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# Multifunctional Architectures based on Lipid Assemblies and Magnetic Nanoparticles

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Amphiphilic self-assemblies are inherently responsive to slight variations of control parameters, such as pressure, temperature, ionic strength, and seemingly subtle changes can produce cascade effects from the molecular scale to the mesoscale. The possibility to induce such transitions remotely and locally, e.g. through application of magnetic fields, has sparked the interest in architectures as magnetoliposomes, where superparamagnetic nanoparticles (SPIONs) are embedded in the bilayer, in the internal pool of liposomes, or bound to their surface. When exposed to alternating magnetic fields, a local temperature gradient is produced which can induce liposomal leakage, without disruption of the lipid vesicle. This feature lends itself to application of these hybrid architectures in fields such as targeted drug delivery.

Here we extend this concept to the design of more complex architectures, where we include hydrophobic and/or hydrophilic nanoparticles in lamellar and non lamellar mesophases, such as bicontinuous lipid phases, and in natural extracellular vesicles.

The structural arrangement at the nanoscale is assessed through Small Angle Scattering Techniques, such as the effects on thermotropic properties. The diffusion of lipids and of confined guest molecules is followed by Fluorescence Correlation Spectroscopy, which allows demonstrating the on-demand release of hydrophobic or hydrophilic payloads.

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## ***Observation and modelling of unexpected colloidal behaviour : a source of « greener » processes ?***

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*Non-covalent binding beyond the first neighbour is considered in the last forty years including « hidden » entropic effects designed with their different names : solvation force, depletion forces and finally Onsager transitions. In all these cases entropy and enthalpy compensate somehow and are the origin of unexpected colloidal behaviour. We will show in this lecture how the experimental investigation of nanostructure AND colloidal interaction of a priori “strange” behaviour led to unexpected applications and progresses in recycling:*

*-Mixing an anionic amphiphile in acidic form and a cationic amphiphile in hydroxide form in slight mismatch of stoichiometry sometimes does **not** produce the ill-defined flocculated precipitate. Phase diagram, osmotic pressure and molecular interaction measurement led to the understanding of the route of formation of giant ultra-stiff colloids(Carrière et al., 2009): Those are used without being theoretically understood in stable concentrated “creams” in routine pharmaceutical application, but led also to more efficient and alcohol-free personal care fluids, while the “green” water-lubrication usages finally did not emerge.*

*- In standard processes to purify rare earths from ore or from tentative recycling of electrical windmills - that some people think as being more renewable than nuclear energy from nuclear waste- two extractants are always used in synergy, without any theory (yet) to predict the selectivities and the controlled free energy of phase transfer is of the order of 5kJ/Mole while supramolecular as well as organo-metallic chemistry consistently measure **five time more** intense “complexation”, at least 20 kJ/Mole. We show how refined scattering experiments and taking into account entropy in all aspects allows to identify “weak aggregation” regime that is at the basis of all efficient liquid-liquid extraction processes of electrolytes, leading to greener “ienaic” processes making full usage of the elusive “synergy” using less acid for the same amount of purified products(Zemb et al., 2015).*

*-Finally, wood dissolution and treatment of cellulosic material in order to obtain are most of the time using concentrated acids or toxic ionic fluids. Patented processes such as “wood-protect”™ have been found by accident without reference to any predictive theory. We will show how the equation of state has been set by including elastic term in the equation of state previously used for DNA gel swelling (Bertinetti, Fratzl, & Zemb, 2016), in order to obtain a general master equation that can be used to understand the effects of wood pre-treatment, and to guide experimental plan in the search for “greener” processing of cellulosic biomass.*

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# **Effect of volume fraction of water, oil, surfactants and silica particles on structural dimensions in a liquid-crystal system**

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The structural dimensions of a lyotropic liquid crystal system consisting of water, *para*-xylene and Triton X-100 were investigated as a function of volume fraction of the water, oil and surfactant components. Replacement of the chosen surfactant (Triton X-100) with a more crystalline surfactant (oleic acid) was also investigated for its effect on the mesophase structure, as was the introduction of hydrophobic and hydrophilic silica particles of two sizes (10 and 20 nm). Small-angle neutron scattering was conducted to analyse structural features, while rheological measurements were undertaken to see the effect of the oleic acid on the flow of the systems.

