NTNU
Norwegian University of Science and Technology

IFE
Institute for Energy Technology

6th Annual International Workshop on Soft & Complex Matter

Theme: Soft Matter and some of its intimate relations.

Norwegian Academy of Science and Letters

Drammensvegen 78, Oslo, Norway, October 6-7, 2019

Invited speakers Oct. 6:

Leide Cavalcanti (ISIS, UK)
Henri van Damme (MIT, USA/ESPCI, Paris, France)
Paul Dommersnes (NTNU, Norway)
Maria Helena Godinho (Univ. NOVA de Lisboa, Portugal)
Carl Fredrik Gyllenhamar (CaMa Geoscience AS, Norway)
Olli Ikkala (Aalto Univ., Finland)
Matti Knaapila (DTU, Denmark)
Ludwik Leibler (ESPCI, Paris, France)
Adrian Rennie (Uppsala Univ. Sweden)
Petra Rudolf (Univ. Groningen, Netherlands)
Patrik Sellin (SKB, Sweden)
Patrick Tabeling (ESPCI, Paris, France)

Invited researchers/postdocs/PhD-student talks Oct. 7:

Paulo Brito (NTNU, Norway)
Kristoffer Hunvik (NTNU, Norway)
Susete Fernandes (Univ. NOVA de Lisboa, Portugal)
Leander Michels (NTNU, Norway)
Etien Martinez (NTNU, Norway)
Konstanse Seljelid (NTNU, Norway)
Osvaldo Trigueiro (NTNU, Norway)
Ana Catarina Trindade (NTNU, Norway)
Héctor Urra (ESPCI, Paris, France)

Practical information:

The workshop starts at 14:30 Oct. 6, and ends at 14:00 Oct. 7, 2019

Organizers and contact:

Jon Otto Fossum (jon.fossum@ntnu.no)
NTNU, Trondheim, Norway
Paul Dommersnes, NTNU, Trondheim, Norway
Geir Helgesen, IFE, Kjeller, Norway
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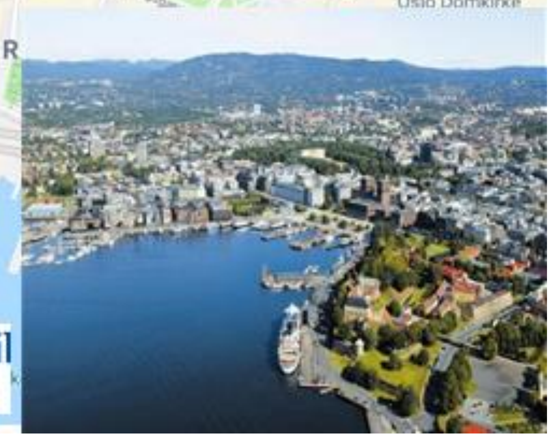
The Research Council of Norway



Program and Book of Abstracts

6th Annual International Workshop
Soft & Complex Matter

Norwegian Academy of Science and Letters, Oslo, Norway

October 6-7, 2019

Program Summary:

Time	Sunday October 6
	Norwegian Academy of Science and Letters, Drammensvegen 78 Oslo 
14:30-14:55	**Arrival and Registration**
14:55-15:00	**Jon Otto Fossum:** *Welcome*
15:00-15:20	**Maria Helena Godinho:** *The morphology of plants micro/nano filaments revealed by liquid crystalline droplets.*
15:20-15:40	**Paul Dommersnes:** *Polar Flocking of Active Clusters*
15:20-16:00	**Henri van Damme:** *Is thermal insulation the right choice for energy savings in buildings? The case of eco-materials.*
16:00-16:20	**Patrick Tabeling:** *Phoamtonics*
16:20-16:40	**Discussions**
16:40-17:00	**Ludwik Leibler:** *From Glass to Vitrimers: A Story of Exchangeable Links*
17:00-17:20	**Petra Rudolf:** *Playing Lego with clay: Deposition and intercalation strategies to produce pillared structures*
17:20-17:40	**Patrik Sellin:** *The activity of sulfate reducing bacteria in bentonite*
17:40-18:00	**Carl Fredrik Gyllenhamar:** *Quick clay for Well Integrity and permanent Plug and Abandonment*
18:00-18:20	**Discussions**
18:20-18:40	**Olli Ikkala:** *From responsive and shape memory materials to materials that mimic behaviorists´ classical conditioning*
18:40-19:00	**Adrian Rennie:** *Effect of the plasticizers used in PVC on lipid monolayers and bilayers*
19:00-19:20	**Leide Cavalcanti:** *Sample environment for SANS at ISIS*
19:20-19:40	**Matti Knaapila:** *Emerging Possibilities for in situ X-ray Experiments: Real-Time Structural Evaluation of Naphthyl End-capped Oligothiophenes in Organic Thin Film Transistors during Deposition*
20:00-23:00	**Dinner including and followed by discussions**

Monday October 7	Time
Susete Fernandes: *Cellulose Nanocrystal Films from Drop Casting: Easy Method to Control CNCs' Deposition*	**09:30-09:45**
Hector Urra: *Bacteria swimming in complex fluids*	**09:45-10:00**
Ana Catarina Trindade: *Tuning Photonic Properties of Cellulose-Clay Nano Structures*	**10:00-10:15**
Ana Catarina Trindade: *Cellulose-based Membranes for Effective Capture of Oil Microdroplets in Water*	**10:15-10:30**
Etien Martines: *Active turbulence/Vortex array transition in Quincke rotating beads*	**10:30-10:45**
Discussions	**10:45-11:00**
Paulo Brito: *Clay hybrid nano-layer assembly, structure and mechanics at the air-water interface*	**11:00-11:15**
Leander Michels: *Water vapor diffusive transport in a smectite clay: Cationic control of normal versus anomalous diffusion*	**11:15-11:30**
Konstanse Seljelid: *Microcapsule controlled gelation in channels and pores such as those in petroleum-reservoirs*	**11:30-11:45**
Kristoffer Hunvik: *Carbon dioxide capture and retention by synthetic Fluorohectorite clay*	**11:45-12:00**
Osvaldo Trigueiro Neto: *Exfoliation of Clay Nanosheets*	**12:00-12:15**
Discussions	**12:15-12:30**
Lunch including and followed by discussions	**12:30-14:00**
Departure 	**14:00 -**

