iron oxide nanoparticles (SPIONs) in the inner phase of single emulsions (MO/W) and to either the intermediate (W/MO/W) or inner phases (MW/O/W) of double emulsions. Because of the presence of SPIONs the resulting droplets display a finite magnetic susceptibility under external fields, experiencing a magnetic force under magnetic field gradients. We show that at low magnetic fields, the emulsion droplets can be transported and collected. By increasing the field, single and double emulsion droplets were fused, resulting in mixing of the reagent. Further we demonstrated for single emulsion droplets, that by addition of small amounts of salt, the stability of the emulsions towards fusion can be altered controllably in order to reduce the external magnetic field needed for droplet fusion. Also, we show programmed release of fluorescent probe molecules from W/MO/W double emulsions and local gelling of PAA dispersed in water by release of the charged SPIONS that were previously protected in a MW/O/W emulsion.
Synthesis of tuneable core-shell rods and their use in stabilizing interfaces of partially miscible liquids

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First a simple method for preparing micron sized rods consisting of an iron oxide (akaganéite, \(\beta\)-FeOOH) core and a silica shell will briefly be introduced. The composition of these particles allows easy modification of their properties, for example: varying aspect ratio, removal of the iron oxide core, fluorescent labelling, and changing surface chemistry.

Second we demonstrate the use of these particles to stabilize emulsions and gels prepared from phase separating liquids. Recent studies pointed out significant effects induced by replacing commonly used spherical colloids trapped at an interface of immiscible liquids by rod-like particles. Pursuing this further, our experiments consist of demixing the liquid components by changing temperature in the presence of the colloidal rods. The particles are swept up by the interface created in the phase separation, in most of the cases resulting in particle stabilized emulsions. A special case occurs when the liquids separate via spinodal decomposition and the particles exhibit approximately neutral wetting, where the percolating interface is populated by particles which ultimately jam, arresting the further coarsening of the liquid domains to form a bicontinuous interfacially jammed emulsion gel (bijel).

Samples are studied with fluorescence confocal microscopy to elucidate morphologies of the stabilized liquid domains and to resolve smaller structural features, also on the single particle level. We have used changes in particle surface chemistry to control their wetting properties, enabling us to alter the contact angle of the rods trapped at a liquid-liquid interface, and also varied sample composition to investigate their effects on the formed structures. Initial results already show interesting features not seen in previous experiments with spheres, like the stabilization of extremely thin liquid bridges.
Soft nanomaterials in capillary electrochromatography: a novel approach for protein analysis

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New approaches for efficient protein analysis are of utmost importance, driven by the breakthroughs in proteomics and emergence of protein-based drugs. In nanoparticle-based Capillary Electrochromatography (CEC), soft nanoparticles are suspended in the electrolyte to prevent adsorption of proteins to the capillary wall during analysis. Assisted by the enhanced mass transfer due to their large surface area, an increased efficiency is demonstrated when soft nanoparticles are used as the pseudostationary phase (PSP). The approach also benefits from the use of an entirely new stationary phase with every run, avoidance of column packing and very low nanoparticle consumption (<10 µg) per analysis. Previous studies in our group demonstrated the use of anionic and cationic forms of lipid-based liquid crystalline nanoparticles (diameter: 70 nm, pore size: 100 Å) in CEC for the microanalysis of genetically modified variants of green fluorescent proteins (GFP) in fluorescence detection mode with high efficiencies (800 000 plates/m) and with selectivity similar to that of capillary isoelectric focusing. Preliminary results for using UV detection mode indicated good baseline resolution with short analysis time (less than 5 min) for BSA, IgG and lysozyme. The system is currently being investigated on the use of Dendrigraft Poly-L-Lysine (diameter: ~ 10 nm) and PAMAM-dendrimer nanoparticles (diameter: ~ 20 nm). The PSP-CEC system holds a promising potential for elucidating protein-nanoparticle interactions. Future studies will be focused toward membrane proteins (e.g. aquaporins) to provide a model to illustrate the protein nanoparticle interactions at the subcellular level since such interactions hold a key position in nature, nanomedicine and nanotoxicity. When hyphenated to MS, the system will enable the handling for complex biological mixtures for proteomics. The PSP-CEC approach for the analysis of proteins mentioned herein enables the comprehension of the physicochemical characteristics of self-assembled, soft nanomaterial to gain more control on their internal structure to optimize their functionality for future applications in biomedical and bioanalytical sciences.
Nanoparticles at the water-oil interface: x-ray determination of the contact angle

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Self-assembly of nano-particles (NPs) at liquid-liquid interfaces is of fundamental scientific interest and great technological impact. Accurate measurements of the NP’s contact angle with the interface $\Theta$ and binding energy $\Delta E$ are very challenging, particularly for NPs residing at deeply buried interfaces between immiscible liquids.

We report x-ray based $\Theta$ measurements and classical $\Delta E$ calculations for silica nanoparticles (NPs) adsorbed at a flat interface between hexane and aqueous solutions of cetyltrimethylammonium bromide (CTAB) surfactant. The interfacial structure is determined with Ångström resolution by high-energy (70 keV) x-ray reflectivity (XRR) for two CTAB concentrations, $\phi_c$, well below and near the critical micelle concentration, $\phi_{cmc}$. The XRR data are analyzed using a physically motivated model in which the fitting parameters are directly related to the main structural properties of the interface.

