

Swiss Soft Days

21st Edition

Lausanne, 8.9.2017

How/when to get there:

Welcome to the 21st Edition of the Swiss Soft Days!

The conference venue is in the room CM 4 of EPFL in Lausanne.

Address

EPFL
1015 Lausanne
Switzerland



The building is easily reachable by public transport from the main train station of Lausanne by taking first the metro M2 from Lausanne gare to Lausanne Flon. There you change and take the metro M1 from Lausanne Flon to EPFL. It is also possible to take the metro M1 from Renens gare to EPFL. From there it is a few minutes walk to the venue.

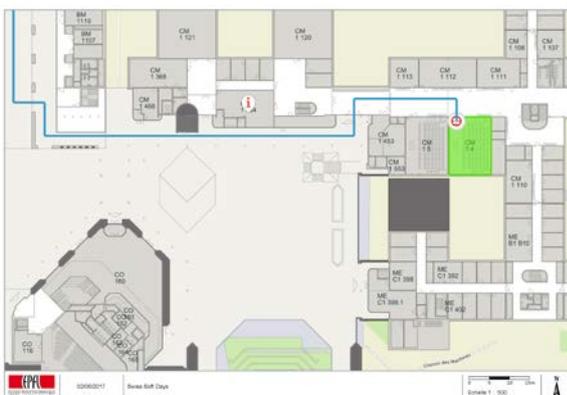
The scientific program (see next page) will start at 10:15 am. Registration and a welcome coffee will be available from 09:30 am.

The program features two keynote speakers (Prof. Costantino Creton from the ESPCI in Paris and Prof. Randy Ewoldt from the University of Illinois) and 11 short talks of 20 minutes each including questions. Time for additional discussions will be available during the lunch break and the poster session.

The poster session will take place between 12:00 and 13:30 in front of the lecture room, together with lunch, in the foyer in front of the lecture room. Poster boards and pins will be provided.

If you have any questions do not hesitate to contact us at swisssoftdays@ethz.ch.

Have a happy meeting!



The organizers,

Karolina Korzeb, Kilian Dietrich Damian Renggli
and Mathias Steinacher

Program

09:30-10:15 Welcome Coffee/Registration

10:15-11:00: Adhesion between polymer gels:
molecular interactions and macroscopic adhesion
(Costantino Creton)

Session 1:

11:00-11:20 Talk 1: Maceroni

11:20-11:40 Talk 2: Crippa

11:40-12:00 Talk 3: Urban

12:00-12:20 Talk 4: Murello

12:20-12:40 Talk 5: Neumann

12:40-14:15 Lunch + Poster Session

14:15-15:00: Sticky droplets and sinking bubbles:
curious physics of yield-stress fluids
(Randy H. Ewoldt)

Session 2:

15:00-15:20 Talk 6: Drab

15:20-15:40 Talk 7: Ortuso

15:40-16:00 Talk 8: Hof

16:00-16:20 Talk 9: Sivak

16:20-16:40 Talk 10: Etienne

16:40-17:00 Talk 11: Malekovic

16:40-17:30 Poster Session

Invited Lecture 1

Adhesion between polymer gels: molecular interactions and macroscopic adhesion

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Introduction

Hydrogels are soft materials, well-known for many potential applications in the food industry, cosmetics or biomedicine. These hydrophilic networks formed by cross-linked polymer chains, are typically highly swollen in aqueous environments. Despite numerous earlier studies, the molecular mechanisms responsible of the adhesive or non-adhesive properties of hydrogels, especially in fully immersed conditions, remain poorly understood. The objective of our study is to investigate and control the adhesive properties under water between a thick (mm) hydrogel and a thin (5-100 nm) polymer brush or gel layer. We will report novel experiments on adhesive properties as a function of immersion time using a newly developed device¹, and discuss hypotheses for the variations observed with gel composition.

The synthesis of hydrogels was carried out at room temperature by free-radical polymerization of monomer in Milli-Q water with N,N'-methylenebisacrylamide (MBA) as cross-linker, using potassium persulfate (KPS) and N,N,N',N'-tetramethylethylenediamine (TEMED) as redox initiators. A variety of neutral and charged monomers were used. The concentration of monomer in water was typically 10-25 wt% while the molar ratio of crosslinker (MBA) to monomer was set at 1-4 mol%. The quantity of initiator was set to 1 mol% of the monomer quantity.

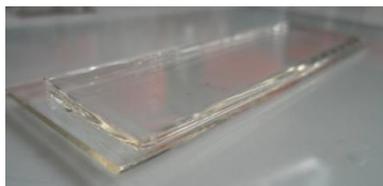


Figure 1. Hydrogel layers after polymerisation, prepared for immersed adhesive tests.

Adhesion tests can be done on samples at different swelling ratios in air and under water, thanks to an original experimental setup (Fig. 2), previously

developed in our group¹. It consists of an immersed flat punch contact test, fitted with a precise control of the alignment between the two surfaces. Contact and debonding tests can be carried out with a precise control of the environment (temperature, pH, composition of the solution) and of the test parameters (velocity, contact time, compression force).

The working principle is represented in Fig. 2: a layer of the soft or swollen sample is attached on the bottom substrate and immersed in water; the modified hard substrate is attached on a mobile punch, which is moved down to come in contact with the soft material. If properly aligned, the two materials come in contact, with a contact area equal to the area of the hard material. After the preset experimental conditions have been achieved (e.g. contact stress and contact time), the probe can be pulled off at a constant velocity. During the experiment, the displacement of the punch and the force applied on it are measured as a function of time.

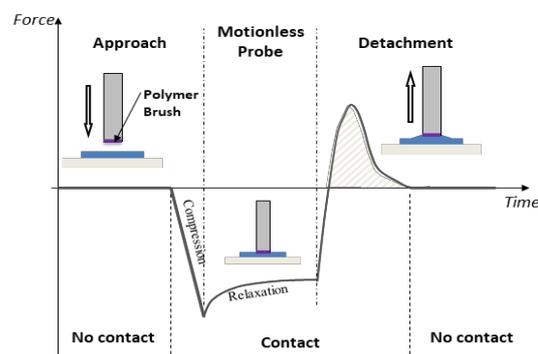


Figure 2. Principle of flat-flat contact test between a hydrogel and a polymer brush.

References:

1. G. Sudre and C. Creton, *Soft Matter*, 2012, **8**, pp 8184–8193.
2. G. Sudre and Y. Tran, *Macromol. Chem. Phys.*, 2012, **213**, pp 293-300.
3. G. Sudre and Y. Tran, *Langmuir*, 2012, **28**, pp 12282–12287.

Invited Lecture 2

Sticky droplets and sinking bubbles: curious physics of yield-stress fluids

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Introduction: Fluids should flow, and bubbles should rise, but we consider phenomena that violate such intuition. The phenomena motivate several applications (fire suppression, spray coating, high performance concrete) and are possible due to a class of soft materials known as yield-stress fluids. Examples include gels, pastes, biopolymer hydrogels, fresh concrete, and colloidal suspensions, which reversibly transition from fluid at high stress to solid at low stress. This talk will discuss our work on droplet impacts (splashing, spreading, sticking) of these non-Newtonian fluids (Fig. 1) [1,2], as well as efforts to design these materials (Fig. 2) [3] and use them for peculiar behavior, such as making bubbles sink (Fig. 3) [4].

Methods: Research approaches include high-speed video, rheological characterization, structure-properties modeling, and fluid dynamics scaling analysis.

Results: Two new dimensionless groups will be described that rationalize the phenomena. For drop impact and splashing of yield-stress fluids, we will show that

$$\Pi = \frac{\rho V^2}{\sigma_y + \eta_\infty V/t} \frac{D}{t} \quad (1)$$

can be used to create simple regime maps of sticking and splashing. For gravity-opposed motion of particles/bubbles, we will introduce the doubly-modified yield-gravity parameter

$$Y_g'' = \frac{\sigma_y(t^*)}{\Delta\rho|g + A|D}. \quad (2)$$



Fig. 1: Shooting yield-stress fluids at screens. At slower speeds, drops cannot penetrate, and the open screen seems closed.

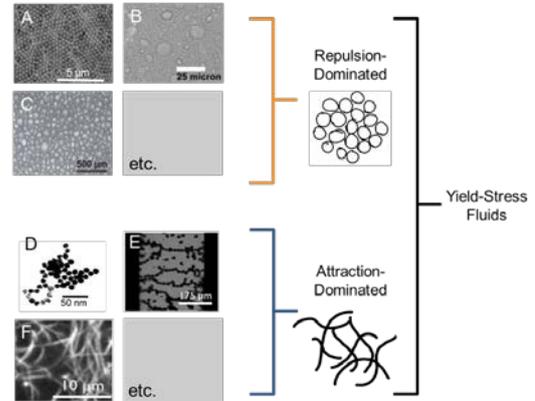


Fig. 2: How many ways to engineer a yield stress fluid? To exploit these nonlinear properties requires design at the structure-rheology level. (adapted from Nelson & Ewoldt [3])

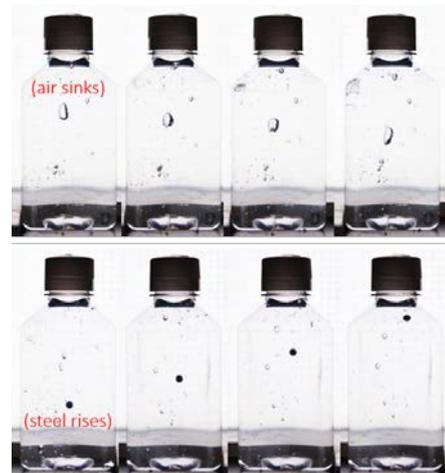


Fig. 3: Bubbles should rise, and higher density steel should sink, but here we cause the opposite in yield-stress fluids using asymmetric accelerations.

References:

- [1] B. C. Blackwell, M. E. Deetjen, J. E. Gaudio and R. H. Ewoldt, *Phys. Fluids*, **2015**, 27, 43101.
- [2] B. C. Blackwell, A. E. Nadhan and R. H. Ewoldt, *J. Nonnewton. Fluid Mech.*, **2016**, 238, 107–114.
- [3] A. Z. Nelson and R. H. Ewoldt, *Submitted*.
- [4] J. A. Koch and R. H. Ewoldt, *In Prep*.

Acknowledgements: This work was supported by the National Science Foundation under CAREER award Grant No. CBET-1351342 and Grant No. CMMI-1463203.

Talks

Characterization and Quantification of SiO₂ Nanoparticle Dissolution in Aqueous Environmental Matrices

M. Maceroni^{1,2}, D. Bossert^{1,2}, L. Rodriguez-Lorenzo^{1,2}, D.A. Urban^{1,2}, A. Petri-Fink^{1,2}, B. Rothen-Rutishauser^{1,2}, F. Schwab^{1,2*}

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Introduction: Silica nanoparticles (SiO₂-NPs) are ubiquitously used in a broad range of industrial applications, such as food, cosmetics and paints. The use of SiO₂ as a delivery vehicle for molecules in plants has been investigated in several recent studies [1]. This could be interesting in order to create nano-agrochemicals, provided that such SiO₂ nanomaterials degrade easily [2]. Different parameters can influence the SiO₂-NP dissolution in aqueous solutions, or the degradation in biological/environmental media. Particle shape and size, porosity, pores size, morphology, and surface functionalization as well as mucilage and exudates of organisms can govern the dissolution/degradation rates [3].