Sunday October 6:

14:30 - 14:55 Registration

***14:55 - 15:00 Jon Otto Fossum**, NTNU, Trondheim, Norway:* **Welcome**

15:00 - 15:20

Ana P. Almeida[1], João Canejo[1], Urban Mur[2], Simon Čopar[2], Pedro L. Almeida[1,3], Slobodan Žumer[2,4] and Maria Helena Godinho[1]

[1]CENIMAT/I3N, Departamento de Ciência dos Materiais, Faculdade de Ciências e Tecnologia, FCT, Universidade Nova de Lisboa, Campus da Caparica, 2829-516 Caparica, Portugal.
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The morphology of plants micro/nano filaments revealed by liquid crystalline droplets.

Recently micro nematic droplets were used to sense surface morphologies and chirality of cellulose electrospun and natural spider silk fibres [1]. This was possible due to the responsiveness of liquid crystals to external fields. Quantitative work was also performed by using fibres that promote planar or helicoidal alignment, inserted in cells with well-defined homeotropic anchoring at the surfaces [2], the application of electric and magnetic fields enabled the disclination loops to acquire controlled translational movements along the fibre main axis as well as the determination of the fibres chirality and handedness. In another hand Plant leaves' tracheary system is comprised of a cellulose skeleton with diverse hierarchical structures. It is built of polygonally bent helical filaments of nano rods coated by different layers, which provide them high resistance, elasticity and roughness. Their function includes the transport of water and minerals from the roots to the leaves. Tracheary helices were also used as biotemplates to produce microswimmers and biosensors. Spotting details about local interactions of the tracheary elements with the surrounding material, which varies between plants, also due to adaptation to different environments, is crucial for the understanding of ascending fluid transport and for tracheary potential applications. In this work, we establish correlations between the surface morphology and the mechanical properties of entangled microfilaments, collected from different plants' species, which belong to the same order and have the same diameter [3]. The surface morphology of the filaments is characterized by liquid crystal droplets. The method used provides access to small, almost imperceptible details of the microfilaments, which appeared crucial for their mechanical behaviour, function performance and interaction with environment.

[1] L. E. Aguirre et al., Sensing surface morphology of biofibers by decorating spider silk and cellulosic filaments with nematic microdroplets. Proc. Natl. Acad. Sci. U.S.A. 113, 1174–1179 (2016).
[2] P. Pieranski, M.H. Godinho, Flexo-electricity of the dowser texture, Soft Matter, 15, 1469 (2019).
[3] A. P. Almeida et al., Spotting plants' microfilament morphologies and nanostructures. Proc. Natl. Acad. Sci. U.S.A. 116 (27), 13188-13193 (2019).

15:20 - 15:40 ***Paul Dommersnes[1]* & Jon Otto Fossum[1,2]**

[1] Department of Physics, Norwegian University of Science and Technology (NTNU), Trondheim, Norway
[2] MMN ESPCI Paris- Institut Pierre-Gilles de Gennes (IPGG), Paris, France

Polar Flocking of Active Clusters

Biological active matter, such as populations of cells and animals, often change between different swarming states. One example is shoaling, milling and schooling fish. Synthetic active matter consist of self-propelled inanimate units and emulates biological active matter.
We combine electric field induced attraction with electro-rolling propulsion [1] in a population of granular beads. A variety of swarming regimes is realized: polar clusters, a stripe phase of clusters, and polar liquid flocks, reminiscent of transitions seen in active matter simulations [2,3,4]. Remarkably, the cluster to liquid transition occurs at a different velocity threshold than the local to global polar order transition. The experimental system offers a physical model for swarming transitions in biological active matter and can also open new routes for controlling self-assembly in soft matter technologies.

[1] A. Bricard, J.-B. Caussin, N. Desreumaux, O. Dauchot, D. Bartolo, Emergence of macroscopic directed motion in populations of motile colloids, Nature 503, 95 (2013)
[2] Living Clusters and Crystals from Low-Density Suspensions of Active Colloids, B. M. Mognetti, A. Šarić, S. Angioletti-Uberti, A. Cacciuto, C. Valeriani, and D. Frenkel, Phys. Rev. Lett. 111, 245702 (2013)
[3] Hydrodynamic interactions in dense active suspensions: From polar order to dynamical clusters, N. Yoshinaga and T. B. Liverpool Phys. Rev. E 96, 020603(R) (2017)
[4] Spontaneous aggregation and global polar ordering in squirmer suspensions, F.Alarcón and I.Pagonabarraga, Journal of Molecular Liquids, 185, 56-61 (2013)

15:40 – 16:00 ***Henri van Damme[1,2]***

([1]) <MSE>[2], The MIT-CNRS joint laboratory; ([2]) ESPCI-Paris, France

Is thermal insulation the right choice for energy savings in buildings? The case of eco-materials.