At low $\phi_c$ (0.05 mM) we find a dense monolayer (ML) of close-packed NPs with $\Theta = (146\pm4)^\circ$, while higher $\phi_c$ (0.75 mM) yields a less dense ML with lower $\Theta = (128\pm6)^\circ$. The NP close-packing scenario suggests a solid-like character for the low-$\phi_c$ ML, which is likely stabilized by strong inter-NP interactions that are neglected in the standard $\Delta E$ calculation. The latter yields $(24\pm10)kT$ and $(5\pm2)kT$ for the high and low $\phi_c$ systems, respectively.

The low $\Delta E$ obtained at low $\phi_c$ contrasts starkly with the highly stable, close-packed XRR-determined monolayer structure, thus highlighting the need for more advanced interfacial binding theories, particularly in the case of dense MLs of interacting nano-sized particles. We hope that this experimental study will motivate novel theoretical and simulation work leading to a deeper understanding of the energetics, formation, and hydrophobicity-related properties of these technologically important nano-scale multi-component interfaces.
Mesocrystallisation of self-assembled nanoparticles: following nucleation and growth using time-resolved grazing incidence small angle X-ray scattering

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The ability to control structure and functionality at all length scale has developed tremendously in the last decades. It is clear that optimal design of nanostructured materials require integration of various approaches to synthesize, functionalize, characterize and process the nanosized species for various applications. We have previously demonstrated how it is possible to assemble iron oxide nanocrystals into superlattices with both translational and orientational order (mesocrystals) through evaporation-induced self-assembly [1, 2]. Here, we will present recent work on the structural evolution and the formation kinetics of large and highly ordered mesocrystals of superparamagnetic iron oxide nanocubes and nanospheres of different sizes using time-resolved grazing incidence small angle X-ray scattering (GISAXS) and video microscopy. The time scale for the formation of relatively large ordered domains was found to be very short, on the order of seconds. The nucleation and growth invariably occur at the three phase contact line for the receding droplet as the solvent slowly evaporates. We will discuss how mesocrystals with sizes exceeding 100 µm can be formed by optimisation of the evaporation rate and how the size, morphology and mesostructure, determined by a combination of GISAXS, electron microscopy and atomic force microscopy, is affected by the magnitude, range, direction and duration of the intrinsic and induced interparticle forces.

References:
An x-ray reflectivity study of core-shell poly(ethylene glycol)-iron oxide nanoparticles at liquid-liquid interfaces

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Nanoparticle (NP) self-assembly and crosslinking at liquid interfaces (SALI) provide a method for the fabrication of large area, ultra-thin functional materials [1]. The full characterization of the NPs at the interface assumes therefore key importance for the design of novel nano-materials. Currently, most experiments deal with SALI of “hard sphere” NPs [2] which tend to aggregate at interfaces, leading to a non-uniform microstructure, to a poor control on the inter-particle separation [3] and to the inability to crosslink the material without direct core contact. Such obstacles can be overcome by using core-shell NPs stabilized by sterically repulsive polymer shells [4]. We applied a novel method developed at the ESRF ID15 beamline [5] to measure by means of high-energy X-ray reflectivity (HE-XR) the water immersion of composite, core-shell iron oxide-poly(ethylene glycol) (PEG) NPs at a buried water/decane interface for a variety of polymer molecular weights and architectures. We obtained time-resolved effective NP contact angles and average inter-particle separations via detailed modeling of the XR profiles. In particular, we found that the effective contact angles of core-shell NPs at the water/decane interface depend on the molecular weight and architecture of the polymer shell, with larger molecular weight linear PEG having higher (but still below 90°) effective contact angles, due to the larger surface activity of longer PEG chains. Our XR results also indicate that the separation between NPs at saturation coverage of the interface (after 12h adsorption) is consistent with deformation of PEG chains. Finally, the time evolution of the XR signal confirmed the slowing down of NP adsorption with increasing times (interface crowding) as we had previously observed by numerical simulations and interfacial tension measurements [6].

Neutron reflectometry study of the interactions of PAMAM dendrimers with surfactant at interfaces

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We have employed neutron reflectometry (NR), using isotopic contrast variation, to reveal the interactions between cationic poly(amidoamine) (PAMAM) dendrimers and the anionic surfactant sodium dodecyl sulfate (SDS) at the silica-water and air-water interface. Dendrimers are highly hyperbranched, symmetric and monodisperse macromolecules that have potential for a range of applications where self-assembly nanostructures are needed. The aim of this project is to understand the interactions between PAMAM and SDS in order to develop a model for adsorption layers of strongly interacting macromolecular mixtures. We have performed measurements at both types of interface using the reflectometer FIGARO at the ILL. PAMAM generations 2, 4 and 8 (G2, G4 and G8 respectively) have been studied to take into account the effects of size, molecular weight and number of surface groups. In sequential addition experiments at the solid-liquid interface, PAMAM G4 and G8 adsorb on silica in an irreversible process. Exposure to SDS results in adsorption on the dendrimer which swells due to a reduction in strength of the electrostatic interaction with the substrate. As the concentration of SDS increases, the surfactant hydrophobic tails penetrate the more hydrophobic interior of the dendrimer and the complexes can be regarded as mixed micelles. In contrast, the layer structures formed on silica by pre-mixed PAMAM/SDS are very different, which shows that non-equilibrium effects are important due to the formation of kinetically-trapped bulk aggregates. At the air-water interface, there is a synergistic enhancement of adsorbed SDS in the presence of PAMAM dendrimers. NR profiles of PAMAM G2 with SDS show a Bragg peak indicating interfacial multilayers, while G4 and G8 do not show this behaviour. Our examination of PAMAM/SDS interactions at interfaces has showed the importance that the polyelectrolyte architecture has on the adsorption behaviour of polyelectrolyte/surfactant systems.
Werner Kunz opened experimentally new grounds at the interface between micellar solutions, ionic liquids and concentrated ionic solutions, by using new combinations of large anions and carefully chosen cations and combining all this colloidal chemistry also with the fields of hydrotropes and osmolytes. To do this he applied his profound knowledge of specific ion effects to invent new substance classes and new commercial applications.