Methods: The dissolution of SiO₂-NPs was investigated in three increasingly complex media (Milli-Q water, KCl solution, soil solution). The particles, synthesized by Stöber's reaction and 70.4 ± 6.9 nm in diameter, were suspended and the free Si dissolved from the NPs was observed in the NP suspension filtrates (30kDa molecular weight cutoff).

Inductively coupled plasma – optical emission spectroscopy (ICP-OES) Si quantification was performed with a Perkin Elmer Avio® 200.

For a more in-depth analysis of the changes in particle shape and size, transmission electron microscope (TEM) imaging was carried out on a FEI Tecnai Spirit. Particle analysis was performed using the software ImageJ.

Dynamic light scattering (DLS) experiments were performed to determine the hydrodynamic radius and surface charge using a Brookhaven 90 Plus Particle Size Analyzer.

Results: We will present initial results of this experimental multidisciplinary approach to characterize and quantify the dissolution of SiO₂ NPs in media with increasing complexity, from

simple aqueous solution to the highly complex soil pore water matrix.

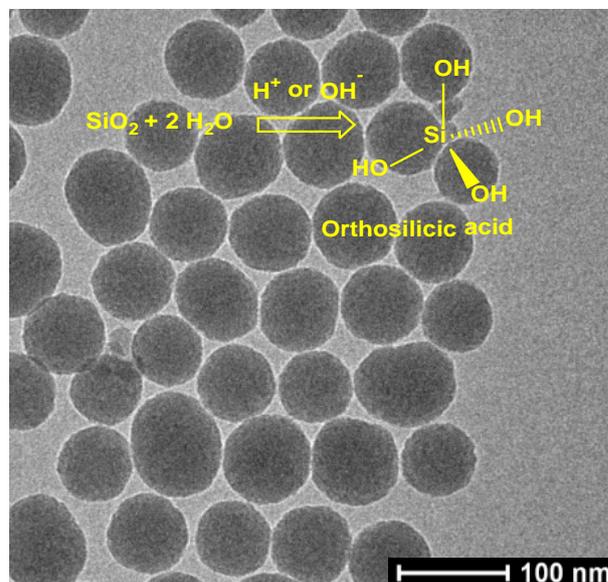


Fig. 1: TEM image of 70.4 ± 6.9 nm SiO₂-NPs synthesized by Stöber method and purified by dialysis

Discussion & Conclusion: Initial results suggest that SiO₂-NP dissolution occurs and is dependent on the background matrix. These findings provide knowledge to understand the environmental behavior of SiO₂-NPs in soil, and eventually the risks and potential applications of SiO₂-NPs in the soil environment.

References:

- [1] H.I. Hussain et al.; **2013**; *J. Nanopart. Res.*; 15:1676.
- [2] F. Schwab et al.; **2016**; *Nanotoxicology*; 10:257-278.
- [3] J.G. Croissant et al.; **2017**; *Adv. Mater.*; 29:1604634.

Acknowledgements: This work was supported by the Swiss National Science Foundation (project 168187).

Lock-in thermography: a new method for magnetic nanoparticles and the quantification of heat

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¹ Adolphe Merkle Institute, Fribourg, Switzerland. ² Zurich University of Applied Sciences, Winterthur, Switzerland, ³ University of Fribourg, Fribourg, Switzerland.

Introduction: Magnetic nanoparticles (MNPs) and their ability to convert magnetic energy into heat are of explicit interest for several applications in biomedicine, in particular magnetic hyperthermia. The heating power of MNPs depends on their physico-chemical properties (e.g. size, colloidal stability and crystallinity) and external parameters such as magnetic field strength and frequency. However, precise evaluation of their heating power is non-trivial with conventional methods (e.g. fiberoptic cables, thermocouples and IR cameras) thus rendering comparative studies challenging.

Methods: We propose a new method based on lock-in thermography to quantify the heating power of MNPs in a precise and reliable way. Nanoparticle suspensions are exposed to a modulated alternating magnetic field and their thermal signature is recorded with a synchronized IR camera. The signal is then processed and can be transformed into the heating slope [1,2].

Results: Lock-in based analysis of the heating properties of MNPs has been particularly useful to:

- 1) Quantify their heating power in suspension, semi-solid and aggregate states in particular to screen the thermal properties of nanoparticles with different sizes, crystallinities, coatings and stability [1].
- 2) Investigate sources of variability and errors related to the alternating magnetic field (e.g. field strength inhomogeneity and its effects on the heating power).

Discussion & Conclusion: Overall lock-in thermography is a new approach to precisely screen and analyse the heating power of different MNPs samples, facilitating comparative studies and the translation of these materials to the clinic.

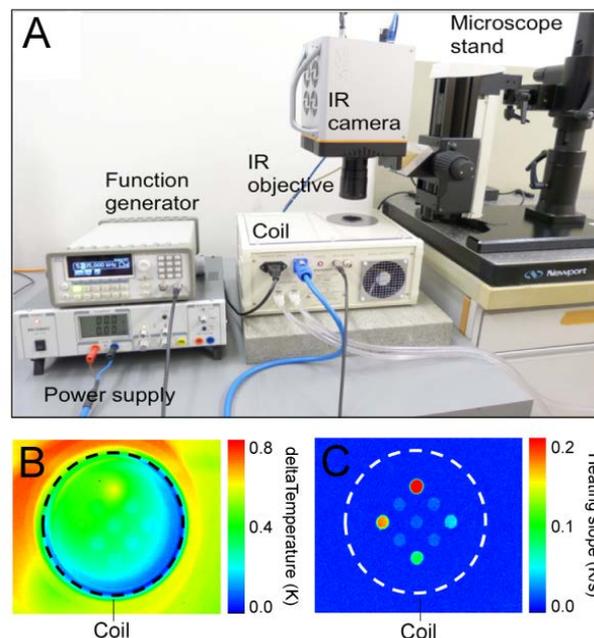


Fig. 1: Lock-in thermography system for the quantification of heat generated by magnetic nanoparticles (A). Comparison between the sensitivity of a normal IR camera (B) and lock-in thermography (C): The temperature variations of MNP suspensions that are recorded by a normal IR camera (B) have a low resolution. The thermal signature of the same samples analysed by lock-in thermography (C) allows an immediate and clear quantification of the MNPs heating properties.

References:

- [1] C.A. Monnier; **2016**; *Nanoscale*; 8: 13321-13332.
- [2] P. Lemal; **2017**; *Journal of Magnetic Materials and Magnetism*; 427: 206-211.

Acknowledgements: This work was supported by the Swiss National Science Foundation (159803 and PP00P2_159258), the National Center of Competence in Research Bio-Inspired Materials, the Commission for Technology and Innovation (Project 18083.1 PFNM-NM), the Adolphe Merkle Foundation, the University of Fribourg and the Zurich University of Applied Sciences (ZHAW).

Taylor Dispersion Analysis: Extending the boundaries of nanoparticle characterization

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S. Balog, B. Rothen-Rutishauser, A. Petri-Fink

Adolphe Merkle Institute, University of Fribourg, Fribourg, Switzerland

The characterization of nanoparticles has always proven to be a challenge, due to the manifold of core materials, shapes, sizes, and consequent issues these have on analytical techniques. Due to the rising interest to use nanoparticles in, for example medical applications (e.g. drug delivery systems, sensors), it is imperative to find instrumentations to cope with accurately characterizing nanoparticles in such complex environments, such as blood or cell culture media. In this project we show the capabilities of Taylor Dispersion Analysis to accurately measure hydrodynamic sizes of various types of nanoparticles, and the advantages this method has over conventional sizing techniques [1].

Taylor Dispersion Analysis (TDA) was introduced in the 1950s, but only in recent years has it been discovered to have potential to characterize nanoparticle size in solution. In short, Taylor Dispersion Analysis is an analytical method to determine the diffusion coefficient of small molecules, proteins, or nanoparticles under flow conditions (Figure 1) [2].

In this project we expand upon the potential of TDA by measuring various particle materials, including proteins, silica, gold and iron oxide nanoparticles. Furthermore, we show the advantages that this technique has over alternative measurement techniques, such as dynamic light scattering and electron microscopy. Among the advantages are the minimal use of sample volume, detection possibilities (absorbance, scattering), wide range of particle sizes that can be measured, and solvents that particles can be measured in. We further demonstrate the future potentials that this system has to offer for more complex nanoparticle characterization. We could show, for the multitude of samples that were measured, that we could reliably acquire sizes for all materials. Furthermore, we could show that particles as small as 4 nm in diameter could be measured via TDA. We extend the study by showing that even

very polydisperse samples from industrial applications could be characterized to an extent, and that measuring nanoparticles in complex environments, such as complete cell culture media, is possible. In conclusion we could show that TDA could be a valuable standard tool for nanoparticle characterization in the future.

Keywords: Taylor Dispersion Analysis, nanoparticle characterization, hydrodynamic size, dynamic light scattering, electron microscopy, proteins.

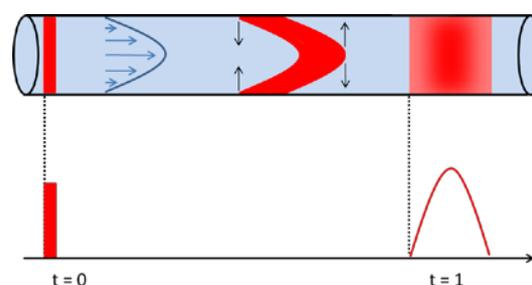


Figure 1: Schematic illustrating the principle of Taylor Dispersion Analysis [2]. A nanoparticle sample introduced with a pressure driven flow into a narrow bore fused silica capillary, will be affected by convection and subsequently increased radial diffusion, causing a band broadening effect, which in turn can be measured and provides values for the particles diffusion coefficient and hydrodynamic size.

References:

- [1] Lopez-Serrano (2014) Nanoparticles a global vision. Characterization, separation, and quantification methods. Potential environmental and health impact. *Anal. Methods*, 6, 38-56.
- [2] Alizadeh, A. (1980) The Theory of the Taylor Dispersion Technique for Liquid Diffusivity Measurements. *International Journal of Thermophysics*, 1, 3, 243-284.

Wetting Behaviour on Heterogeneous Surfaces at the Nanoscale

A.Murello^{1*}, Z.Luo^{1*}, D.M.Wilkins¹, F.Kovacik¹, J.Kohlbrecher², A.Radulescu³,
N.Nianias¹, H.I.Okur¹, N.Smolentsev¹, Q.Ong¹, M.Ceriotti¹, S.Roke¹,
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Introduction: The surface of biomolecules is composed of multiple domains at the nanometre-scale and characterized by a different degree of affinity for water. Despite the well-known presence of these domains a quantitative understanding of how nanostructures influence interfacial energy is lacking.