The current strategy of the construction sector in order to obtain energy savings in buildings is to decrease the thermal conductivity of walls, ceilings and floors. Thermal conductivity norms are imposed by law for new constructions, without any concern for other thermal properties. This leads to material science research entirely oriented toward the lowest possible λ values. Aerogels are good examples of the outcome of this type of research.
Yet, other strategies are possible. In particular, vernacular architecture is a hidden deposit of what we should better consider as high-tech knowledge and a good source of inspiration for industrial construction materials and technologies. Vernacular construction is mostly using either geo-sourced (stones, raw earth) or bio-sourced (bamboo, straw, reed, hemp…) materials which are often providing remarkable hygro-thermal comfort even in harsh conditions. A closer look shows that this comfort is essentially due to two properties. The first, rather trivially, is a high thermal mass, which limits and shifts the amplitude of daily and seasonal thermal fluctuations. The second interesting property, encountered in natural fibres and to some extent in raw earth (which is nothing but a natural concrete with clay as binder), is a multi-scale open porosity which is allowing for an active control of temperature and humidity, thanks to high latent heat of the liquid-vapor transition (and vice-versa) of water in the low end part of the pore size distribution, while the high end part permits rapid exchanges of vapour with the environment.
An industrial material – that is, a material that can be mass produced according to given standards – with similar properties would be a game-changing material. A composite of natural fibres (hemp for instance) in a clay-rich matrix would be a good candidate.

16:00 - 16:20 ***Patrick Tabeling***

MMN ESPCI Paris, website: www.mmnlab.com

Phoamtonics

I will describe the work that we are performing in my group (MMN) at ESPCI, dedicated to the obtention of new materials using microfluidics. This work ranges from the assembly of droplets to create self-assembled materials, to the realization of foams at the microscale, enabling the obtention of photonic band gap materials in the IR, and hopefully in the future, in the visible range. The word "phoamtonics", coined by S.Torquato, means: "Photonic band gaps of 3D foams".

16:20 - 16:40 ***Discussions***

16:40 - 17:00 ***Ludwik Leibler***

Gulliver, ESPCI, Paris, France

From Glass to Vitrimers: A Story of Exchangeable Links

During cooling, amorphous silica, the archetype glass-former gradually increases its viscosity over a wide temperature range and it becomes so viscous that for all practical purposes it behaves like a hard solid, the glass. In striking contrast and without exception all organic glass-formers increase their viscosity and rigidify very abruptly when cooled. Moreover, silica is not soluble. We imagined, vitrimers, organic materials that undergo gradual glass transition and are not soluble just like silica. Vitrimers are molecular networks that through thermo-activated exchange reactions are able to change their topology without changing the total number of bonds. Solid at low temperatures and malleable when heated yet insoluble whatever the temperature, vitrimers constitute the third class of polymers along with thermoplastics and thermosets (elastomers).
After recalling vitrimer design strategies, I will focus on some fundamental aspects related to flow properties and solution behavior of vitrimer networks. I will also discuss a recent progress in understanding incompatibility effects and self-assembly in vitrimer systems.

17:00 - 17:20 ***Petra Rudolf***

Zernike Institute for Advanced Materials, University of Groningen, Netherlands,
e-mail: p.rudolf@rug.nl

Playing Lego with clay: Deposition and intercalation strategies to produce pillared structures

Even since the Mayans first used clays to make dyes, mankind has made use of layered materials. Over the past two decades this effort is mirrored in scientific research leading to pillared materials for application in gas storage, catalysis, drug delivery and environmental remediation. I shall first illustrate the synthesis and intercalation strategies we use to construct pillared montmorillonite clay structures and then showcase a few of these hybrids with different pillaring agents and different pore structure for different applications, from elimination of herbicides from drinking water to acid catalysis for isopropanol dehydration.

17:20 - 17:40 ***Patrik Sellin*, Daniel Svensson***

Svensk Kärnbränslehantering AB, Solna, Sweden
****patrik.sellin@skb.se***

The activity of sulfate reducing bacteria in bentonite

In the Swedish KBS-3 concept highly radioactive used nuclear fuel is emplaced in copper canisters embedded in compacted bentonite deep down (-500 m) in crystalline rock (designed lifetime is 100 – 1000 ka). The bentonite is protecting the canister chemically and physically from the surroundings. One of the main functions of the bentonite is to inhibit activity of microbes, e.g. sulfate reducing bacteria (SRB). During favourable conditions SRB may reduce sulfate (S(VI)) from the bentonite or from the groundwater) into sulfide (S(–II)) that may corrode the copper canister.

Under saturated conditions the microbial sulfate reduction will be limited if the bentonite density is sufficient. Current observations indicate that at the high swelling pressure that arise during water saturation (> 2 MPa), microbes become inactive. In order to investigate the impact of swelling pressure on the activity of sulphate-reducing bacteria, special test equipment has been developed. The principle of the equipment is that a bentonite sample is charged with a cocktail of bacteria, pressed to a given density and pre-saturated with water. The sample is then moved to the test cell where it is provided with a copper plate underneath and a solution of sulfate, with added radioactive sulfur on top, where also a nutrient is injected. After a given time, the activity of microbially-formed sulfide can be measured on the copper plate. The results indicate that there is a clear cut-off in dry density, or swelling pressure, where sulfide production ceases. For a Calcigel bentonite the microbial activity disappears above a saturated density somewhere in the range 1 850–1 900 kg/m^3.This cut-off is, however, different between different bentonites. This means that it is that there are other factors that the degree of compaction that affects the microbial activity.

During the early period of the repository lifetime and before the bentonite is water saturated, there may be a window of opportunity where the bentonite is neither dry, nor wet, when a high enough relative humidity is present for SRB to potentially be active. This study aims at finding out if there is a moisture threshold limit for the bentonite when SRB may produce sulfide.

Several types of experiments were performed using (i) natural SRB in Wyoming bentonite, (ii) added commercial strain of SRB (*Pseudodesulfovibrio aespoeensi*) and (iii) enrichment from a natural groundwater from the Äspö underground laboratory borehole.