Werner designed in the last few years a new family of non-toxic surfactants/emulsifiers, based on choline as counterion to long-chain alkylcarboxylates and -sulfates. The secret is that the natural (and even healthy) choline cation decreases the Krafft temperature so that even long-chain anionic surfactants are soluble in water at room temperature. This may open new perspectives for the use of triglycerides from European plants instead of palm oil.

He also managed to make the first room temperature ionic liquids with sodium as simple cation. Another highlight is the combination of a simple, but carefully chosen anionic surfactant with a standard cationic surfactant so that this true cat-anionic surfactant is liquid at room temperature (the first cat-anionic surfactant room-temperature ionic liquid) with several promising potential applications.

In the field of microemulsions he combined this knowledge to make low toxic systems and new ionic liquid based microemulsions that are liquid and structured between -50 and +250°C. He has even emulsified efficiently biodiesel via a colloidal approach and introduced this new system to the chemical engineering world.

One of his other remarkable achievements is the mixing of triglycerides in water with a minimum amount of surfactant and further additives, again carefully chosen and based on his knowledge of hydrotropes and specific ion effects. He edited in 2010 a reference book on “specific ion effects”, i.e. the state of the art in Hofmeister effects, from observation to modelling and usage in colloid science based products. He is co-author on the cutting-edge quantification of an extension of simple chaotrope/cosmotrope view towards matching affinities, which is currently the only theory able to provide parameter-free predictions.

All this recently published work – most of it in high impact journals – results from work at University as well as in a small eclectic company with linked activities. His work has a large impact not only in the scientific community, but also in large industry. In fact, most of Werner’s work is sponsored by industry and many of his inventions led to new and improved products, especially in the field of cosmetics and household products that are on the market.
These completely new and elegant knowledge-based colloidal systems, designed by an artful combination of thermodynamics with ion-specificity and surfactant science gave him the 2012 ECIS-Rhodia prize.
From solvents and ions to surfactants and interfaces

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Surfactants and polymers can aggregate in solvents. Both the amphiphilic molecules and the solvents can be charged, if the solvent consists only of ions, it is termed Ionic Liquid. From an academic point of view it matters to consider the particularities of each species in aggregation colloids: amphiphiles and / or macromolecules, ions and solvents. To do so, we came back to very basic questions that are seemingly out of date. For example: how to characterise the continuous cross-over from unstructured solvents to loosely structured hydrotropic solutions to clearly structured surfactant solutions? Can we establish general models for specific ion effects in colloidal systems? Can we really make surfactant-free microemulsions? How to dissolve very hydrophobic polymers in aqueous solutions? What is the pH in a colloidal system?

Having a little bit more understood these issues, we can take profit of this knowledge to design new systems with better or particular properties. Example: specific ion effects can be used to make new and “greener” surfactants, to transform classical Ionic Liquids into green solvents or to extend the temperature range of the liquid phase in microemulsions. The comprehension of such effects allows also a deeper insight into more special phenomena such as the spectacular self-organisation of salts in so-called biomorphs, silica gardens, and dissipatively ordered alginate gels.

Finally, I will give some examples how to link this basic research to more applied fields such as the formulation of new materials and consumer products, under special consideration of Green Chemistry.
OVERBEEK PRIZE 2012 FRIDAY
**Dominique Langevin** started her PhD on light scattering of liquid surfaces in 1974. As post-doctoral fellow she then studied the diffusion of nanoparticles dispersed in a mesh of semi-dilute polymers and in emulsions, as an independent new research themes at the Kastler-Brossel laboratory for atomic physics. This was the start of the physical branch which is now designed as colloidal science or soft master, following the lead of Pierre-Giles de Gennes. He was at that time working on supra-conductivity and liquid crystals physics. Dominique created the soft condensed matter team at ENS in the statistical and atomic physics so-called Kastler-Brossel laboratory.

Using optical methods to study interfaces and the different energy terms that define surface tensions allowed clear distinction between emulsions and microemulsions. She investigated experimentally and theoretically the origin and control of ultra-low liquid surface tension by microemulsions, driven by interests in enhanced oil recovery.

From there, she got interested in what was then referred to as “anomalous” phases, like giant o/w micelles and sponge phases, that were first reported in Åbo and Lund by K Fontell. At the time, these systems had not been clearly identified and there was no previous understanding of their surface tension properties.

Thirty years ago, Dominique spent some months collaborating with the Ninham group in Canberra, who made parallel work in giant w/o micelles. She was also invited several times at ITP in Santa Barbara.