In line with expectation, the interlamellar distances in the LC system were found to be inversely proportional to the volume fraction of surfactant (but not simply dependent on the volume fraction of water or oil). Doping with oleic acid and silica particles was found to increase the viscoelasticity of the system without altering the interlamellar distances, likely due to a rigidification of the lamellar bilayers. In one instance, silica particles were found to cause a partial phase transition, adding to a question raised in the literature on where such particles are likely to exist in an LC system.

These results allow for the fine tuning of a liquid crystalline phase with desired structural dimensions and viscoelastic properties via simple changes in the volume fraction of each component, and give further clues to an ongoing mystery related to where guest particles exist within liquid crystalline systems. By further understanding how to control the structural and flow properties of liquid crystal systems, and the interrelationship between them, the applications of liquid crystals in materials synthesis,<sup>1</sup> micro/nanofluidics,<sup>2</sup> and biological models (e.g. for intracytotic processes)<sup>3</sup> may be bolstered and advanced.

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## Sessile droplet growth on fiber by solvent exchange process

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Micromodel sessile droplets are of great interest for their relevance in board droplet-based technologies. Deriving from ‘Ouzo effect’, the solvent exchange process is a simple bottom-up approach to produce surface nano-/micro-droplets by the nucleation and growth mechanism. The oil oversaturation pulse is created as a good solvent (ethanol) for the oil displaced by a poor solvent (water) in the flow cell. In this work, different from the general strategies to set the 2D substrate as a channel wall, we investigate the droplet formation on a single 10  $\mu\text{m}$  hydrophobic fiber (1D substrate) at the middle of the channel, perpendicular to the flow direction. The experimental results demonstrated a prominently different growth mode of droplet on fiber and complicated local flow situation during solvent exchange process. The profiles of velocity and oil oversaturation from Comsol Simulation reveals that the special droplet growth mode on fiber may be attributed to the local flow patterns and concentration gradient, those induced by the boundary conditions and geometry variation during droplet growth.

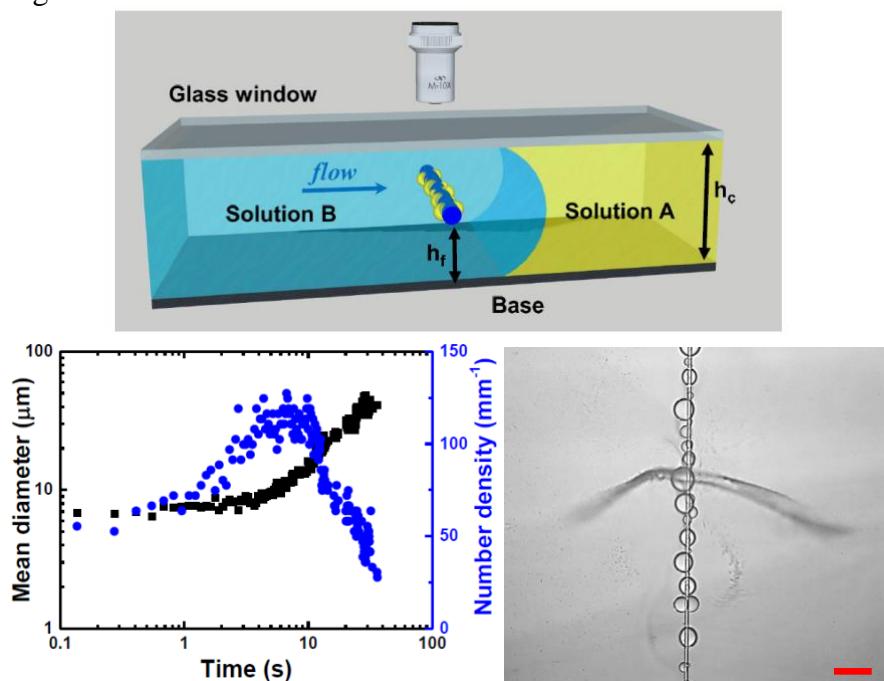


Figure 1. The sketch of the experimental setup is provided. After the droplets coalescence happened, the droplets kept growing at a faster speed, which was quite different from the growth mode on 2D substrate. This special growth mode is relative to the local flow vortex induced by the boundary conditions and spatial geometry. Scale bar: 100  $\mu\text{m}$ .