Tube experiments were performed in an anaerobic box (N_2-atmosphere) where an open glass tube was emplaced within a bigger closed plastic tube. In the smaller glass tube bentonite, gypsum, lactate, commercial SRB, and nutrients were emplaced and in some cases liquid water. In the outer plastic tube a solution was placed with either pure water or saturated salt solutions, and with a small addition of $CuSO_4$ salt as an indicator of formed hydrogen sulfide from the small tube. The different salt solutions gave rise to different relative humidities. Microbiological reduction of sulfate to hydrogen sulfide was detected to occur when the microbes had access to liquid water (in addition to gypsum, lactate, and nutrients and no swelling pressure), while no hydrogen sulfide production was detected when the microbes were restricted to a relative humidity of 75-100%. The amount of CuS formed was lower when bentonite was present compared to with no bentonite, which is compatible with previous experiments, where bentonite was found to react with hydrosulfide, possibly redox active Fe(III) in montmorillonite is responsible for oxidising the S(-II) to S(0).

17:40 - 18:00 *Carl Fredrik Gyllenhammar*

CaMa GeoScience AS

Quick clay for Well Integrity and permanent Plug and Abandonment.

Quick clay is being proposed as a material to permanently plug oil and gas wells.
Oil and Gas well abandonment is regulated following the NORSOK standard D-010. Permanently plugged wells shall be abandoned with an eternal perspective. If any potential inflow there must be a minimum of two barriers.
Today, cement is the preferred product for plug and abandonment of oil and gas wells. There are several good reasons for this, e.g. abundant supply of cement, a familiar well-functioning supply chain, cement is easy to pump, and it sets quite fast. However, there is concern for areas where the reservoir subsides. The cement will crack, and the well can start to leak. Such instances have proved very difficult to stop permanently. Alternative barrier materials have been suggested, such materials include cementitious materials, polymer resins, unconsolidated sand slurries, grouts, etc. Grouts are interesting groups which have outstanding properties but some limitations to be solved.

Case 1: Offshore Norway subsidence has not been a critical topic or concern, except for at the Ekofisk field. Phillips Petroleum and The Norwegian authorities were quite surprised when, at the end of the 70's, it was discovered by accident that the Ekofisk offshore installations had subsided several meters. It is not the installation itself, but the seabed over the chalk reservoir that is subsiding, probably from the day the production started. The consequence is that all cement between the steel casings has cracked. Thus, there is need for a product that can replace cement to secure well integrity in such circumstances. This research proposal suggests that quick clay, possibly with some chemical additives such as loss circulation materials and/or salt (NaCl and/or KCl) could be used in the anulus as an alternative to today's drilling fluid. The density of virgin quick-clay is approximately 2 g/cc. One may want to decrease that by adding water, of increase the density by adding quartz (2.65 g/cc) and or barite (4.48 g/cc)

Case 2: Further, there are plans to pump substantial amounts of CO_2 into underground formations. In principle, the seabed can rise, with the challenges this may cause. But a bigger concern is the requirement that cement used to plug wells in the area up to 50 years ago, must be able seal and sustain the CO_2 pressure. Thus, with underground forces that potentially may crack the cement plugs, also for permanent plugging of abandoned wells there is need for a product that can replace cement. For this operation, we propose an alternative plug and well slurry based on quick clay or sensitive clay, which largely is composed illite and chlorite. Illite and has low cation exchange capacity (CEC) and considered non-swelling minerals. Equally important is the low salt concentration and high-water content. Such plug will have the same ceiling capacity as the cement, but never go so hard that it will crack or fracture.
Chevron took out a patent in 2004 suggesting using smectite or swelling clay to replace cement to plug wells with. Due to the swelling properties (high CSC value) it has not been a success. The plug starts to swell to quickly and if made to big or long will expand and fracture the well. Quick clay or sensitive clays are mainly illite and chlorite, no swelling clays with low CEC value. Under normal circumstances illite is a solid rock due to its content of salt that holds the clay particles together. Quick clays and/or sensitive clays are found in areas which were once glaciated during the Pleistocene epoch (1.65 million to 10,000 years ago) and have been identified mainly in northern Russia, Norway, Finland, Sweden, Canada and Alaska and may in literature be referred to as Leda and Champlain clays. The clay was deposited under sea water in areas that experienced isostatic rebound. So much rebound that the clay deposits was uplifted above sea-level relatively short time after the ice cap had melted. Rainwater has during the last 10,000 years drained thru a lot of these clay deposits. The leaching has diluted the salt content in the pore water from originally above 30 g/l to less than 2 g/l.
The quick clay has been tested at the University of Stavanger (UiS). The rheology test show that it can be pumped like cement but must be pumped slower. On the other hand, when the temperature increases downhole, cement becomes more and more difficult to pump. Quick

Clay becomes more viscose with increasing temperature. And therefore, easier to pump with increasing temperature.
The clay was dried and mild into powder. The water content was measure to 26% of the weight. The powder looked and felt like dry Portland cement. The powder was mixed with 26% water. And the same tests were run. No change in properties was observed.
Finally, a 3.5-meter vertical plastic tube with 5 cm inner diameter was filled with quick clay. Pressure sensors was placed at each 50cm.
While pumping in fresh water at the base, the whole column of clay started moving up while we exceeded the pressure equivalent to the weight of the overburden column.
No channelling between the clay and the tube of the over-pressure water influx could be observed. So far the initial observations and test results are encouraging.