The main break-through, thanks to the light scattering approach combined with neutron and X-ray scattering was the explanation of non-monotonic behaviour of the surface tension of polymer-surfactant mixtures, and its dependence on frequency. This is the key to understand drainage in foams and emulsions. It was known and reported several times that surface tensions are not always monotonic with concentration. This is seemingly incompatible with Gibbs adsorption, but it is so for binary solutions. However, when the fluid is a complex fluid, exhibiting several pseudo-phases, mixed micelles may act as micro-reservoirs and deplete polymers from the interface. This is the rationale of non-monotonic interfacial tension with activity. This is now of general knowledge and is extremely useful in designing foaming and anti-foaming agent. It has relevance in a vast number of applications, and the advances of Dominique Langevin’s research have decreased the part of black magic in the different regimes she identified, including kinetic stability. This is nowadays the basis for shampoo and water-based lubrication.
She then broadened her expertise in film studies from light towards neutron scattering and made ground-breaking contributions to understand the viscoelastic properties of monolayers and ultrathin liquid films, as well as the frequency dependence of these properties. Before the work of Dominique Langevin, surface tension was in France attributed to the presence of a critical point, and not to a monolayer of surfactant. This included also the surface tension of thin films with polyelectrolytes and polymeric films. She discovered that ellipsometry can be used as a direct method for measuring roughness of thin films loaded with fluctuating polymers. For such systems, the layering close to an interface of micelles or proteins was known from liquid thin film balance. Dominique Langevin discovered that polyelectrolytes, formed a mesh, with sizes apparent in force-distance measurements and thus unexpectedly indicating a layered structure.

In the last ten years, she was the leading scientist in stability and evolution of foams of colloidal solutions. Most prominent has been her work on oscillatory forces encountered in the thinning of thin liquid films. This could be related to characteristic sizes of polymeric particles or blobs in polymer solutions.

She has not only been an eminent scientist, but her work triggered several working groups, such as the groupe de recherches “films de tensioactifs flexibles” (1991-1995), an equivalent to what would be a French Colloid Society, who legally does not (yet?) exist. She established the surfactant group at ENS, the surface of fluids group at CRPP Bordeaux, which both persisted after she has left. She led the soft matter group in the famous Guinier-Castaing-Friedel laboratory in Orsay.

She was awarded the CNRS Silver medal, which is the highest award in Chemistry at CNRS, and the l’Oreal price for women in Science and the Gentner-Kastner award of the German Physical Society.

Dominique Langevin has also been a successful mentor in colloid and interfaces science. Among her former post-docs, a large number came via French-German exchanges such as Michael Gradzielski now professor in Berlin, Thomas Hellweg now professor in Bielefeld, Cosima Stubenrauch now professor in Stuttgart, Regine V.Klitzing now professor in Berlin, Some came also from outside Germany like Bernard Binks, the 2011 ECIS-Rhodia prize-winner who is currently professor at Hull University, Vance Bergeron now in Lyon, and also Antonio Stocco now in Montpellier to name a few. Between 1994 and 1998, Dominique Langevin was the director of Centre de recherches Paul Pascal in Bordeaux, one of the largest laboratories in colloids (organizer on ECIS 1988).

She is the author of more than 300 papers, among which many are essential references now. She published papers in different sub-fields, applying general scientific approaches to practical problems in several areas, ranging from microemulsion and oil extraction in the seventies, to foaming and anti-foaming agents, including physics, chemistry as well as biophysics.
Dynamic properties of adsorbed layers at liquid interfaces

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It is now well admitted that in surfactant systems, the behavior of wetting, two phase flows, emulsions and foams, is strongly influenced by the dynamic properties of the surfactant monolayers adsorbed at oil/water and air/water interfaces. During the last years, a number of commercial devices allowing the study of these properties became available, stimulating the research in the area. They include dynamic surface tension, surface compression and surface shear rheology instruments. We will give a few examples of correlations between adsorbed layers properties (including polymers and particle layers) and the behavior of the fluids at macroscopic scales.
Phase diagram and arrested states of Laponite in aqueous solutions: insights from simple models for a complex phase behavior

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Laponite is a synthetic smectite clay resembling the natural clay mineral hectorite. In water, it forms a colloidal dispersion of nanometer-sized discotic platelets with positive charges on the plates and negative ones on the rims. The anisotropy in the shape and in the interactions, in addition to the nontrivial and very slow aging dynamics, makes it the prototype of a complex colloid. To explain recent experimental findings, which have provided evidence of different non-ergodic states with varying clay concentration, we use a combination of modeling and numerical simulations. At extremely low clay concentration, a phase separation is observed between colloid-poor and colloid-rich phases, the latter arresting into a so-called equilibrium gel state [1]. This phenomenology is strikingly similar to that of patchy models, pointing to the fact that the behavior of the system is largely determined by the attractive directional interactions between face and rim charges. We indeed describe the experimental findings with a simplified patchy model, in which each Laponite platelet is schematized as a hard rigid disk composed by 19 sites on a hexagonal regular mesh. The disks are decorated with five attractive sites: three on the rim and one at the center of each face. Only face-rim bonds (so-called T-bonds) are allowed. We also compare the structural evolution of the system after quench at low temperatures - mimicking the quasi-irreversible aggregation process taking place experimentally with increasing waiting time - is compared to the experimental results. In these conditions, we argue that the platelets are preferentially organized in a “house-of-cards” network. At larger, but still very low clay concentration, a different type of structure (with different aging dynamics) is observed. With a simple but impressive dilution experiment, we are able to distinguish whether attractive or repulsive interactions are dominant in the formation and stability of the arrested structure. The comparison of theoretical and numerical calculations with Small Angle X-rays Scattering (SAXS) data provides an estimate for the effective interactions between Laponite platelets. We then argue that this arrested state is a disconnected Wigner glass, stabilized by the screened Coulomb repulsion [2]. The finding of a percolating network structure at low clay concentration and a disconnected one at larger (but still very low) concentration is attributed to the different timescales controlling respectively the competing attractive and repulsive interactions. Finally we illustrate our view on some of the many (still) open questions regarding Laponite and on how the different physical ingredients could be taken into account to design a more realistic model for such a complex system [3].