# **Structural basis for mixed stabilisers and salt effects on the performance of polymeric stabilisers at the oil-water interface**

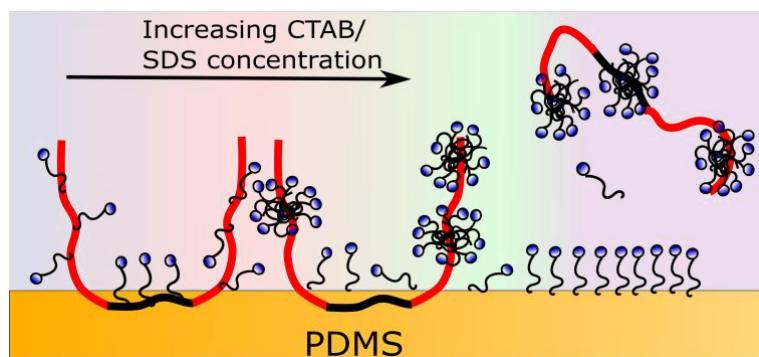
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Industrial formulations commonly consist of mixtures of polymers and surfactants in the presence of electrolytes. A typical hair conditioner formulation, for example, contains low molecular weight poly(dimethyl siloxane), PDMS oil, emulsified by Pluronic polymers (including F127) in a buffer solution. The complex interactions between the polymers, surfactants and the electrolytes at the interface will affect the performance of the final product. In this study we utilise covalently anchored PDMS chains as an immobilised oil layer to study the structures of Pluronic F127 adsorbed at the oil/water interface in a salty environment as well as the interfacial structures of mixed stabiliser systems F127/SDS and F127/C<sub>16</sub>TAB.

Results from neutron reflection and QCM highlight that the structures information at the oil/water and air/water interface differ. It was found that F127 forms a loosely packed thin layer at the PDMS/water interface, while addition of SDS and CTAB causes desorption of F127 from the interface. Mixed stabilisers exhibit competitive adsorption behaviour where low concentrations of surfactants cause swelling of the PEO segments of F127 and high concentration surfactants completely solubilise the F127. The effect of salt on the adsorption behaviour was also investigated. Both UV-Vis absorbance and QCM studies showed addition of different sodium halide salts leads to dehydration and collapse of the adsorbed F127 layer and that the effect is larger for more kosmotropic anions. These results help elucidate the mechanism of adsorption of amphiphilic molecules, their function as colloidal stabilisers at the oil/water interface and guide the selection of surfactants for industrial formulations.



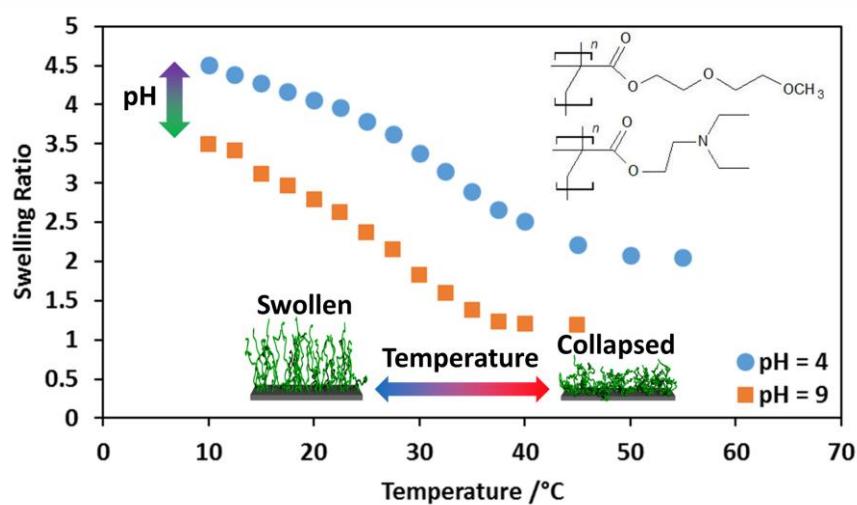
# Synthesis and characterisation of multi-stimulus responsive polymer brushes