18:00 - 18:20 ***Discussions***

18:20 - 18:40 ***Olli Ikkala,[1] Hang Zhang,[1] Hao Zeng,[2] Arri Priimagi[2]***

[1]Department of Applied Physics, Aalto University, Espoo, Finland.
[2]Tampere University, Faculty of Natural Science and Engineering, Tampere, Finland
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From responsive and shape memory materials to materials that mimic behaviorists´ classical conditioning

Biological material inspire development of ever more complex materials whose properties respond to external conditions. Therein, as a background, we will first describe the notions of (multi)stimuli responsive and shape memory materials, which have amply been described in the previous literature. Even if they allow switching between different states and functions, their properties do not evolve with time, i.e., their behavior under subsequent applications of stimuli remains the same. A conceptual question can be posed whether materials could evolve or "learn" to become active for a new stimulus. Of course, biological "learning" is a formidably complex phenomenon, which obviously cannot be reproduced using inanimate materials. Still, could materials be algorithmically equipped by responses and functions, inspired by the some of the simplest forms of learning. Therein, classic conditioning is an elementary psychological model. Herein, a new paradigm beyond stimuli-responsive and shape memory materials is shown, as inspired by the classic Pavlovian conditioning.[1] Historically, a dog shows a natural response of salivation upon showing food (unconditioned response and stimulus), whereas no response is obtained upon ringing a bell (neutral stimulus). However, upon conditioning by simultaneously showing food and ringing a bell, the dog learns to salivate also solely upon ringing the bell (conditioned stimulus and response). Therein, simplistically, responsivity and a triggerable memory are needed. We show agarose hydrogels containing spiropyran-based photoacids and tailored Au-nanoparticles that melt upon heating (unconditioned stimulus) but do not melt upon exposing light (neutral stimulus). However, upon simultaneous heating and exposing proper light (conditioning), the system learns to melt by sole irradiation (conditioned). The triggerable memory is formed due to irradiation-driven proton release from the photoacid, resulting to pH change and nanoparticle chaining, and subsequent plasmonic change.[1] Even forgetting can be engineered pushing the system out-of-equilibrium based on systems chemistry approaches.[1] In general, we foresee a wealth of further possibilities to identify different materials systems,[2] combinations of stimuli and different "memory" concepts for this new type of algorithmic functional materials, inspired by psychological behaviors.

1. Zhang, H.; Zeng, H.; Priimagi, A. Ikkala, *Nat. Commun.* 2019, **10**, 3267.
2. Zeng, H.; Zhang, H.; Ikkala, O.; Priimagi, A. submitted.

18:40 - 19:00 ***E. Gustafsson, T. M. Bowden, M. S. Hellsing, A. R. Rennie***

Dept. of Physics & Astronomy, Uppsala University, Sweden

Effect of the plasticizers used in PVC on lipid monolayers and bilayers

Plasticizers are widely used to provide desirable mechanical properties of many polymeric materials. These small molecule additives are also known to leach from the finished products, and this may not only modify the physical properties but the distribution of these materials in the environment and in the body can cause long-term health concerns and environmental challenges. A surprising observation has been that blood stored in PVC bags that are plasticised with di-2-ethylhexyl phthalate resists osmotic stress and is viable for longer than when other additives are used. We have been studying mixtures of plasticizers and lipids to understand the differences between the traditional plasticizer and modern alternatives. We will report on light scattering studies of vesicles and studies of the composition of monolayers that are subjected to different surface pressures.

19:00 - 19:20 ***Leide P. Cavalcanti, Adam Washington, Diego Alba-Venero, James Doutch, Najet Mahmoudi, Steve M. King, Rob M. Dalgliesh, Sarah E. Rogers***

ISIS Neutron Source, STFC, Didcot, Oxfordshire, UK

Sample environment for SANS at ISIS

In this talk, I will present few details of SANS sample environment available at ISIS Neutron Source in UK. A brief introduction of the ISIS spallation source and an example of the wide range of materials investigated by SANS technique, with emphasis on Soft Condensed Matter. Four instruments will be highlighted here: SANS2D, LOQ, LARMOR and ZOOM, with their specific uses: Q range, resolution, intensity, SANS/WANS or simultaneous probes and sample environment.

19:20 - 19:40 ***Mathias Huss-Hansen[1], Matti Knaapila[1], Nada Mrkyvkova[2], Jakub Hagara[2], Peter Siffalovic[2], Martin Hodas[3], Frank Schreiber[3], Jakob Kjelstrup-Hansen[4]***

[1]Department of Physics, Technical University of Denmark, [2]Insititute of Physics, Slovak Academy of Sciences, [3]Institute für Angewandte Physik, Universtität Tübingen, [4]Mads Clausen Institute, University of Southern Denmark

Emerging Possibilities for in situ X-ray Experiments: Real-Time Structural Evaluation of Naphthyl End-capped Oligothiophenes in Organic Thin Film Transistors during Deposition

We discuss emerging possibilities to study vacuum deposited thin films by X-ray diffraction *in situ*. As an illustrative example, we have investigated the thin film growth behavior of 5,5′-Bis(naphth-2-yl)-2,2′-bithiophene (NaT2) and 5,5′′-bis(naphth-2-yl)-2,2′:5′,2′′-terthiophene (NaT3) during the deposition process using real-time grazing incidence X-ray diffraction GIXRD. Films were prepared by vacuum sublimation atop various substrates, including monolayer graphene on 90 nm SiO_2, in a custom-built ultra-high vacuum (UHV) chamber with a 360° cylindrical beryllium window that allows for *in situ* X-ray measurements within realistic deposition time scales. The crystal structure analysis revealed that the preferred orientation of the molecules is dictated by the substrate and that the unit cell underwent significant changes when transitioning from monolayer to multilayer structure. The changes of the unit cell are not readily observed when measuring thin films of corresponding thicknesses *ex situ*, suggesting that the molecules undergo further re-organization/relaxation upon terminating the deposition. From the evolution of the crystal structure, a connection to the film growth mode and kinetics is made. These findings are rationalized based on the surface energies of the studied substrates and supplemented with AFM and helium ion microscopy.