Using spectroscopic and scattering methods to assess the quality of musculoskeletal tissues

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The number of patients with musculoskeletal diseases, such as osteoporosis and osteoarthritis is steadily increasing due to the aging population. Therefore, urgent scientific development in diagnosing and treating these disorders are needed. We are currently investigating several methods that may provide unique knowledge and understanding of how the quality of the biological tissues are affected by these diseases. High-resolution x-ray imaging methods are used in combination with Fourier Transform Infrared imaging spectroscopy (FTIR) and small-angle x-ray scattering (SAXS) to characterize how the molecular composition, structure and orientation in these tissues are affected on the macro-, micro- and molecular-level. The long term aim is to understand how these factors affect the anatomical function and mechanical competence of the tissues.

By using bone and cartilage tissues from diseased human donors or animal models where osteoporosis or osteoarthritis have been inflicted, it is possible to elucidate how tissue quality altered with these diseases. How is the mineral crystal size and collagen fibre orientation and anisotropy are altered in patients that are highly prone to bone fractures, e.g. osteoporosis, or patients with severe knee pain and cartilage damage, e.g. osteoarthritis? Moreover, the use of FTIR in musculoskeletal tissues is in focus by analysing spatially the organic and inorganic components of the tissues in combination with advanced multivariate and clustering analyses methods. The improved sensitivity and specificity is promising for future disease identification.

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

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Hydration of solid nanomaterials and biopolymers occur via two different mechanisms. When water interacts with solid porous materials, that possess large solid - gas interfaces, the solid - water interactions have similar nature as the solid – inert gas interactions, where typically three regimes of sorption are distinguished: adsorption, capillary condensation and post-capillary condensation uptake [1]. Examples of materials hydrated by this mechanism are MCM-41 and SBA-15 [1-3]. In this case, the initial part of the sorption isotherm can be described by the BET or similar models.

When water interacts with initially dry solid biopolymers (such as proteins or carbohydrate polymers), the mechanism of interaction is different. Capillary condensation of water in biopolymers is typically not observed on the sorption isotherm. This indicates that water uptake in such materials occurs at a solid-solid, not at a solid-vapour interface [4, 5].

Sorption calorimetric results obtained on several biopolymers show that at low water contents in the glassy state the hydration enthalpy is not strongly dependent on the nature of the biopolymer. For example, for both ionic and non-ionic polymers it is close to -18 kJ/mol, which was explained using ab initio calculations. Based on sorption calorimetry data we showed that for water sorption at solid-solid interface, strong enthalpy-entropy compensation is observed [4]. Due to this compensation, the Langmuir sorption isotherm can be used to model water sorption at solid-solid interface. This approach was validated for the cases of microcrystalline cellulose [5] and lysozyme [4]. The sizes of structural units of these materials calculated using the Langmuir model are in good agreement with structural data.

The long-range interaction component in electrostatics of DNA: effects of ion hydration shell dielectric saturation

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Engelhardt Institute of Molecular Biology, Moscow, Russian Federation

The epsilon-modified Poisson-Boltzmann (e-MPB) theory [1,2] is a development of the modified Poisson-Boltzmann (MPB) theory [3] of the electrical double layers. This implicit-solvent approach incorporates a dielectric model of the peculiar polarization of the ion hydration shells in addition to the interionic correlations and short-range ion-ion interactions described in the MPB theory. The set of e-MPB equations has been solved on a three-dimensional grid for an all-atom geometry model of B-DNA [2,4]. Results were obtained for the models of one and two DNAs in solutions of chlorides of alkali and alkaline earth metals and their mixtures. The e-MPB study allows evaluations of the ion size, interionic correlation, and solvent dielectric saturation effects on the ion distributions around DNA and DNA-DNA interaction energies. It suggests that inclusion of the dielectric saturation of the ion hydration shells drastically affects distributions of alkaline earth metal ions around DNA. It results in high affinity of calcium or magnesium ions to the phosphate groups of DNA and a decrease of the mean permittivity around DNA, compensating the DNA-DNA attraction arising from interionic correlations. As a result, two DNA macromolecules in solution of MgCl₂ repel each other in accordance with experimental data. Calculated ionic distributions around DNA and DNA-DNA interaction energies agree with previously published simulations and experimental data. As follows from comparison of the e-MPB results with experimental energies of DNA-DNA interactions in solutions of LiCl, NaCl, CaCl₂, and MgCl₂, the ion-specific effects for different cations of equal valency originate from the short-range ion-DNA interaction. On the other hand, the long-range interaction effects on electrostatics of highly charged macromolecules can be approximated by the dielectric model of the cation hydration shell.