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Polymer brushes are ultrathin coatings wherein polymer chains are end-tethered to an underlying substrate at a sufficiently high density so as to force the chains to extend from the surface. Smart polymer coatings consist of stimulus responsive polymers for which the conformation of the polymer chains is influenced by stimuli such as temperature or pH, allowing for the modification of interfacial properties of a material. The copolymerisation of the thermoresponsive 2-(2-methoxyethoxy)ethyl methacrylate (MEO<sub>2</sub>MA) with the pH responsive 2-(diethylamino)ethyl methacrylate (DEA) via ARGET ATRP has resulted in the formation of a brush coating which responds reversibly to both pH and temperature. P(MEO<sub>2</sub>MA-*stat*-DEA) brushes (90:10 and 80:20 mol%) were synthesised and the thermoresponse examined using ellipsometry, atomic force microscopy (AFM), contact angle measurements, and quartz crystal microbalance with dissipation (QCM-D) in both the charged (pH = 4) and uncharged (pH = 9) regimes. The presence of chargeable sites within the polymer chain allows for an *in situ* avenue through which the temperature transition of the brush can be modified. In the charged state, the brush remains in a swollen state until higher temperatures when compared to the uncharged brush as shown by ellipsometry. Brush thickness also increases in the presence of charge.



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# **Adhesive Emulsions: Using Atomic Force Microscopy and Microfluidics to investigate the link between drop interactions**

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Adhesive emulsions are systems where the drops in solution have attractive interactions such that they stick or aggregate and remain stable rather than coalesce with contact. These interactions provide beneficial properties that can be used to control processes such as gel formation, coating deposition and fluid microstructure. Such processes are critical in the development of complex fluids useful for personal care products, formulated food products, pharmaceuticals and paint. Polymer surfactant systems are commonly used in formulated products to induce adhesive behaviour, due to their ability to control both solution and surface properties, as well as their potential to provide structure to form aggregated emulsions. This study investigates the adhesive interactions between tetradecane drops in SDS and PVP using both direct force measurements on an atomic force microscope (AFM) and microfluidic drop chaining observations. The AFM results provide quantitative measurements, which can be correlated to the overall force profiles of the microfluidic observations to provide insight into the adhesive interactions in drops under equilibrium and dynamic flow conditions

## **Effect of embedment of DNA on cubic phase**

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The cell membrane environment, which is ubiquitous in biological systems, provides an exceptional model to devise “smart nanostructures” based on the molecular self-assembly of biological macromolecules such as carbohydrates, lipids, nucleic acids, and proteins. Amphiphilic biomolecules such as lipids can self-assemble into nanostructures of well-defined geometry.<sup>1</sup> Lipid-DNA self-assembly and their application in gene delivery through lipid cell membrane is a significant field of research in biological science. Cationic surfactants can make the DNA in more condensed form, which could increase the crossing rate through the membrane with highly protective environment<sup>2</sup>. To date the assembly of DNA double helix and cationic or zwitterionic surfactants has been studied and which can form different to lyotropic liquid crystalline structures including lamellar, normal and reverse hexagonal and cubic phase.<sup>3</sup> The lipidic cubic phase offers a range of advantages compared to liposomes, which have been more commonly used for gene delivery. The intrinsic properties of the bicontinuous cubic phases make it a highly efficient lipid vector for endosomal escape and cellular delivery of siRNA. For example, the modified cuboplexes may improve cellular uptake due to a high curvature which can enhance the fusion with the cell membrane. Previous research by Leal *et al.* has demonstrated the potential of PEGylated cuboplexes to “deliver siRNA and specifically knockdown genes that surpass those achieved by traditional lipoplex systems.<sup>4</sup>

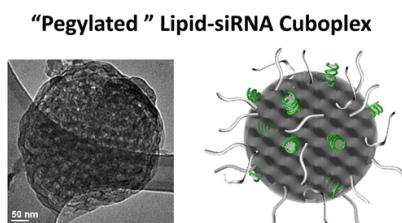


Fig 1 Schematic representation of the spherical PEGylated cuboplex particles with internal Im3m cubic structure

Our main focus is on cationic-cuboplexes, complex mixture of different lipidic materials which provided us different size of lipidic cubic phase. On the basis of the size of cubic lattice parameter, it allowed us to load the different size of DNA molecules. We also investigated the structure-property relationships between the lipid nanostructure and the encapsulated DNA molecules by using the High throughput (HT) formulation and characterization techniques.

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# Sensing Local Stresses with Mechano-Responsive Polymer Structures

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We utilize mechano-responsive polymer structures as spatially resolving force sensors. By labelling with fluorophores, the local mechanical deformation of the polymer can be translated into an optical signal that can be read out with confocal microscopy.