19:40 - 20:00 ***Discussions***

20:00 - 23:00 ***Workshop Dinner***

Monday October 7:

09:30 - 09:45 ***Susete N. Fernandes** (1), Diogo V. Saraiva (1), Beatriz M. De Abreu(1), Ricardo Chagas (1), Pedro L. Almeida (1,2), Paul Grey (1), Luis Pereira (1), Maria H. Godinho (1)*

(1): i3N/CENIMAT, Department of Materials Science, Faculty of Science and Technology, Universidade NOVA de Lisboa, Campus de Caparica, 2829-516, Caparica, Portugal
(2): Área Departamental de Física, Instituto Superior de Engenharia de Lisboa, R. Conselheiro Emídio Navarro, 1, 1950-062, Lisboa, Portugal

Cellulose Nanocrystal Films from Drop Casting: Easy Method to Control CNCs' Deposition

Cellulose nanocrystals (CNCs) solid films, produced from lyotropic aqueous suspension, present remarkable optical properties such as iridescence, selective reflection of left circularly polarized (LCP) light and transmission of right circularly polarized (RCP) light, mimicking what is observed in the exocuticle of certain insects [1,2]. Recently, we showed that a photonic structure of an iridescent CNC film with a nematic liquid crystal micrometric-layer between two left-handed CNCs chiral nematic layers, acting as a half-wave phase retarder, reflects both RCP and LCP light. Furthermore, this optical response can be tuned by temperature variation or application of an external electric field [3].

When a drop of CNCs suspension is casted into a circular solid film, the solvent evaporation is mainly controlled by flow dynamics (convective macroscopic flow) within the sessile droplet and the three-phase contact line is enriched in CNCs. This led to a ring shape deposition pattern usually referred to as "coffee-stain" that presents different structural coloration along the film's radius [4]. Nevertheless, such droplets can be integrated into transistor devices capable of sensing distinct polarization states of circularly polarized light [5]. In this work the optical response of millimetric drop-casted films was controlled by adjusting the substrate wettability and drying condition with successful suppression of the "coffee-stain" effect. The reflection/transmission of circularly polarized light (LCP and RCP light) within the film's diameter was analyzed and distinct LCP and RCP light reflection/transmission was observed within each ring. The structural arrangement of the nanoparticles within the structures' cross-section indicates distinct arrangements in the CNCs' chiral nematic pitch and helicoid orientation.

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09:45 – 10:00 ***Ana C. Trindade[1,*], Miguel Carreto[2], Susete Fernandes[2], Matthias Daab[3], Josef Breu[3], Maria Helena Godinho[2], J. O. Fossum[1]***

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Tuning Photonic Properties of Cellulose-Clay Nano Structures

Fascinating structural colors, which varies with the angle of observation (iridescent) can be seen in animals (ex: feathers of the peacock), as well as in plants (ex: polia berries). These colors are very vivid and remain in time due to periodic structures at the nanoscale, which are at their origin. Mimicking these iridescent colors with natural materials is one of the main targets of this work.

In fact after solvent evaporation, aqueous suspensions of cellulose nanocrystals (CNCs) can be used to produce iridescent solid films. [1,2] These films have a left-hand helicoidal structure, which reflects left circular polarized (LCP) light and transmits right circular polarized (RCP) light.

Clay particles can present lamellar periodic organizations in the solid state, while aqueous suspensions of clay biaxial platelets can form nematic uniaxial liquid crystalline phases.[3] In this work mixed suspensions of CNCs and high-aspect ratio sodium-fluorohectorite (NaFh) were used to develop nanostructured surfaces with engineered optical functionality for coloration and reflection of light. A midified clay, Cesium double layer fluorohectorite, was also used in some of the studies.

Aqueous solutions, with various CNCs/Clay (NaFh) ratios, were administrated dropwise on different substrates and the water solvent was allowed to evaporate under different conditions. The solid "pancakes" obtained were characterized by different techniques and then set side by side, with the intention of selecting the best ratio and conditions to be used in the preparation of solid thin films. The phase diagram of the system NCC/NaFh was obtained.

The "pancakes" as well as the solid films prepared show iridescence. It is possible to adjust the photonic properties of these solid iridescent films, and a green shift was obtained for higher concentration of clay.

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10:00 – 10:15 ***<u>Ana C. Trindade</u>[1], Ana P.C. Almeida[2], João Oliveira[2], João P. Canejo[2], Susete N. Fernandes[2], Jon-Otto Fossum[1], M. Helena Godinho[2]***

[1] Department of Physics, Norwegian University of Science and Technology, Trondheim, Norway. [2] i3N/CENIMAT, Department of Materials Science, Faculty of Science and Technology, Universidade NOVA de Lisboa, Campus de Caparica, Caparica, Portugal.

Cellulose-based Membranes for Effective Capture of Oil Microdroplets in Water

Disasters are sometimes associated with the transportation of oil across big distances using oil tankers. When an accident occurs large volumes of oil are spilled into the ocean and have to be collected from the water. However, the traditional oil-removing methods fail to collect the micro-sized droplets of oil originated by the mechanical action of the waves.[1] Oil/water separation is an important field, not only for scientific research but also for practical applications aiming to resolve industrial oily wastewater and oil-spill pollution, as well as environmental protection [2,3].
This work focusses on the recovery of oil microdroplets suspended in water, using a manufactured cellulose acetate (AC) non-woven electro-spun membrane coated with different patterns of cellulose nanocrystals (CNCs). Efficiency tests show that these membranes can remove up to 80% of the oil microdroplets present in a water emulsion.
The imprint the different designs of the CNCs layer was performed using screen-printing and the adhesion of the CNCs with the fibers of AC was promoted by a thermal treatment. The removal of the oily micro droplets is achieved when a water/oil emulsion flows through the membranes, due to the hydrophilic character of the AC fibers, and the CNC-coated regions collect micro droplets of oil, due to its oleophilic character.
This work demonstrates that it is possible to produce efficient all-cellulosic composite membranes for the capture of micro-oil droplets dispersed in water, with easy tailoring of the ratio CA membrane and annealed NCC allowing to maximize the micro-droplet oil collection and at the same time the water flow. More, the combination of annealed NCC with non-woven electrospun membranes opens the door to a low-cost environment-friendly method of treating polluted ocean and waste-oily waters.