tOR-SANS of polymer-like micelles under large amplitude oscillatory shear (LAOS)

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Polymer-like micelles (PLMs, also worm-like or thread-like micelles) are models for understanding polymer physics as well as of fundamental technological interest across a broad range of applications ranging from enhanced oil recovery to many consumer products. Recently, significant interest has developed in the LAOS behavior of PLM solutions as both a characterization method as well as for its intriguing nonlinear rheology. Here we study a well-characterized formulation of 6 wt% (2:1 molar ratio) cetylpyridinium chloride (CPCl) and sodium salicylate (NaSal) with 0.5M NaCl in D2O by simultaneous time resolved SANS combined with LAOS. Specifically, we show that the steady, shear banding shear rheology can be predicted from LAOS measurements using the Giesekus constitutive model. The LAOS response exhibits “looping” characteristics in the viscous Lissajous representation, which is postulated to be a consequence of large stress overshoots that may be associated with dynamic shear banding. Here, we examine the PLM solution microstructure during LAOS via a newly developed method of time-resolved SANS under LAOS in the velocity-gradient (1-2) plane and velocity-vorticity (1-3) planes of flow (tOR-SANS). This enables both time and spatial resolution of the microstructure and local rheology. We demonstrate how the microstructure tracks the applied oscillatory deformation rate at low Deborah numbers, but becomes nearly constant at higher Deborah numbers. Furthermore, we demonstrate the microstructure origin for the massive stress overshoots observed during start-up flow. These results provide the first, complete measurement of the structure-property relationships for dynamic oscillatory and flow start-ups for a model PLMs and as such can be used to validate and improve constitutive equation models developed for polymer rheology as well as aid formulators of this technologically important class of complex fluids.
Multilayers at interfaces of an oppositely charged polyelectrolyte/surfactant system resulting from the transport of bulk aggregates under gravity

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The formation of functionalized interfaces using multilayer assemblies has attracted considerable recent interest both from fundamental and applied perspectives. Spontaneous multilayer formation at air/liquid and solid/liquid interfaces has been reported in a range of soft matter systems, in many cases involving macromolecules. A common interpretation of such observations is surface-self assembly, i.e., spontaneous ordering of molecules induced by the presence of an interface. We show conclusively for the first time that multilayers at interfaces of an oppositely charged polyelectrolyte/surfactant system can instead result from the transport under gravity of bulk aggregates with internal molecular structure that have a different density to the liquid. This process was proven by a series of measurements at the air/liquid and solid/liquid interfaces of poly(diallyldimethylammonium bromide)/sodium dodecyl sulfate solutions using neutron reflectometry. In the latter case a novel approach involving the comparison of reflection up vs down measurements was used. A strong Bragg peak and clear off-specular scattering are exhibited under three strict conditions: (1) only for samples in the phase separation region, (2) only for fresh samples where a suspension of bulk aggregates remains in solution, and (3) only when the creaming or sedimentation process occurs in the direction of the interface under examination. This bulk transport mechanism is an alternative route of formation of interfacial multilayers to surface self-assembly where the two processes surely give rise to interfaces with very different physical properties. Also, while it is most intuitive to consider an air/liquid interface with air above a flat liquid surface, this is not the situation in droplets and foams. As such, we believe that our findings may be relevant to nanostructured interfaces concerning a range of synthetic and biological macromolecule/surfactant systems such as those involving proteins or DNA.
Colloid and Interface Science is a field that involves many scientific disciplines, from physics and chemistry to biology and medicine. Attention to the field has increased tremendously in recent years, through the strong developments of what is now known as nano-science and soft matter research - both of which may be regarded as subfields of Colloid and Interface Science.

In the present half-day symposium, which starts directly after the 26th ECIS conference, leading scientists representing the diversity of strong European traditions in Colloid and Interface Science will give broader overviews describing how various key themes of the field have developed to their roles of today. A very special circumstance has made it possible to attract very prominent speakers to the symposium: Björn Lindman, one of the founding fathers of ECIS, turns 70 in 2012. Internationally, Björn is a central figure in Colloid and Interface Science since several decades. He is also an outstanding member of the Swedish scientific community, where he is a member of both the Royal Swedish Academy of Sciences and the Royal Swedish Academy of Engineering Sciences.

Björn became Professor of Physical Chemistry at Lund University in Sweden in 1978 and rapidly built up a large research group, with activities ranging from theoretical work on intermolecular and interparticle interactions to problem-solving in collaboration with industry. In his own research on a wide range of amphiphiles, such as polar lipids, surface active proteins, surfactants, surface active block copolymers and hydrophobically modified cellulose derivatives, Björn has made very important contributions to several research topics.

Björn was a pioneer in using molecular self-diffusion data, obtained by NMR methods, to obtain information on microheterogeneous media such as microemulsions and micellar solutions. NMR diffusometry is now an established technique, but in the 1970’s this was exploratory work, which turned out to be very successful. Thus, NMR diffusometry was one of the main tools to demonstrate that microemulsions need not consist of closed domains surrounded by a continuous domain; the oil and the water domains may both be continuous.
During the end of the 70’s and the 80’s Björn and his students made thorough studies of the phase behavior of aqueous surfactant systems. The work comprised both binary surfactant-water systems and ternary systems involving oil. In the 90’s Björn Lindman turned to the even more complex aqueous mixtures of polymers and low-molecular weight surfactants. Such systems are extremely important from a practical point of view, with a very rich physico-chemical behavior. To understand the intermolecular interactions, and the effect of temperature, electrolytes, and other added solutes on these systems, is a very demanding task. Here a broad experimental approach, involving different NMR techniques, scattering methods, microscopy, and ellipsometry turned out to be very powerful.