Traditional thermodynamics treats the interfacial energy solely as a function of composition, but it has been shown experimentally and computationally that the structure of the domains plays an important role in its determination [1-3]. A macroscopic, averaged, description of a surface is, thus, not sufficient to describe its wetting behavior. Furthermore, it has been shown that hydrophobic interactions are modulated by proximal ions, demonstrating that hydrophobicity is not an intrinsic property of any given non-polar domain, but that it is strongly modulated by functional groups as far away as one nanometer [4].

Methods: We use a model system to demonstrate that the morphology of the domains plays a role in the interfacial energy. It consists of a set of two kinds of gold nanoparticles with the same core size, both coated with the same pair of ligands in the same ratio, but with different size and shape of the domains.

Small Amplitude Modulation Atomic Force Microscopy (SAM-AFM) is used to locally quantify the work of adhesion, with the advantage that inhomogeneities on the nanometre-scale are easily discriminated. Sum Frequency Generation (SFG) allows for the recognition of different hydrogen bonding networks around the nanoparticles. Numerical simulations allow to have a straightforward interpretation of experimental data.

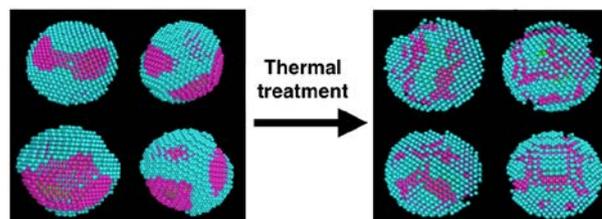


Fig. 1: Change of the size and shape of the domains, due to thermal treatment, as measured by fitting Small Angle Neutron Scattering data.

Results: SFG measurements reveal that the hydrogen bonding network is weaker around the annealed nanoparticles. Nanoparticles are more hydrophobic after annealing, as measured by SAM-AFM. Furthermore, the water density on the first hydration layers appears less homogeneous after annealing. Numerical simulations reveal that the interfaces between the domains have a huge effect on the water structure, influencing both the hydrogen bonding network and the hydrophobicity of the nanoparticles.

Conclusion: We present experimental results on how nanoscale heterogeneity of the surface, on top of other parameters, affects the structure of interfacial water. Two surfaces differing only in the size of the domains (from around 1.2 nm to >2 nm) show significantly different hydration behavior.

References:

- [1] Centrone *et al.*; **2008**; *PNAS*; 105:9886-9891.
- [2] Kuna *et al.*; **2009**; *Nat. Mat.*; 8:837-842.
- [3] Giovambattista *et al.*; **2007**; *J. Phys. Chem. C*; 111:1323-1332.
- [4] Ma *et al.*; **2015**; *Nature*; 517:347-350.

Mechanically Unravelling Metallosupramolecular Polymers

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S. Balog¹, D. Urban¹, M. Borkovec², C. Weder¹

¹Adolphe Merkle Institute, University of Fribourg, Fribourg, Switzerland. ²University of Geneva, Sciences II, Geneva, Switzerland.

Introduction: Directional supramolecular interactions can form non-covalent bonds, which play an important role in many biomimetic materials [1]. Accordingly, an improved understanding of these interactions and the influence of different stimuli is desirable. Metal-ligand (ML) complexes are of particular interest, because their interaction strength and dynamic nature can be tuned by simple variation of the metal ions, ligands, or counter-ions [2]. To explore the response of different ML complexes to the application of mechanical force, single molecule force spectroscopy (SMFS) was employed. This approach provided insights into the mechanically induced scission and reversible formation of the ML complexes.

Methods: A side-chain functionalized polymer was prepared by RAFT-mediated copolymerization of butyl acrylate and an acrylic monomer carrying the methylbenzimidazolyl pyridine ligand. The copolymer with 5 mol% pendant ligands was collapsed into SCPNs by intramolecular complex formation that occurred upon slow addition to dilute solutions of metal ions (Fe^{2+} , Zn^{2+} , Eu^{3+}). The particles were thoroughly characterized, and forces associated with the dissociation of the ML complexes were determined using SMFS by means of atomic force microscopy (AFM).

Results: Successful formation of SCPNs with various metals (Fe^{2+} , Zn^{2+} , and Eu^{3+}) was demonstrated and the particles were characterized by means of dynamic light scattering, AFM, and Taylor dispersion measurements.

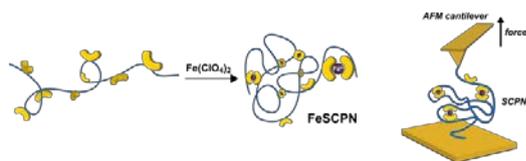


Fig. 1: Formation of single chain polymeric nanoparticles (SCPNs) (left). Single molecule force spectroscopy using SCPNs (right).

The forced unfolding of nanometer-sized SCPNs by means of SMFS was used to investigate the complex scission events under mechanical stress at the molecular level. The dissociation of ML complexes is reflected in the extension profiles by

discrete transitions prior to detachment of the polymer chain from the substrate or tip. Fitting of the extension profiles to the worm-like chain (WLC) model of elasticity [4] yields the persistence length $\ell_p = 0.72 \pm 0.04$ nm and force constant $K = 54.7 \pm 0.5$ nN of the polymer, as well as the rupture forces for the different ML complexes: $F_r = 165 \pm 53$ pN (FeSCPn), $F_r = 138 \pm 38$ pN (ZnSCPn), and $F_r = 73 \pm 19$ pN as well as 159 ± 31 pN (EuSCPn).

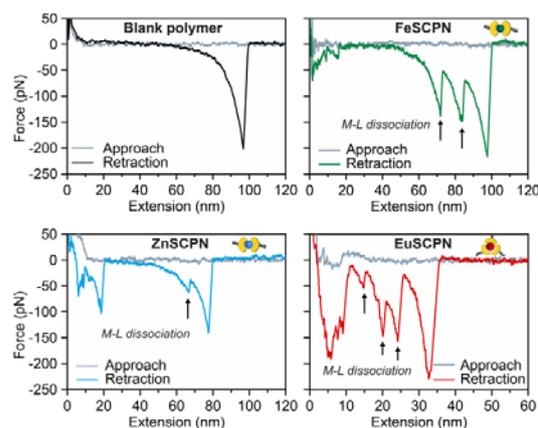


Fig. 2: Force extension profiles for the blank polymer, FeSCPn , ZnSCPn , and EuSCPn show M-L dissociation when ML complexes are present.

Discussion & Conclusion: This approach permits an analysis of the binding parameters of different bi- and tri-fold coordinated ML complexes. The strongest Fe^{2+} complexes show the largest rupture force, whereas for the trifold coordinating Eu^{3+} a clear difference between the dissociation of the weakest coordinating 3rd ligand and the 2nd ligand could be observed. Advancing our understanding of the nanomechanical properties of these ML complexes is an important step towards the development of novel biomimetic- and mechanoresponsive materials.

References:

- [1] M.J. Webber, E.A. Appel, E.W. Meijer, R. Langer; **2016**; *Nat. Mater.*; 15:13-26.
- [2] A. Winter, U.S. Schubert; **2016**; *Chem. Soc. Rev.*; **2016**, 45: 5311-5357.
- [3] F. Wang, H. Pu, M. Jin, D. Wan; **2016**; *Macromol. Rapid Commun.*; 37:330-336.
- [4] M.D. Wang, H. Yin, R. Landick, J. Gelles, S.M. Block; **1997**; *Biophys J.*; 72:1335-1346.

Mechanism of the cooperativity in antimicrobial peptides

E.Drab, K.Sugihara

University of Geneva, Geneva, Switzerland

Introduction: In this study we aim to elucidate the fundamental mode of action of antimicrobial cooperativity.

To investigate the cooperativity between two different human peptides defensin and cathelicidin we synthesized artificial lipid bilayer onto highly doped p-type silicon electrode covered by naturally formed silicon dioxide as a clean platform to separate peptide-peptide and peptides-bilayer interactions from influence of other components within cell.

Lipid bilayer was obtained by fusion of unilamellar vesicles from buffer solution and investigated mainly by impedance spectroscopy which is non-destructive and sensitive technique.

In addition we performed Fluorescence Recovery After Photobleaching (FRAP) measurements as a complementary technique to confirm the bilayer formation by its recovery in photobleached region.

To elucidate how peptides influence on the lipid bilayer we injected LL37 and α -defensin in micro molar concentration.

Next step in our investigation is to inject mixture of cathelicidins and defensins and elucidate what is the mechanism of cooperativity of two different peptides.

Methods: In this project we use POPC lipids for bilayer formation by vesicles fusion and NBDPE as a fluorescent agent. The bilayer is formed on highly doped p-type silicon/silicon dioxide wafer with activated surface. For electrochemical properties we use impedance spectroscopy with 3-electrodes set up and fluorescent microscopy to confirm and measure bilayer recovery after photobleaching.

Results: Figure 1 shows impedance spectra before (blue line) and after (red line at highest frequency) adding the vesicle solution. This small shift in impedance indicates bilayer formation as it acts as a very good natural insulator. Two red lines at lower frequencies correspond to changes in electrical properties of bilayer after peptide injection. The obtained spectra were interpreted in terms of equivalent R(RCRC) circuit consisting of simple electrical elements.

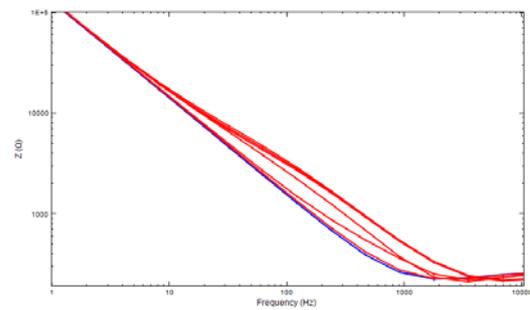


Fig. 1: Impedance spectra of the supported lipid bilayer. Blue line represents Si wafer with HEPES buffer before adding vesicle solution and red lines correspond to changes in electrical properties of bilayer after LL37 peptides injection

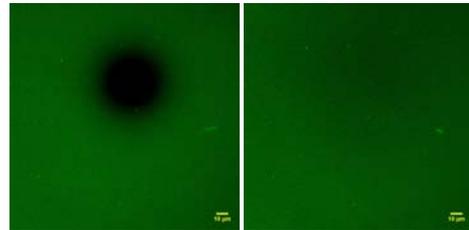


Fig.2: FRAP images of POPC bilayer photobleached region and its recovery within 320s.

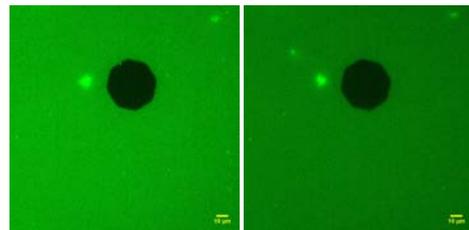


Fig.3: FRAP images of POPC bilayer photobleached region after adding peptide.

Discussion & Conclusion: After LL37 peptide injection we observed drop in resistance and capacitance as bilayer's permanence is disturbed. Similar changes in bilayer resistance were observed for α -defensin, but interestingly, no significant changes in capacitance were detected in this case. After investigating peptide interaction, we found for the first time negative cooperativity of LL37/ α defensin mixture on POPC lipid membrane which proves peptides affinity limited to pure lipid component of bilayer.