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10:15 – 10:30 ***<u>Etien Martinez</u>[1], Tommy Kristiansen[1], Barbara Pacakova[1], Jaako Timonen[2], Paul Dommersnes[1], Jon Otto Fossum[1]***

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Active turbulence/Vortex array transition in Quincke rotating beads.

Insulating particles suspended in a carrier liquid may start to rotate with a constant frequency when subject to an electric field. This phenomenon is known as the Quincke rotation instability. A single isolated rotating particle exhibit no translational motion at low Reynolds number, however interacting rotating particles may move relative to one another or in the presence of a wall. Here we present experimental results on collective dynamics and self-assembly in a suspension of electro-rotating granular particles. Depending of the value of the electric field or the concentration of the particles is it possible to find different behavior like gas or vortices. We perform Particle Image Velocimetry in order to compute the vorticity and velocity of the systems and also simple tracking procedure were performed as a complementary way of characterization. One of the main striking results found is that the size of the vortices can be controlled with the electric field. This fact points out to future applications of the systems, for example, as photonic materials. Also we find a transition from active turbulent behaviour to a orderded vortex array.

10:30 – 10:45 ***<u>Hector Urra</u>[1], Gaspad Junot[1], Paul Dommersnes[3], Jochen Arlt[2], Vincent Martinez[2], Jon Otto Fossum[3], Eric Clément[1]***

[1]Laboratoire de Physique et Méchanique des Milieux hétérogènes (PMMH), CNRS, ESPCI Paris, PSL Research University, Sorbonne Université, [2]Scottish Universities Physics Alliance (SUPA), School of Physics and Astronomy, University of Edinburgh, Edinburgh EH9 3FD, United Kingdom. [3]Norwegian University of Science and Technology (NTNU), Trondheim, Norway.

In this work, we analyze the movement of bacteria with a 3D Lagrangian system in suspensions with complex fluids, for the building of biobarrier, As a complex medium, we use Carbopol. The rheology of this polymer has been studied, which will be the complex medium where the bacteria will swim. With the preliminary characterization of the clay, and swimming characteristics which has yield interesting results.

First, the pH evolution of the Carbopol (C) plus Mobility Buffer (MB) solution was monitored, finding that it does not change over time. I presented a simple experimental model of swimming bacteria in a complex fluid where the rheology could be tuned progressively from a law power fluid to a gel following an Herschel Bulkley rheology.

Second, using the Lagrangian track system in 3D, I find that adding the polymer, one can induce a progressive stoppage dynamics ending in the building of a motility barrier.

Surprisingly in the gel phase the swimming velocities even deep in the gell phase was found constant. The translational diffusivity strongly drops at the gel onset and decays by orders of magnitudes when Carbopol concentration is increased.

10:45 – 11:00 ***Discussions***

11:00 – 11:15 ***<u>Paulo H. Michels-Brito</u>[1], Antonio M. Gasperini[2], Lina Mayr[3], Ximena Puentes-Martinez[4], Rômulo P. Tenório[5], Leide P. Cavalcanti[6], Koiti Araki[7], Josef Breu[8], Kenneth D. Knudsen[1,4], Jon Otto Fossum[1]***

[1]Norwegian University of Science and Technology – NTNU, Trondheim, Norway, [2]Brazilian Synchrotron Light Lab – LNLS, Campinas, Brazil, [3]University of Bayreuth, Germany, [4]University of Boyacá, Colombia, [5]Northeast Regional Center of Nuclear Sciences, Recife, Brazil, [6] ISIS Neutron Source, STFC, Didcot, Oxfordshire, UK, [7]University of Sao Paulo – USP, Sao Paulo, Brazil, [8]Institute for Energy Technology – IFE, Kjeller, Norway

We present a study on the structural organization and mechanics proprieties of clay colloidal particles at the air-liquid interface where we monitor the film self-assembly and stability. Clays and modified clays are studied on a Langmuir Trough using Grazing Incidence techniques (GID and GIXOS). The film thickness and in-plane organization are monitored to determine the efficiency of the mechanisms for adsorption control on different interfaces. The results are important for particle coating studies and for the development of new methods of assembling clay colloidal particles on liquid surfaces/interfaces. This effect is currently much studied in relation to Pickering emulsions where particle coatings on droplets effectively prevent droplet coalescence and produce very stable surfactant-free emulsions. The adsorption of colloidal particles at the surface of liquid droplets [1] has applications in several areas like pharmaceutics, oil and gas sector, not only for encapsulation properties using surfactant-free emulsions, but also for the features of manipulation of the colloidal particles such as clays through external forces, like electric field [2]. Here we show that we can study the ordering of clay nanoparticles in a confined two-dimensional surface like a Langmuir Trough and recreate the process of Janus clay-platelets preparation using synthetic Fluorohectorite clays [3].