In more recent years Björn has directed his interest towards the interactions between DNA and small amphiphiles, including the important topic of DNA compaction by surfactants and polymers. In this research Björn has returned to biologically oriented surface chemistry, which was where he once started his research at Lund, as a young lecturer in the group of Professor Sture Forsén.
Lipid self-assembly: Structures and applications

Kåre Larsson
Camurus Lipid Research Foundation, Lund University, Lund, Sweden

I started my Ph D work in 1959 on structure and functionality of glycerides, before the Luzzati and coworkers had reported the fundamental structures of the aqueous liquid-crystalline phases. My approach therefore followed a tradition of that time, to first determine the crystal structures. Monoglyceride crystals were seen to form aqueous phases above a certain temperature, and at cooling a remarkable dispersion of hydrophilic crystals was observed. The applications of such dispersions in foods and in pharmacology then became a side-track which will be described.

Some historic aspects on research of lipid self-assembly will be summerized, with focus on monoglycerides. Phase diagrams of monoglyceride-water systems were reported by Lutton (in 1966 - the long-chain types) and myself (in 1967 - short-chain members). I then worked for several years on the cubic phases, and some steps in this work will be described.

Björn Lindman with Sven Engström introduced the concept Fluid Crystals for drug delivery in 1983, and I was invited to collaborate. A first application involved paradontitis, and was launched on the market in 1994. The first steps in this development which took place in Camurus, Ideon Science Park, will be summerized.

Finally a current collaboration with Björn Lindman will be discussed. It concerns the possibilities to hinder adhesion formation after surgery. The first approach was based on lipid self-assembly, but later we found a more efficient protection-film formed at tissue surfaces by use of polypeptides.
The significance of lipid peroxidation for cardiovascular diseases

Günter Siegel
Charité - Universitätsmedizin Berlin, Campus Benjamin Franklin, Berlin, Germany

Introduction: The metabolic syndrome as sickness entity with its multifacet symptoms is an excellent example for targeting polyorganic impairment such as cardiovascular (cardiac infarction), cerebral (stroke, Alzheimer’s), pancreatic (diabetes), hepatic (cholestasis), and gastrointestinal (irritable bowel syndrome) diseases.

Methods: Photometric methods, ELISAs, EIAs and nanotechnologic biosensor ellipsometry were applied.

Results: A wide spectrum of more than twenty oxidative stress, cytokinic, inflammatory, lipidic and arteriosclerotic biomarkers, all interconnected with each other, and navigated by reactive oxygen species (ROS) serves for a detailed diagnosis and point-of-care therapy monitoring. Special focus is concentrated on the significance of lipid peroxidation for cardiovascular diseases. As one example, correlations between alkaline phosphatase (ALP) and various biomarkers are presented.

In our clinical studies it appeared, that many biomarkers (e.g., hs-CRP, cytokines, liver values) are up- or down-regulated in the subthreshold range, but so much the more have an important early diagnostic, preventive and prognostic significance (Siegel et al.: Cardiovasc. Res. 58 (2003) 696; Biosens. Bioelectron. 24 (2009) 1512; Eng. Life Sci. 9 (2009) 363; Diabetes 60, Suppl. 1 (2011) A626; Am. J. Gastroenterol. 106, Suppl. 2 (2011) S130). The picture is the more complicated in that some inflammatory biomarkers (e.g., TNF-α, TGF-β1) in the sense of a cytokine balance behave inversely.

Conclusion: Preclinical and clinical trials with statins, ginkgo, ω3-fatty acids and blackcurrant extract unravelled this network of biomarker interactions and demonstrated its usefulness for theranostics.
From self-assembly fundamental knowledge to nanomedicine developments

Maura Monduzzi
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The story of a rigorous scientific approach to understand self-assembly in surfactant systems and biological membranes starts in the early seventies when the progresses of SAXRD and NMR technology allowed to demonstrate the existence of ordered soft matter and the validity of Tanford approach. Particularly, NMR relaxation of dipolar and quadrupolar nuclei, NMR chemical shift anisotropy measurements in micellar solutions, microemulsions, and liquid crystals proved the existence of an ordered polar-apolar interface. NMR data, rationalized in terms of the two-step model of relaxation, allowed to quantify the dynamic aspect of the supramolecular aggregates in different soft matter systems. In addition, NMR self-diffusion proved without any doubt the existence of bicontinuous cubic phases, earlier suggested by SAXRD investigations. Analogously NMR self-diffusion played a fundamental role in the understanding of emulsion and microemulsion nanostructure, phase transitions, and particularly percolation phenomena in microemulsions. Since the nineties, globalization of the knowledge along with many other technical facilities such as Electron Microscopy produced huge progresses in surfactant and colloid science. Actually we talk of nanoscience: bottom up/top down strategies allow to build nanodevices with applications spanning from ICT to food technology, whereas progresses in theoretical skills have increased predictive ability, hence more ambitious targets can be planned. Nanomedicine represents a major challenging field with its main aims: targeted drug delivery, diagnostic, theranostics, tissue engineering, personalized medicine. Few recent examples will be mentioned. Although the real applications of these systems still need major work, nevertheless new challenges are open, and perspectives based on integrated multidisciplinary approaches would enable both a deeper basic knowledge and the expected advances in biomedical field.