The milestone goal is to quantify the antimicrobial peptides cooperativity effect on the bilayer resistance and capacitance.

Mechanosensitivity of Polydiacetylene with a Phosphocholine Headgroup

R. D. Ortuso, R. Sugihara

University of Geneva, Physical Chemistry Dept., Geneva, Switzerland.

Membrane forces play pivotal roles in numerous physiological processes such as endocytosis, cell mutations and calcium signaling.

Currently used characterization methods such as atomic force microscope (AFM) or optical tweezers allow for the controlled force application but not for the detection of the forces applied to the bilayers. Micro- aspiration of giant unilamellar vesicles (GUV) enables the quantification of surface tension, however, its conversion into local forces is difficult.

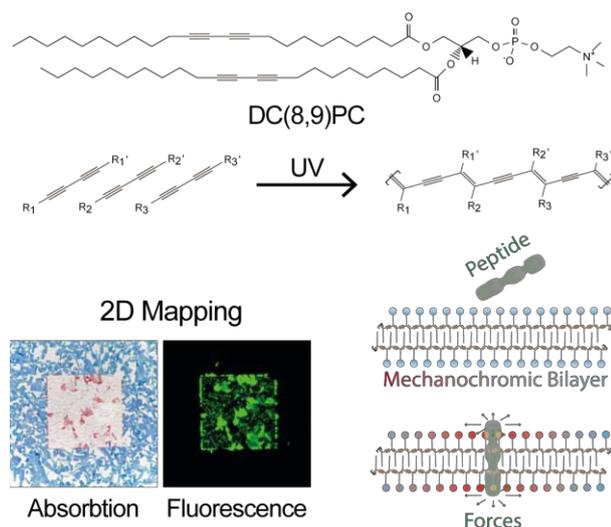


Figure 1: The chemical structure of DiynePC and polydiacetylene. Confocal microscopy image; outer part of the square blue state PDA, inner part red state PDA. The scheme shows the concept of the membrane force sensor we are developing.

We are developing a mechanosensitive membrane probe that enables direct measurement of molecular forces applied within lipid bilayers. We employ a mechanochromic polymer polydiacetylene (PDA, Figure 1) that changes its color and fluorescence intensity upon application of forces (Figure 2B). As a first step, we demonstrated a PDA vesicle assay that detects melittin, a transmembrane peptide from bee venom. First we fabricated PDA vesicles by self-assembling the lipid monomer and subsequently crosslinking by UV. Figure 2B shows the absorption spectra before (blue) and right after (red) addition of melittin to the PDA vesicle solution. These spectra overlap, suggesting no colorimetric response due to the addition of melittin. However, we found that the spectra shifted over the next several hours (Figure 2B). After 4 hours, the shape of the spectra changed completely, as the color change was clearly visible also by naked eyes (Figure 2A). The slow

response from our PDA sensor against melittin is different from what has been reported with other types of PDA sensors [1,2].

A Appearance of Vesicle Suspension

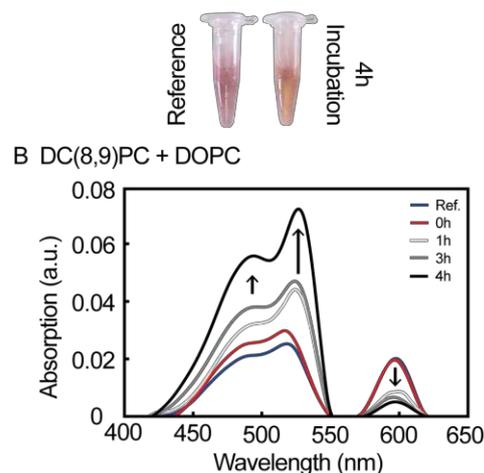


Figure 2: A. PDA vesicle samples before and four hour after addition of melittin. B. UV-Vis spectra before and after the addition of melittin. A spectra shift was observed depending on the incubation time.

In this work, we further analyze the unexpectedly slow interaction kinetics, fluorescence spectra of PDAs, and discuss possible origin of the slow kinetics [3].

The developed assay proved that DiynePC PDA is sensitive to mechanical stimuli. The advantage of our PDA sensor is that it has a phosphocholine head group that mimics cell membranes. Its combination of sensitivity and the biocompatibility will allow us to incorporate this probe in live cells in future, providing us a further possibility to investigate force distribution during mitosis, cell migration, and interactions with medical implants etc.

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Acknowledgements:

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Programming hydrogel properties with DNA

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Introduction: Physical hydrogels have attracted great interest as promising platform for 3D cell scaffolds [1]. To be compatible with the ever-changing cell culture development, these biomaterials need to be adaptive while maintaining a long-term stability. Upon bio-functionalization, hydrogels are attractive candidates for the engineering of artificial extracellular matrices (ECM), due to their high water content, compliant elasticity and facile diffusion of bio-macromolecules. Nevertheless, limitations of many hydrogel systems include a too narrow pore size that prevents cell mobility and expansion, and a stiffness that does not match the biological relevant range [2]. An enhanced, and ideally programmable, control over emergent material properties in polymer hydrogels is required.

Methods: Introducing DNA as programmable cross-linkers for polymer materials, we aim to achieve an unmet level of control in fine-tuning of material properties. The strength of the cross-links is related to the binding strength between the complementary DNA strands, which is directly related to the number of nucleotides and GC content in the sequence. This programmable combination of parameters allows for a unique fine-tuning of material properties. Aiming to assemble hydrogels that can serve as cell matrix, the range of material stiffness should include the natural scenario, ~500-1000 Pa [2].

Stimuli responsive behavior can be programmed to reversibly or irreversibly alter materials properties in time. pH-sensitivity can be included using the i-motif DNA sequence [3], a cytosine rich sequence that upon a slight decrease in pH (<6), transforms from a double helix to a single stranded secondary structure stabilized by intercalated hemi-protonated C-C bridges. When this sequence is programmed as the cross-linking moiety, a reversible gel-sol transition will take place upon slight variation in pH. Furthermore, restriction enzyme recognition sites can be included to selectively degrade crosslinks. Thirdly, when using sequences with a low melting temperature, crosslinking can be broken up through a slight raise in temperature. Interactions can be weakened and programmed to release in a tight transition window between physiologically compatible 35°C and 45°C.

Results: DNA-amine and polymer-SH were coupled using the SMCC heterobifunctional x-linker. First, the DNA was reacted to the NHS-ester, followed by EtOH precipitation. In a second step, the polymer was coupled to the maleimide in 1:1 stoichiometry. Currently, we are verifying the reaction yield and purity, and we will characterize the obtained materials by DLS, size exclusion, and NMR. Hydrogels will form upon combination of complementary-DNA functionalized polymers and subsequently material characterization with rheology will set the baseline properties for further engineering and fine tuning.

Discussion & Conclusion: After the initial combination of our polymers, we will be able to make an educated guess on how to change the DNA cross-linking sequence to alter material properties. Future work includes the bio-functionalization of the DNA cross-links with peptides or proteins that can be used to mimic the natural ECM to create programmable cell niches.

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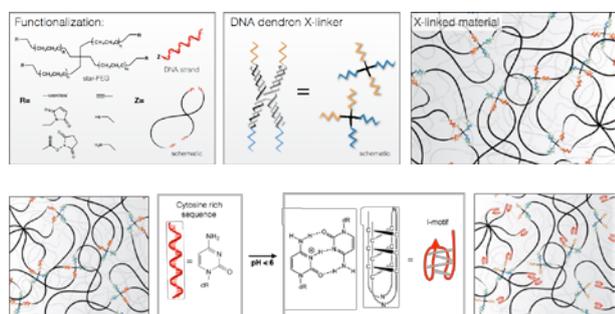


Fig. 1: (top) DNA end functionalization of branched PEG polymers through orthogonal conjugation chemistry. Both linear and branched (depicted) monomers will be used and are x-linked through complementary DNA dendrons into a hydrogel network. (bottom) Formation of i-motif DNA through cytosine protonation in acidic environment. (left) cross-linked gel at neutral pH transfers to a viscous solution (right) at acidic pH when i-motif sequences are present in the DNA cross-linking region

Straight to oscillating transition in fracture path of thin sheets

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³Universidad de Santiago de Chile, Santiago, Chile

Introduction: Oscillating cracks were observed [1, 2] by pushing a blunt tool at a notched thin polypropylene sheet clamped on the sides (Fig. 1).

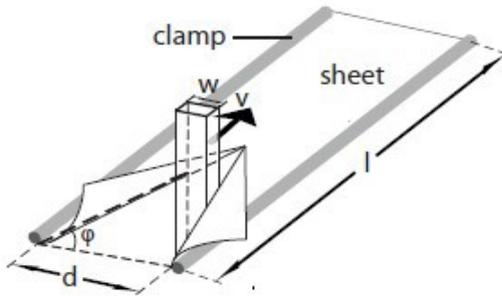


Fig. 1: Experimental setup.

But what happened to the straight solution in the symmetric setup? Is it unstable? Could one ever see it?

Methods:

The instability of a straight crack that propagates along a sheet by pushing a blunt object is investigated experimentally, theoretically and numerically.

Results:

The path turned out to be straight at the beginning, but there is a transition where it starts to oscillate (typical fracture path is shown on Fig. 2).



Fig. 2: Typical fracture path.

We look at a simple geometric model and define a control parameter for the instability, which is the angle between furthest clamping points and the tool ϕ (see Fig. 1). We find that there is a critical angle after which the oscillating path is observed.

Experiments with constant angle ϕ and simple geometric model [2-4] that captures the instability are shown on Fig. 3.

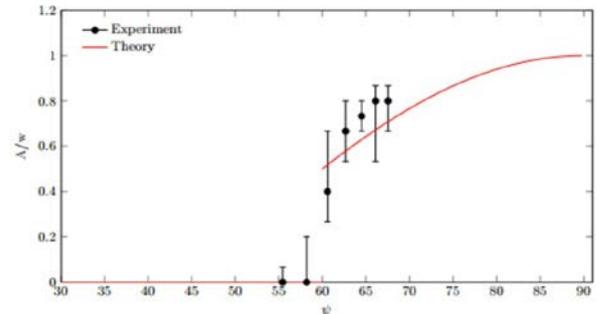


Fig. 3: Comparison of experimental and numerical results for amplitude of the oscillations.

Discussion & Conclusion:

Our results bring some insight for the mechanics and metrology of ultra-thin films as in this case the tool is always blunt compare to the thickness of the sheet. Our findings suggest that by controlling the geometry one can avoid the oscillatory instability and show how a simple geometric model can capture the instability.

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Acknowledgements:

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IS is grateful to Erasmus Mundus for funding her masters in Complex Systems Science. EH thanks Fondecyt 1140225 project, BR thanks ECOS-CONICYT C12E07.

Influence of Fluorinated Surfactants on the Leakage from Double Emulsions

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Introduction: Emulsion drops formed by microfluidics can for example be used as reaction vessels to perform high throughput biochemical screening assays. The accuracy of these assays depends on the stability of each drop. In addition, it relies on the fact that reagents are not exchanged between different drops. The most common systems used to perform biochemical screening assays are aqueous drops dispersed in a fluorinated oil. These single emulsion drops have a limited stability if loaded with salt-containing solution. Moreover, they are affected by exchanges of reagents between drops even though the reagents have no solubility in the oil phase. To address these shortcomings, we use water-oil-water double emulsion drops. We investigate the influence of the surfactant concentration and composition on the stability and permeability of these drops.