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11:15 – 11:30 ***Leander Michels[1], Yves Méheust[2], Mario A. S. Altoé[3,4], Éverton C. dos Santos[1,5], Henrik Hemmen[1], Roosevelt Droppa Jr.[6], Geraldo J. da Silva[3], Jon O. Fossum[1]***

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Water vapor diffusive transport in a smectite clay: Cationic control of normal versus anomalous diffusion[1]

The transport of chemical species in porous media is ubiquitous in subsurface processes, including contaminant transport, soil drying, and soil remediation. We study vapor transport in a multiscale porosity material, a smectite clay, in which water molecules travel in mesopores and macropores between the clay grains but can also intercalate inside the nanoporous grains, making them swell. The intercalation dynamics is known to be controlled by the type of cation that is present in the nanopores; in this case exchanging the cations from Na+ to Li+ accelerates the dynamics. By inferring spatial profiles of mesoporous humidity from a space-resolved measurement of grain swelling, and analyzing them with a fractional diffusion equation, we show that exchanging the cations changes mesoporous transport from Fickian to markedly subdiffusive. This results both from modifying the exchange dynamics between the mesoporous and nanoporous phases, and from the feedback of transport on the medium's permeability due to grain swelling. An important practical implication is a large difference in the time needed for vapor to permeate a given length of the clay depending on the type of intercalated cation.

[1]Physical Review E 99, 013102 (2019).

11:30 – 11:45 ***Konstanse K. Seljelid[1], O.T. Neto[1], V.V. Liljeström[1], I. Fjelde[3], M. Carvalho[4], J.O. Fossum[1]***

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Microcapsule controlled gelation in channels and pores such as those in petroleum-reservoirs

High water production in mature oil reservoirs is a major issue in oil recovery. It leads to more wear on the equipment, higher expenses related to the post processing of the produced oil and water, and maybe even abandonment of the well. To mitigate this problem, several methods within enhanced oil recovery (EOR) have been developed. One of these methods is a gel treatment. The gel treatment aims at blocking preferential paths in which the water flows, diverting it into unswept areas of the reservoir. However, limited control over the gelation time may result in poor placement of the gel, blocking areas close to the injection site or blocking oil rich areas.

To improve control of the gelation time, microcapsules carrying gelant activators have been suggested as a possible solution. The laboratory production of such microcapsules can be achieved using a microfluidic device, yielding precise control over capsule size and shell thickness. Tuning the microcapsule properties, an external trigger such as pH, temperature or salinity could rupture the capsule, enabling a more precise placement of the gel in the porous medium. In this project we will study basic phenomen involves in capsule transport through microporous media, including controlling capture rupture, e.g. by time, pH, salinity, temperatire or other..

The project is granted from the Research Council of Norway, Petromaks2: "Nanofluids for IOR and Tracer Technology", project number: 268252.

11:45 – 12:00

Kristoffer W. Bø Hunvik[1*], Leide Cavalcanti[2,3], Martin Riess[4], Patrick Loch[4] Konstanse K. Seljelid[1], Vegard Josvanger[1], Dirk Wallacher[5], Roosevelt D Droppa[6], Barbara Pacakova[1], Paulo Brito[1], Kenneth D Knudsen[1,2], Josef Breu[4], Jon Otto Fossum[1]

[1]Department of physics, NTNU - Trondheim, Norway, [2] IFE – Kjeller, Norway, [3]ISIS Neutron and Muon Source, UK, [3]Lehrstuhl für Anorganische Chemie, University of Bayreuth, Germany, [4]Helmholtz-Zentrum Berlin, Germany, [5]Centro de Ciências Naturais e Humanas,Universidade Federal do ABC - Santo André SP, Brazil

Carbon dioxide capture and retention by synthetic Fluorohectorite (Fh) clay

Experiments and simulations have shown that CO_2 (like H_2O) intercalate in smectite clays, both in the supercritical ($scCO_2$) [1], and in the gaseous/liquid phase [2,3,4]. Understanding how clay swells and adsorbs CO_2 is vital for CCS and potential capturing applications, e.g. filtering of combustion gases.
By investigating synthetic smectite clays we can distinguish how different properties lead CO_2 adsorption without the influence of impurities. We have investigated temperature and pressure dependence of the adsorption with different interlayer charge balancing cations and with different charge of the clay platelets. Our recent studies show crystalline swelling of Ni-Fh within seconds in response to CO_2 exposure, and we find that neither Cs-Fh, Ca-Fh and Ba-Fh show any sign of crystalline swelling when exposed CO_2. By investigating CO_2 capture in Ni-Fh for three different clay layer charges, 0.3, 0.5, 0.7 per formula unit ($Si_4O_{10}F_2$) respectively. We find that Ni-Fh display crystalline swelling at 5-10 bar (0.3 layer charge), 10-15 bar (0.5 layer charge) and 30 bar (0.7 layer charge) at room temperature.

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12:00 – 12:15 ***Osvaldo Trigueiro Neto[1], V. V. Liljeström[1], K. K. Seljelid[1], L. Mayr[2], J. Breu[2], P. G. Dommersnes[1], J. O. Fossum[1]***

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Exfoliation of Clay Nanosheets

The goal of this project is to develop ways to encapsulate/wrap assemblies of nanoparticles, to "protect" them, and understand the processes involved. Such an objective can, for example, be carried out by encapsulating materials in shells consisting of colloidal particles [1] or nanosheets [2].

To wrap droplets or nanoparticles with ultrathin sheets of clay, exfoliation of clay into single sheets must be achieved in a controlled manner. For that purpose, a precise study of how this process occurs is required. With the "wrapping goal" in mind, analysis on clay behavior with different salt concentrations were done.

Dialysis experiments were done with Na-Flourohectorite high-aspect ratio clay and NaCl solutions to map out what role salt concentration plays on the exfoliation process. Here we present the initial results and the possible outcomes of this study.

The project is partly motivated by the prospect of enabling protected (from environment) transportation and controlled release of magnetic tracer nanoparticles in oil-reservoirs. The project is granted from the Research Council of Norway, Petromaks2: "Nanofluids for IOR and Tracer Technology", project number: 268252.

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12:15 - 12:30 ***Discussions***

12:30 - 14:00 ***Workshop Lunch***

14:00 - ***Departure***