Surfactant research during the last 30 years

Krister Holmberg
Chalmers University of Technology, Department of Chemical and Biological Engineering, Göteborg, Sweden

Surfactants are used in a very large variety of applications ranging from personal products, such as food and pharmaceuticals, to large scale industrial processes, such as paper-making and mineral ore flotation. Even if the end uses differ, the physical-chemical behavior is basically the same: one takes advantage of the tendency of surfactants to self-assemble in bulk and at interfaces. In order to optimize the performance, surfactants are tailor-made to fit specific applications and the design of the amphiphiles involves variations of both the polar headgroup and the hydrophobic tail. The two main driving forces behind surfactant research during the last decades have been

- to make more environmentally benign surfactants
- to make more efficient surfactants

The environmental aspects are extremely important because surfactants are the largest class of specialty chemicals. The strive towards surfactants more benign to the environment has lead to the development of amphiphiles with built-in weak bonds, so called cleavable surfactants. It has also been one of the driving forces for the development of polymerizable surfactants. Surfactant efficiency is a measure of the concentration required to obtain proper adsorption of a surfactant at an interface. The more efficient the surfactant, the better it is from both an economic and an environmental point of view. Gemini surfactants and polymeric surfactants (surface active polymers) are examples of particularly efficient surfactants. The talk will deal with some of the more important developments in the above mentioned areas.
The Ekwall tradition - past, present and future

Jarl B. Rosenholm
Åbo Akademi University, Laboratory of Physical Chemistry, Åbo, Finland

To a great extent Scandinavia owes its traditions in surface and colloid chemistry to Per Ekwall, former Professor in Chemistry, preferably Physical Chemistry at Åbo Akademi University in Åbo (Turku), Finland. In 1923 he began working on his doctors Thesis on Surface Activity of High-Molecular Fatty Acid Sodium Salt Solutions (in Swedish) which he completed in 1927. He identified the existence of a critical concentration (cmc) above which the soap molecules assembled to colloidal complexes denoted micelles. This observation was made in parallel, but independently of Bury and Hartley. He extended the systematic investigation to phase-equilibria in two- and three-component systems with reference to McBain’s studies on binary Surfactant - Water equilibria. He published a complete analysis of the phase equilibria in the Sodium Caprylate (Octanoate) - Decanol - Water system in 1960. This pioneering work has for long been denoted the “holy system” among Scandinavian scientists. It was later corrected for the semi-stable liquid-cristalline phases B and C being actually parts of the lamellar phase D. He manifested himself as one of the founders of the self-assembled system science by publishing an exceptionally complete set of 60 binary (temperature-composition) and 102 ternary (constant temperature) own and previously published phase equilibrium diagrams in 1975.

He collected the published works on surface activity and self-assembled systems at Åbo Akademi under five topics illustrating his cross-disciplinary interests: 1.) Alkylated ammoniumpicrate (1932-1933); 2.) Structure of soap solutions (1928-1960); 3.) Resin acid and spruce oil research ((1941-1960); 4.) Cancer research (1947-1955); 5.) Hormone research (1949-1955)

After his retirement he was invited in 1963 by the Royal Science Academy of Engineering Sciences to establish a Swedish Laboratory of Surface Chemistry in Stockholm, which later developed into the multi-disciplinary Institute for Surface Chemistry. The research in surface chemistry expanded to applied systems, but were for a long time based on the traditions set at Åbo Akademi. At his 95th birthday he provided another set of published papers which over-bridged the research at Åbo Akademi and the Surface Chemistry Laboratory with the topic: “Solutions of alkali soaps and water in fatty acids (1969-1989)”. The coauthors of the included publications (K. Fontell, S. Friberg, B. Lindman, L. Mandell, P. Solyom) express the widening network induced in Sweden. The long-term relationship between Laboratory of Physical Chemistry at Åbo Akademi and Laboratory of Physical Chemistry at Lund University is explored. The successors at the chair at Åbo Akademi, Professor Ingvar Danielsson and Jarl B. Rosenholm and other senior scientists from Åbo such as Per Stenius, Krister Fontell, Johan Sjöblom and Mika Lindén have all since continued the collaboration with Lund. The collaboration is illustrated with some jointly published research topics. The focus is, of course placed on the jubilar Professor Björn Lindman.
Colloids in Trondheim

Johan Sjöblom
Ugelstad Laboratory, NTNU, Trondheim, Norway

The talk summarizes essential contributions and contributors from NTNU in the field of physical chemistry / colloid chemistry. In physical chemistry / thermodynamics the impact by Prof Lars Onsager will be described. In colloid chemistry / polymer chemistry Prof John Ugelstad research impact within monodisperse polystyrene particles is viewed.

Most of the activities in colloid chemistry at NTNU is per today organised in Ugelstad Laboratory. The activities comprise crude oil processing and transport (Johan Sjöblom, who is also the Head of the Lab), waste water management (Gisle Øye), bionanotechnology (Wilhelm Glomm) and colloid chemistry modelling (Brian Grimes). A brief presentation of the activities within the Ugelstad Lab will be given.
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