Methods: We produced water in oil in water double emulsions using a microfluidic PDMS device as shown in Figure 1. The stability of the drops is characterized with optical microscopy, the permeability with fluorescence microscopy.

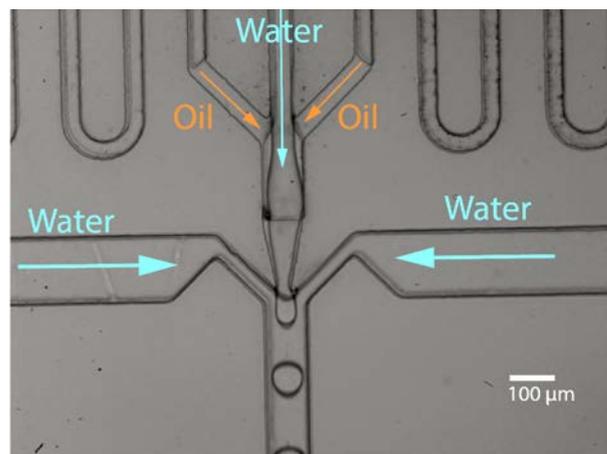


Fig. 1: Microscope image of a PDMS microfluidic device producing water in oil in water double emulsions.

Results: Fluorescein encapsulated in double emulsions stabilized by 1 mM surfactants leak within 5 hours. The permeability of these double emulsions significantly decreases with decreasing surfactant concentration.

Discussion & Conclusion: Although the fluorescent dye is not soluble in the oil shell of the double emulsion, double emulsions are highly permeable to this dye. As recently suggested, these types of surfactants can form aggregates that transport the fluorescent dye between single emulsion drops [1]. Our results indicate that such aggregates also form in double emulsion shells, rendering them highly permeable to hydrophilic encapsulants.

References:

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Nature inspired distributed Bragg reflectors with controlled order and disorder

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Introduction: Colours in nature originate from absorption of dyes or from interference of light with nanostructured materials. In many cases, the structure of the materials is not perfect and introducing disorder results in additional photonic effects. How are the optical properties influenced by disorder? Can we replicate natural photonic structures with a controlled degree of disorder? These are two of the questions we are currently trying to solve. We here systematically investigate colour change of materials with relation to (i) the numbers of layers, (ii) the various refractive index profiles and (iii) thickness. Stacking multiple layers and introducing disorder gives rise to new optical properties.

Methods: Pronounced structural coloration can be adjusted by varying the refractive index n , the thickness t and the number of alternating layers [1]. Our group has established the protocol for producing alternating layers of varying refractive indices by self-assembly of poly(isoprene-*block*-ethylene oxide) block copolymers and TiO₂ (Fig. 1) [2]. The process is quite generalizable, e.g. for other metals such as SiO₂ and can be repeated to form a DBR (Fig. 1c).

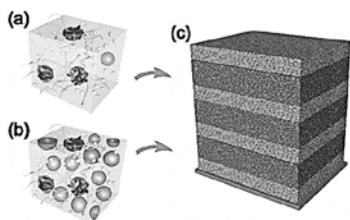


Fig. 1: Schematic representation of the fabrication pathway

Results: We have deposited several layers of alternating porous materials, TiO₂ and SiO₂, respectively. Up to six layers were achieved, each layer having different thickness and refractive index. Several samples with different layer numbers were successfully manufactured. The results of experiment and model agree well. Reflectance peaks of manufactured disordered DBR's are slightly blue shifted, which can be attributed to mismatches between simulation and measurement on spin-coated layers (refractive index, thickness, quality of each layer, Fig. 2).

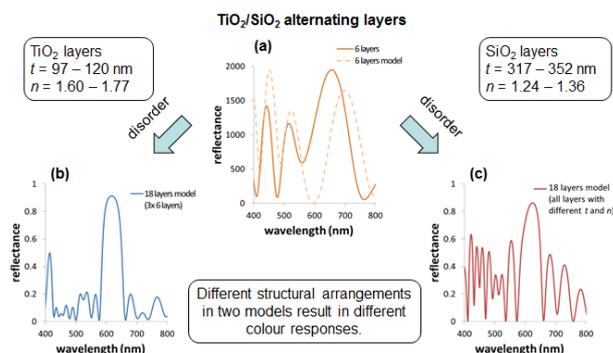


Fig. 2: a) Measured reflectance on six layers and comparison to the model. Simulated optical response of disordered Bragg reflector: b) constructed of three repeating series of six layers, each having different refractive index and thickness. c) constructed of 18 layers with each having different refractive index and thickness.

Discussion & Conclusion: We show that DBRs with different numbers of layers and various refractive indices and thicknesses show a strong colour change. Stacking more layers in the same manner gives optical properties which are a result of disorder introduced into the reflector's structure (Fig. 2b). This signature is reminiscent of the optical response observed in living organisms whose structures are not perfectly aligned (e.g. in *Morpho* butterflies). Here, the disorder is not complete (which would ultimately result in broadband reflectance, Fig 2c), but small deviations in the structure have a large influence to the colour and still have narrowband response. Manufacturing materials with such a designated reflectance will open new fields in photonic-interested areas, for example, by being employed as optical sensors or structural coloured dyes for use in fashion, cosmetics and art.

References:

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Acknowledgements: Funding by the NCCR *Bio-inspired Materials* is gratefully acknowledged.

Posters

Nanomechanical Characterization of Soft Matter Using Bimodal Atomic Force Microscopy

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EPF Lausanne, Lausanne, Switzerland.

Introduction: As technology advances, thin multilayer materials are becoming more and more attractive for a wide range of industrial applications. These complex and versatile products exhibit inhomogeneous properties, which often need to be spatially or surface resolved. The mechanical properties of such materials, generally considered an important quality indicator, are of particular interest. With the currently employed characterization techniques, nanomechanical characterization of soft matter with small indentation depths ($<2\text{nm}$) and small loads ($<20\text{nN}$) is not possible. Those are however the conditions required for the characterization of organic self-assembled monolayers (SAMs) on hard surfaces. Furthermore, direct correlation between topographical and mechanical features on a surface cannot be readily achieved.

Methods: In the presented research we investigate the applicability of bimodal Atomic Force Microscopy [1], known to yield good results in the characterization of thin polymer films, to molecularly thin soft films. The technique allows for the simultaneous topographical and nanomechanical characterization of the probed surface, giving information on surface elasticity and loss tangent.

Results: Thiol SAMs on Au (111) have been synthesized as a model system. Surface elasticity has been reliably determined and found to be ligand-length dependent. This variation can be related to a change in the molecular ordering within the monolayer. [2] Moreover, thiolated mixed-ligand SAMs show phase separation, evident in $\sim 10\text{nm}$ domain formation, that can be resolved in the mechanical images of the surface. A similar investigation has been extended to the characterization of octadecylphosphonic acid SAMs on Al_2O_3 [3], where surface ordering as a function of monolayer formation time was explored. The evolution of surface elasticity allows distinguishing between the consecutive steps of ligand adsorption, monolayer ordering and multilayer formation. The AFM results are

in good agreement with XPS and static Contact Angle data.

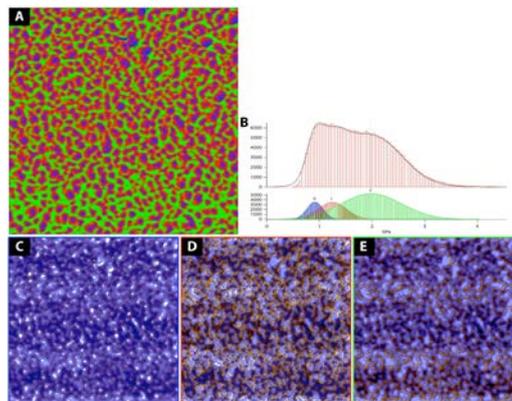


Fig. 1: Map of surface elasticity, E^ , for an OT:TMA mixed ligand SAM (A). Distribution of E^* can be fitted with three peaks, each centered at a different value (B). Features with an average $E^* = 0.90\text{ GPa}$ appear as tall islands, and are ascribed to TMA rich multilayers (C). Features with average $E^* = 1.23\text{ GPa}$ correspond to areas of median height, surrounding the multilayers and are ascribed to a TMA rich phase (D). Features with average $E^* = 1.97\text{ GPa}$ correspond to the shortest areas and are ascribed to an OT rich phase (E). All AFM images correspond to the same area and are $500 \times 500\text{nm}$.*

Discussion & Conclusion: Bimodal AFM can combine the high lateral resolution of conventional AFM with additional observables, from which information on nanomechanical properties can be extracted. Topographical features and nanomechanical properties can be directly correlated. Our results demonstrate that the technique allows for accurate characterization of surface nanomechanical properties in SAMs, as well as the way those scale with varying ligand length, monolayer formation time and monolayer ordering.

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Reactive hexayne surfactant for the formation of 2D carbon nanosheets at the liquid-liquid interface

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Introduction: The liquid-liquid interface is a high energy interface that can be stabilized using interfacially active molecules. The employed surfactants self-assemble at the oil-water interface, exposing their polar head group to water and apolar tail group to the oil phase, which allows using the liquid-liquid interface as a template for two-dimensional assembly. Here, we report the in-situ formation of 2D carbon nanosheets at the liquid-liquid interface, starting from reactive hexayne surfactants that undergo a two-dimensional interfacial polymerization under mild conditions. Our laboratory had previously used a hexayne surfactant with a methyl ester head group and a reactive hexayne segment to fabricate functional carbon nanosheets at the air-water interface [1]. In the work presented here, we now employed an amphiphile composed of a polar phosphonic acid as a head group, a highly reactive hexayne reactive segment, and a long alkyl chain.

Methods: The liquid-liquid interfaces studied are the toluene/water and chloroform/water interfaces. The pH of water was adjusted using 1M NaOH and 1M HCl solutions. The transition from molecular surfactant stabilization to a colloidal stabilization of the interface can be followed by drop tensiometry as well as interfacial rheology and allows to gain fundamental insights into the emulsion stabilization process. Scanning electron microscopy was performed on the Langmuir-Blodgett transferred carbon nanosheet.

Results: The drop tensiometry images show the formation of a buckled interface upon change of interfacial area, which suggest the formation of a carbon nanosheet at the liquid-liquid interface (Figure 1). This nanosheet is formed spontaneously between pH 4 and pH 8, whereas for lower pH values a UV irradiation step seems

to be necessary to produce a film. The formation of a continuous carbon film was confirmed by electron microscopy. Finally, interfacial rheology shows a drastic change in behaviour of the liquid-liquid interface upon formation of the carbon nanosheet.

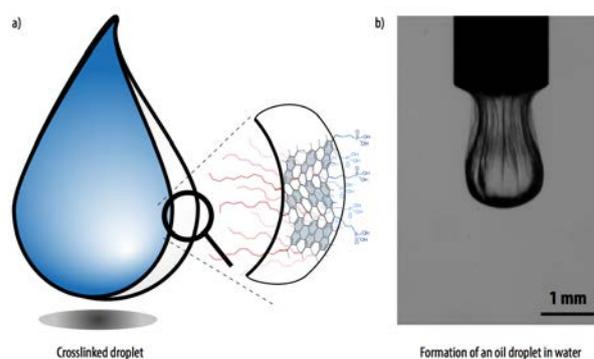


Figure 1: a) Carbonization at the liquid-liquid interface upon mild heat or UV treatment b) buckling of the chloroform/water interface due the formation a carbon nanosheet at the interface

Discussion & Conclusion: The precursor amphiphiles spontaneously self-assemble at the oil-water interface and can be crosslinked by mild heat treatment or UV irradiation, forming a thin Janus 2D carbon nanosheet. In contrast to molecular surfactants, the resulting carbon nanosheet presents distinctive properties of a Pickering emulsion, such as buckling upon a change in the interfacial area. Finally, the mechanical and barrier properties (retention properties) of the formed carbon nanosheets will be investigated in depth.

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Food-grade capsules based on microfluidic

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*Materials Science and Engineering Section, École polytechnique fédérale de
Lausanne, Switzerland*

Motivation

Capsules are often used in food, for example, to protect active ingredients or to control their release [1]. They can conveniently be made from double emulsion templates. One method that offers good control over the size and composition of double emulsions is microfluidics. We employ this method to produce food-grade capsules that are soft but mechanically robust.

Experiment

We produce food-grade capsules from water-in-oil-in-water double emulsion templates that we assemble using microfluidics [2]. To make capsules soft, we fabricate the shell from sunflower oil and stabilize it using food-grade surfactants. We tune the mechanical properties of the shell as well as its permeability by adjusting the shell thickness, and surfactant

composition. By choosing the right surfactant, capsules can be stable for months. Then in the future, by adding some responsive agent in the oil shell so that we can control released the encapsulation of active compounds inside capsules.

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Programmable assembly of hybrid colloidal microswimmers

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Introduction: Patchy colloids with directional interactions have attracted enormous interest in the research community, where they have been applied for the formation of colloidal superstructures or as self-propelled, actively moving objects. Such active colloids, or artificial microswimmers, convert uniform sources of fuel (e.g. chemical) or uniform external driving fields (e.g. magnetic or electric) into directed motion by virtue of asymmetry in their shape or composition. These materials are currently explored as models for out-of-equilibrium systems, with the promise of being used as micro- and nanoscale devices for health and environmental applications. Currently the fabrication of active colloids is limited in the choice of materials, geometries, and modes of motion. Here we use sequential capillarity-assisted particle assembly (sCAPA) to link microspheres of different materials into hybrid colloidal clusters of designed shapes that can actively translate, circulate, and rotate powered by asymmetric electro-hydrodynamic flows. We characterize the active motion of the clusters in a vertical AC electric field, and discuss how composition and shape can be used to tune their trajectories.

Methods: The sequential capillarity-assisted particle assembly (sCAPA) is depicted in Figure 1.

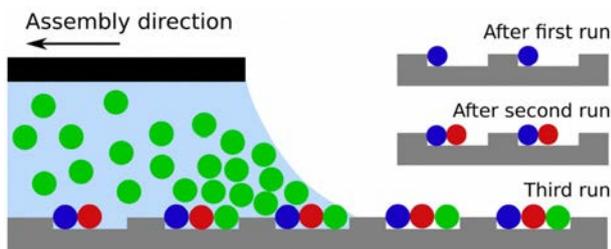


Fig. 1: Controlling the trap depth and surface tension of the colloidal suspension it is possible to trap only a single particle per assembly step with the sCAPA technique.

The shape of the clusters is defined by the shape of the trap and the composition of the clusters is defined by the assembly sequence. The sCAPA technique does not require specific materials or surface chemistry, thus different materials can be easily combined. The assembled colloidal clusters can be linked permanently and harvested from the template for further applications [1].

Results, Discussion & Conclusion:

The microswimming is achieved through the active motion of the colloids synthesized by sCAPA in AC electric fields. The particles on an electrode generate electrohydrodynamic flows in their vicinity in the presence of a vertical AC field. And the imbalanced flows around asymmetric (in shape or composition) micro-objects generate a net force and active motion [2]. The velocity of the microswimmers varies with the composition and shape of the colloidal constituents as in Figure 2 [3]. The shape of the microswimmers also determines their motion modality. The microswimmers can be additionally guided by patterned electrodes and can transport single colloidal particles as cargo.

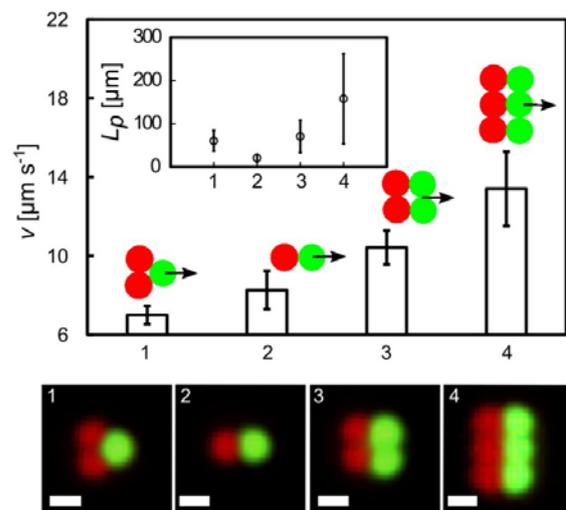


Fig. 2: Velocity of active microswimmers of different shapes. The scale bars are 1 μm .

References:

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Ferrocene derived mechanophores for stimuli-responsive materials

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Introduction: There is increasing interest in the design and development of new stimuli-responsive polymers, materials that upon application of external stimuli, such as light, heat, pH, or redox potential, are able to change their physical and chemical properties [1]. Here, we investigate two different polymers containing ferrocene units as novel mechanophores: polyurethane, with randomly distributed ferrocene units (**Fc-PU**), and poly(methylacrylate) (**Fc-PMA**), with only one chain-centered mechanophore.

Methods: The **Fc-PU** **ref-PU** (without mechanophores) were obtained by a polycondensation [2]. A single-electron transfer living radical polymerization (SET-LRP) was performed to obtain **Fc-PMA** and **ref-PMA** [3]. We subjected these materials to mechanical stress in solution via ultrasonication, focusing on a detailed kinetic study developed using size-exclusion chromatography (SEC) following previous works by Moore and C. Weder [2-4]. In this work we focused on the selective scission of ferrocene derivatives embedded in polymers with the consequent extrusion of the central metal ion. Because of its stability and high susceptibility to a large number of organic reactions, ferrocene is an ideal initiator for polymerization reactions, such as ATRP and SET-LRP or polycondensation [5].

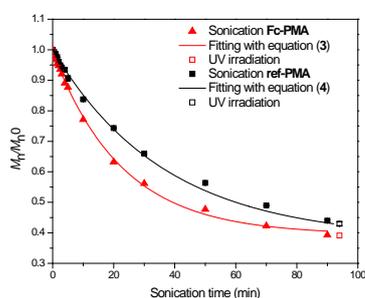


Fig. 1: Time-evolution of the ratio of the molecular weight for **Fc-PMA** (red) and **ref-PMA** (black) during the ultrasonication experiment.

Results: SEC studies show a decrease in the initial molecular weight as a function of time for both polymers **Fc-PU** and **Fc-PMA**. The kinetic

study allowed distinguishing between random and specific chain scission (Fig. 1).

To prove the selective breaking of ferrocene under stress, the release of the central metal ion in solution, and the subsequent oxidation from Fe(II) to Fe(III) for **Fc-PU**, we used KSCN as a ligand to form the red complex FeSCN^{2+} in solution (Fig. 2).

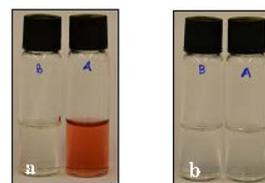


Fig. 2: **a)** **Fc-PU** solutions before (transparent on the left) and after sonication (formation of the red complex FeSCN^{2+} on the right). **b)** **ref-PU** solutions before and after sonication: the solutions did not change color.

Discussion & Conclusion: We proved that ferrocene derivatives are optimal mechanophores with the ability to break and to release the central metal under mechanical stress in solution. The mechanophores break more quickly than the random bonds as proved by SEC studies and comparing the results with free-mechanophores polymers, as references. Further proof of the ferrocene breaking and subsequent release of the central metal has been performed with a color change experiment, adding the ligand KSCN, which readily coordinates with Fe ions.

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Gold nanowire fabrication with surface-attached lipid nanotube templates

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Introduction: The fabrication of conductive nanostructures is the key technology in semiconductor industry and has gained importance in biology for applications such as biosensors and drug delivery.

We have demonstrated a high-throughput approach to fabricate gold nanowires on surfaces with a lipid nanotube template [1].

Methods: Biotin-tagged lipid nanotubes are formed from lipid blocks in inverted hexagonal phase adsorbed on polymer-coated surfaces upon application of shear force. Streptavidin-coated gold nanoparticles were attached to the biotin-tagged lipid nanotubes and gold nanoparticle-encapsulated LNTs were cross-linked by chemical fixation. Samples were dried and treated with oxygen plasma to remove the organic template and connect the particles.

Discussion & Conclusion: An approach to surface-assemble nanoparticles with a lipid nanotube template was demonstrated. The method is advantageous because the small size of lipid nanotubes enables the fabrication of solid nanostructures with a higher throughput without using expensive electron beam lithography. The approach can further be combined with single lipid nanotube patterning with a micromanipulator [2] to create distinct patterns instead of random networks

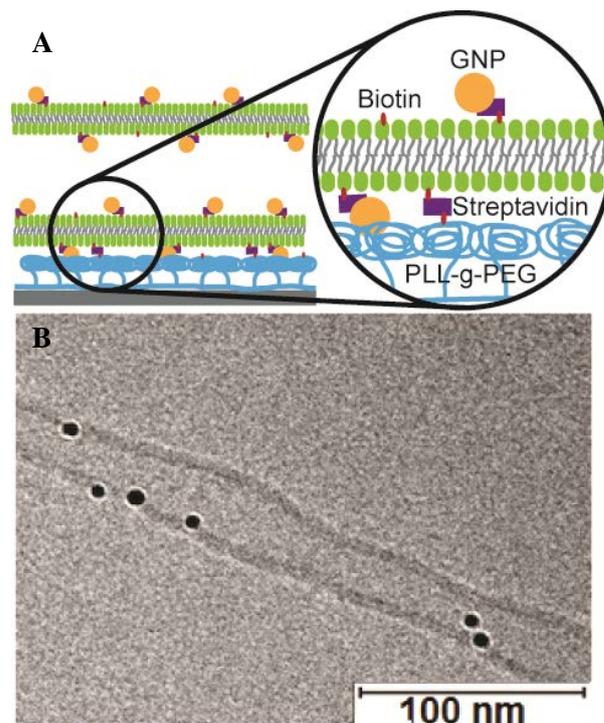


Fig. 1: (A) An illustration showing the strategy of attaching gold nanoparticles to lipid nanotubes. (B) A cryo-TEM image of gold nanoparticles attached to a lipid nanotube.

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Gold Optical Metamaterials made by Block Copolymer Self-Assembly

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Introduction: Optical metamaterials are a group of artificial materials that show extraordinary properties that cannot be found in nature, e.g. a negative refractive index. [1, 2]. Block copolymers (BCPs), which consist of two or more homopolymers linked together by covalent bonds, self-assemble into ordered structures with a wide range of morphologies such as gyroids, cylinders and lamellae with a sub-wavelength period. Self-assembly of BCPs can be considered as a promising method to prepare three-dimensional chiral structures that serve as a sacrificial network to synthesize plasmonic gyroid metamaterials on a large scale.

Methods: We use a bottom-up method established in our lab [3]:

1. Self-assembly of PI-b-PS-b-PEO triblock terpolymers resulting in the fabrication of a triply gyroid network.
2. Selective etching of one component from the polymer network resulting in a voided structure.
3. Backfilling of the voided network with a plasmonic material, e.g. by electrodeposition.
4. Removing the remaining polymer matrix through plasma etching.

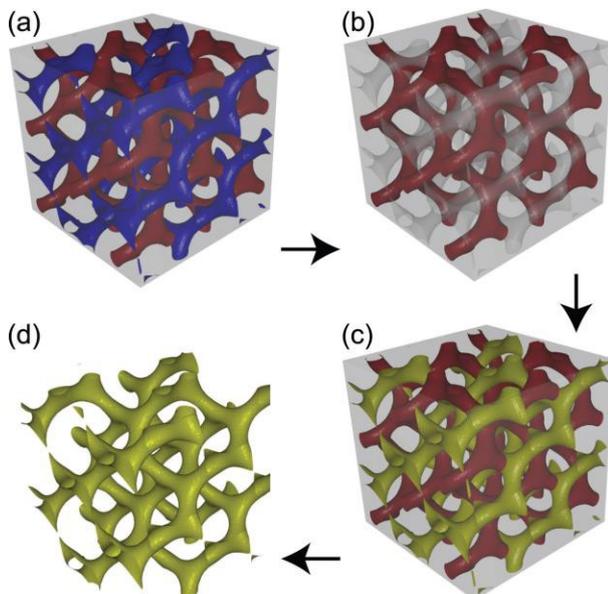


Fig. 1: Schematic of the fabrication of a gyroid metamaterial. [3]

Results: With the sub-wavelength structure of the gyroid (period of 50-60nm), we expect to observe different optical properties from metallic samples. For instance, the plasma frequency of a gold gyroid sample is noticeably reduced compared to the bulk gold plasma frequency. Moreover, optical microscopy of gold replicated gyroids under linearly polarized light shows large domains with a pronounced dichroism both, in reflection and transmission.

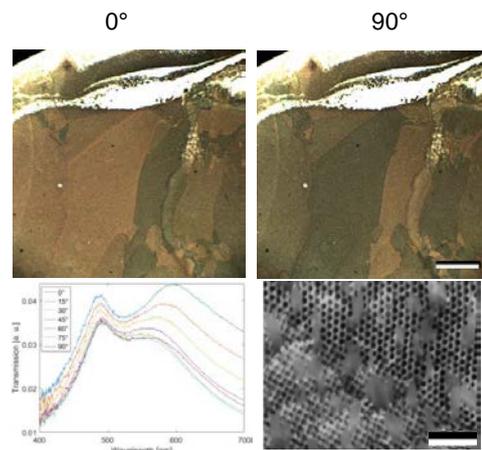


Fig. 2: Optical microscope images of a 700nm thick gold gyroid sample in transmission with a linear polarizer (axis at 0° and 90°). Transmission depending on the linear polarization axis. SEM image at the border of two large domains.

The linear dichroism axis depends on the orientation of the gyroid structure within a single grain. It is characterized by a red shift of the plasma edge and the appearance of a transmission peak around 600 nm.

Discussion & Conclusion: Gold gyroid optical metamaterials are created from a block copolymer template. Additional understanding of the copolymer self-assembly behavior is very important to further optimize the process and to gain full structural control. Our aim is to investigate different approaches to control structure orientation and uniformity in the future.

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Polydiacetylene-peptide interaction mechanism in mixed lipid systems

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Introduction: Information transfer between cells always starts with forces applied to the membrane. Opening Ion channels, cellular virus infection and killing bacteria by antimicrobial peptides, all apply forces to the bilayer (BL) of the cell. However, until now there is no possibility to measure the local molecular forces in lipid BL. Therefore, the goal of this project is to develop a calibrated fluorescence probe by the mechanosensitive polymer PDA.

Biosensors are used to convert a biological response into a signal. The sensor is triggered by its analyte e.g. viruses, proteins and peptides. Here, the conjugated polymer PDA is investigated because of its optical characteristic in visible range and its easy self assembling property like BL and vesicles. By perturbation of the backbone chain, PDA undergoes a mechanochromism from the blue to the red conformation. The disturbance is induced by a peptide which penetrates the bilayer. But as previous experiments have shown, the sensitivity of a 100% PDA film is very limited. Hence, the layer is enhanced by mixing it with a fluidic lipid. The peptide interacts at the raft boundaries between the lipids in solid and liquid phase. The right ratio is a key element to improve the system. The next step is to understand how the peptide exactly rotates the pedant side groups.

Methods: The first step is to improve the sensitivity of the system. Therefore, a series of different ratios between lipids in solid and liquid phase in solution has been studied by UV-VIS Spectroscopy before and after adding peptides. We selected DOPC as an example for a lipid in liquid phase and Melittin as a representative peptide. Furthermore, bilayers were investigated by inverted fluorescence microscopy and AFM.

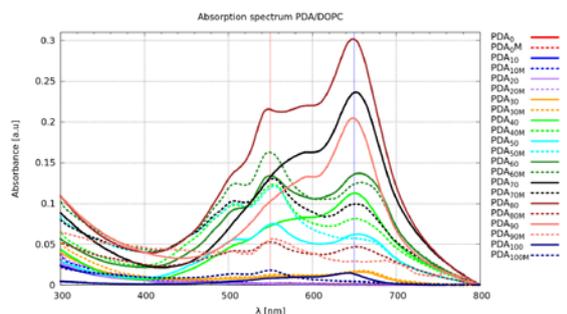


Fig. 1: Absorbance spectrum of different DOPC/PDA ratios with (dotted line) and without Melittin (continuous line).

Results: A quantitative value for the blue-to-red transition of PDA is given by the colorimetric response:

$$\%CR = (\Pi B_0 - \Pi B_1) / \Pi B_0 \quad (1)$$

$PB = A_{blue} / (A_{blue} - A_{red})$, A is the absorbance at either the blue (550nm) or red (650nm) maximum of the UV-VIS Spectrum. PB_0 is the ratio of the control sample without the peptide. A maximum for the CR for a PDA/DOPC ratio has been found at 70/30.

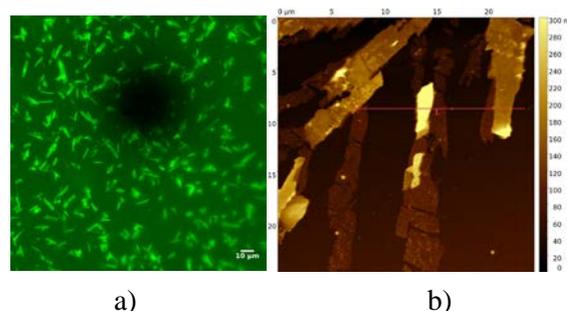


Fig. 2: (a) shows DA flakes incorporates into the DOPC BL (recovery within 10min.) imaged by microscopy in GFP channel. AFM height measurement (b) reveals stacking of multilayers.

Further investigation on planar systems showed, that the DA incorporates into the DOPC BL and start stacking multilayers

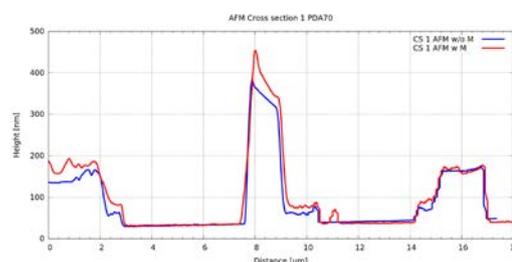


Fig. 3: Cross section of DA structure (from fig.2b) before and after adding Melittin. A slightly increase of height is visible.

Discussion & Conclusion: As we could show, a minimum amount of soft lipid is required for proper PDA polymerisation. The optimal ratio was 70/30. The sensitivity of the biosensor is triggered by the peptide Melittin and confirmed by CR. Investigation on BL shows incorporation of DA as multilayer into DOPC BL.

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Surface Stress and Adhesion of Hydrogels

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Introduction: Existing contact mechanics theory accurately predicts the force-indentation behavior of hard and semi-compliant materials, but fails for very soft materials – due to the effect of solid surface stress, Υ . The cross-over point at which it breaks down is the elastocapillary length:

$$L_c = \Upsilon/E \quad (1)$$

Recent experiments with silicone gels have shown that surface stress can be critical to the contact mechanics of highly compliant materials. In addition, it has been shown that the surface stress, and thus its contribution to force-indentation behavior, may be strain-dependent.

We aim to investigate the role of surface stresses in hydrogels. Hydrogels are common both in synthetic and natural forms. As such, they find applications in a wide variety of fields like tissue engineering, cell culture, stretchable electronics, and hygiene products. Given their ubiquity and their uses, a fundamental understanding of hydrogel adhesion and contact mechanics is of great importance.

Methods: Using the model system of polydimethylacrylamide (PDMA) gel, we set out to confirm measurement techniques for the regime of contact mechanics behavior above the elastocapillary length. In particular, we aim to verify a technique for zero-force adhesion and a method for applied force measurements

The PDMA gel comprises ~92% water and has stiffnesses in range 500Pa to 60kPa.

For zero-force adhesion, gels made with 100nm fluorospheres and are cast in thin films. Silica beads, ranging from 10 μ m to 40 μ m are placed on the films and the resulting surface profile is observed, as shown in Figure 1.

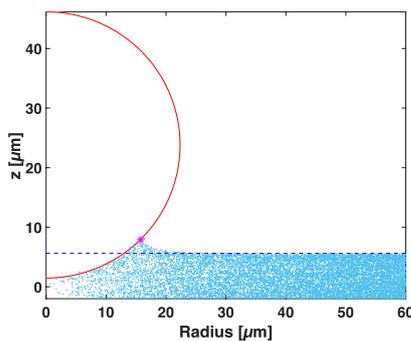


Figure 2: Profile of a silica bead on PDMA

The relationship between contact radius and bead radius is then compared with the Johnson-Kendall-Roberts zero-force prediction:

$$a = [9\pi W(1 - \nu^2)/2E]^{2/3} R^{1/3} \quad (2)$$

Applied force measurements are also performed on a bulk sample of PDMA using a nm-resolution capacitive force probe.

Results: The measurements taken for zero-force adhesion on PDMA above the elastocapillary length, as shown in Figure 2, verified the confocal technique as a viable measurement method.

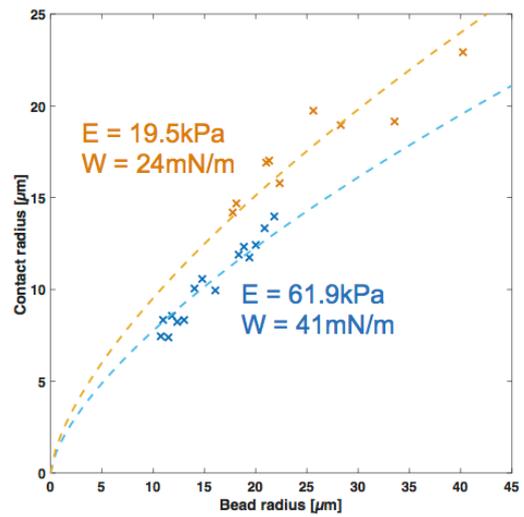


Figure 3: Contact v. bead radius for two gels

Through the applied force measurements, we were also able to obtain data showing some potential strain-dependent behavior.

Discussion & Conclusions: Having verified two measurement techniques, we are moving to explore the contact mechanics behavior in the regime below the elastocapillary length. To this end, we are working to create thin films of softer gels.

Network forming solid polymer electrolytes for rechargeable lithium batteries

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Introduction: Rechargeable lithium batteries are ubiquitous. Since their commercialization in 1991, they have quickly become the first choice for energy storage in portable electronics and they are in the process of supplanting all other battery chemistries across application. Two important future uses are electric vehicles and grid-level storage. However, to economically meet the enormous forecasted increase in demand for capacity, it is imperative to find ways to raise energy density and safety.

Methods: The aim of this project is to do just that, develop a solid polymer electrolyte (SPE) alternative to state of the art alkyl carbonate electrolytes. Specific performance targets for the final electrolyte include; lithium ion conductivity $\sigma_{\text{Li}} > 10^{-4}$ S/cm from 0-40 °C, 4-5 V stability window, and film forming capability with a modulus $E' > 10^5$ Pa. The principal approach leveraged polymer self-assembly to form 3D network structures (see figure 1) with an optimal salt concentration, $r = 0.08$ (lithium ion to ether oxygen Li^+/EO), then confirming conductivity and mechanical properties. Following verification, the structure was manipulated with P(LiMTFSI) or PHEA-*l*-PEG to optimize conductivity.

Results: Various techniques were used to confirm 3D structures in the resulting SPEs, AFM, GISAXS, SEM, and EIS (See Figure 1).

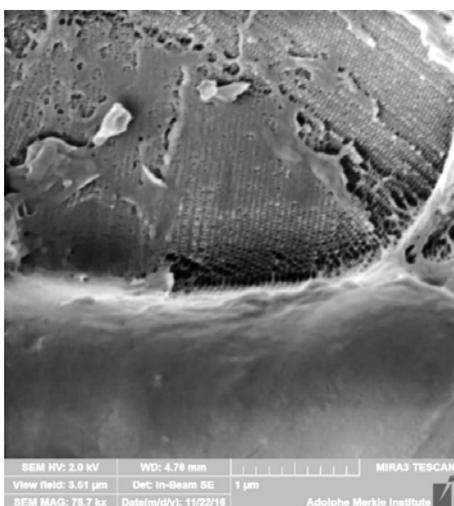


Fig. 1: SEM image of gyroid structured SPE.

Conductivity data was normalized to PEO content to assess the structural effect on Li transport

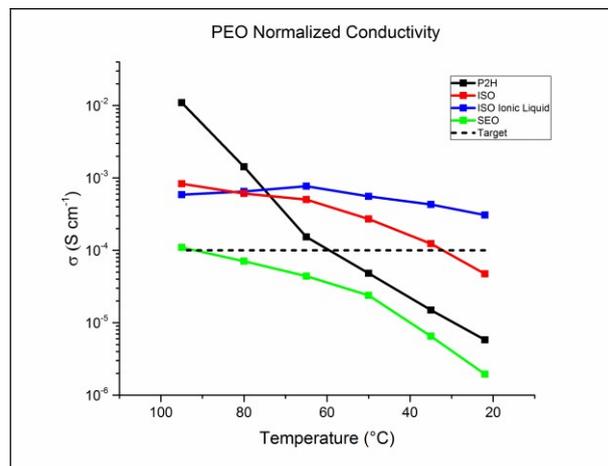


Fig. 2: Conductivity of various SPEs with target conductivity line.

Discussion & Conclusion:

Initial results show the effect of structure on conductivity (See Figure 2). The SEO sample serves as a control, while the ISO and P2H samples show the ability to increase conductivity of the PEO in the SPE by structure and plasticizing. The next step is to test the mechanical properties and integrate the SPE into a cell.

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Limits of Throughput in Droplet Microfluidics

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Introduction: Droplet microfluidics is a common tool in the life sciences and chemistry to manipulate small volumes of liquid. Picoliter-sized solvent droplets are generated inside an immiscible outer phase (e.g. aqueous droplets in oil) and manipulated in a network of micrometer-sized ducts [1]. The core strength of microfluidic technology is the high rate at which the same steps of droplet manipulation can be repeatedly executed in an automatic fashion: Droplets can be generated and processed at a rate of thousands per second, greatly increasing the statistical sample size in any experiment. We investigate the fundamental limits of high-throughput droplet processing, namely the interplay of the surface tension forces that hold the droplets together, and different effects that tear droplets apart at high flow rates.

Methods: We developed a fully resolved 3D Boundary Element Method (BEM) scheme to simulate the deformation of the free surface of single or multiple droplets in arbitrary microchannel geometries and under the influence of electric fields. The incompressible Newtonian fluids that make up droplets and outer phase are described by the incompressible Stokes equations,

$$\mu \Delta \mathbf{u} = \nabla p, \quad (1)$$

$$\nabla \cdot \mathbf{u} = 0, \quad (2)$$

with μ the dynamic viscosity, \mathbf{u} the flow velocity and p the fluid pressure. The stress on the droplet interface due to surface tension is of Young-Laplace type. The scheme solves for electric fields, which are commonly used to manipulate individual droplets via dielectrophoresis (DEP) [2], and includes their effect in the surface stress.

Results: We present two applications of this model: The breakup of droplets in dense emulsions as they enter a constricted microchannel, and the limits of sorting droplets with dielectrophoresis.

For the purpose of storage (e.g. during an incubation phase), droplets are often collected outside a microfluidic device in a container in which they form a dense emulsion. Reinjecting this emulsion into a microchannel for serial processing is unreliable if droplets break up at the channel entrance. We show how the breakup is

driven by a difference in Young-Laplace pressure between two droplets (Figure 1).

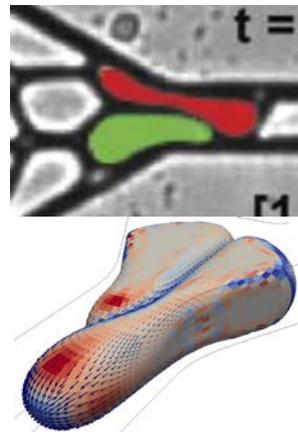


Fig. 1: Droplets squeeze into a constricted channel: Experiment (left, [3]) vs. fully resolved 3D simulation (right).

In the application of droplet sorting by electric fields, the throughput is limited by electrosplitting of droplets in overly strong electric fields. With our 3D model for the field in the presence of a droplet (Figure 2), we present electrode designs that are capable of providing high throughput, while keeping the field strength below the splitting threshold.

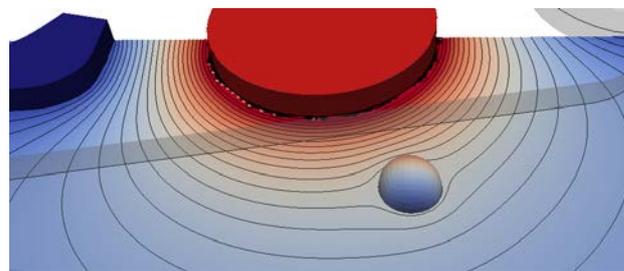


Fig. 2: The presence of a polarizable droplet deforms the electric potential: The droplet moves towards high field strength.

References:

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Forced dewetting for robust scalable generation of Double Emulsions drops with thin shells

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Introduction: Ultra thin shell double emulsions are stable capsules because the offset between the inner and outer drop caused by the difference in density is reduced. The membrane mechanical properties and thickness can be tuned by the chemical structure of the block copolymer used to form the membrane.

Methods: We use water / oil / water double emulsion droplets as a template to assemble the polymersomes. Block copolymers assemble into two monolayers, one at each liquid-liquid interface. Then the oil phase is removed to form the bilayer membrane of the vesicle.

To remove the oil we push those double emulsion drops through a micro channel containing a constriction as shown in fig 2.

We studied the influence of the flow rates and the wettability of the channels on the efficiency of oil removal.

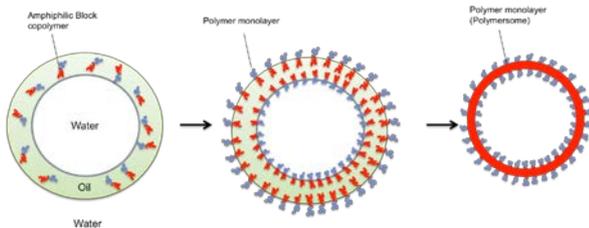


Fig. 1: Schematic of the production of monodisperse polymersomes. First a double emulsion is formed using microfluidics. The amphiphilic copolymers dissolved in the oil adsorb at the liquid-liquid interface to form one monolayer at the outer interface of the double emulsion. Finally, the oil is removed from the shell to form a bilayer.

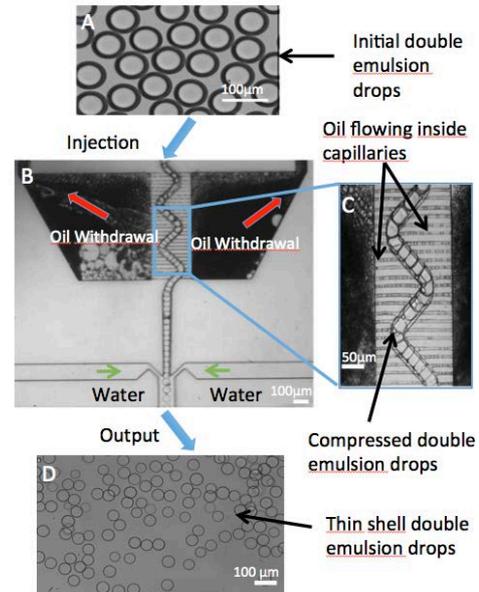


Fig. 2: (A) Double emulsions formed initially. (B) Double emulsions are injected in a narrow channel and stack. (C) oil from the double emulsion shell is removed through the microcapillaries. (D) Ultrathin shell double emulsion of less than 300nm shell thickness are collected at the output

Discussion & Conclusion: Double emulsion drops were used as templates to make capsules with thin shells produced using microfluidics as this method allows high encapsulation efficiency, good control over the size and composition of the capsules and polymersomes. Those double emulsions were used as templates for the production of capsules with thin shells. We decreased the shell thickness of the double emulsion droplets by mechanically confining them; this results in a partial removal of the oil from the shell.

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