Recently, we applied a mechano-responsive polyelectrolyte brush to resolve the stress distributions in the contact area of gecko-inspired microstructures. The polyelectrolyte brush was synthesized from a cationic monomer and labelled with a fluorescent dye. The charges of the polyelectrolyte are able to quench the dye. Accordingly, compressive and tensile stresses could be read out from locally decreased and increased fluorescence intensity, respectively.<sup>1</sup> For the quantification of the stresses, the calibration with Soft Colloidal Probe AFM is possible. In earlier work, the sensitivity upon compression was determined to 10 kPa with a spatial resolution better than human skin (1 μm).<sup>2</sup>

With polymer brush layers being limited to a two-dimensional, planar geometry, we aim for transferring the mechano-response principle to polymer networks as an extension to the third dimension.

Currently, we focus on designing mechano-responsive microparticles. In need of mechanically defined properties, we utilize droplet microfluidics for controlling the homogeneity of a polymer network in spherical hydrogel microparticles.<sup>3</sup>

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# Nanocellulose Hydrogels

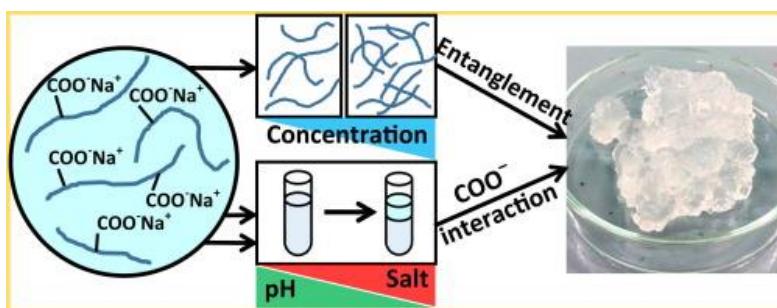
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Hydrogels are fascinating soft materials consisting of polymeric networks which entrap substantial amounts of water (~99wt. %). Cellulose, the most abundant polymer occurring in nature, can be processed into nanofibers and biodegradable, biocompatible hydrogels. Carboxylated nanocellulose fibres which form hydrogels present interesting potential for high value products, particularly in food and biomedical applications. However, characterisation and fundamental understanding of their gelation mechanism are required to efficiently engineer their property for specific applications.

Here, TEMPO-oxidised cellulose nanofibres are used to produce hydrogels. Rheological measurements were employed to characterise the structure and understand the mechanism of hydrogel formation. We have determined that the formation of gels and their properties are heavily influenced by fibre concentration. Moreover, gel colloidal stability is dependent on factors such as pH and ionic strength, affecting swelling and bond strength. The combination of critical length-scale and high surface charge density of the nanocellulose fibres allows the formation of stable colloidal gels. Through control of this structure-property relationship, we are modifying and functionalising hydrogels by optimising fibre content, and their response to environment (ionic strength and pH).



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# Characterization of Flocculated Dispersions Using Acoustic Backscatter Systems

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Acoustic backscatter systems offer a flexible technique to measure dispersion concentration and particle size, through inversion of the return echo voltage response of pulse-echo signals in the MHz range. Although there have been recent developments<sup>1</sup> in how backscatter models can be broadened from current common applications in marine environments for non-cohesive particle systems, there is little data available for the Rayleigh regime ( $ka < 1$  where  $k$  is the acoustic wavenumber ( $\text{m}^{-1}$ ) and  $a$  is the particle radius (m)) and for aggregated particle systems. For these small particle systems direct inversion is not applicable as they attenuate to a significant degree and have a particle structure that is not well understood. Such systems are of great interest for engineering applications such as in the mining, wastewater treatment and nuclear industries and so understanding the limits of current acoustic modelling techniques would be invaluable to allow for optimisation of settling and sediment transport processes.

The Ultrasonic Array Research Platform II, developed at the University of Leeds<sup>2,3</sup>, was used to collect acoustic backscatter data for three sizes of glass particle species (Guyson - Honite-12, Honite-16 and Honite-22<sup>4</sup>) and Calcium Carbonate (Calcite) flocs (~150  $\mu\text{m}$ ) produced with anionic, polymeric flocculants (SNF Chemicals – AN934SH/ AN945) suspended in water. The calcite flocs' size and fractal dimension (the power to which the floc mass scales with size) were measured using a Mastersizer 2000T so that measured acoustic parameters could be normalised to structure related particle properties such as the specific gravity. Measurements were taken in an agitated, recirculated, homogeneously mixed calibration tank over a 0.3 m range using transducers arranged radially from the centre of the tank facing perpendicular to the base. The transducers, of which there were eight Olympus and two Sonatest probes, were excited at frequencies of 2, 2.25 & 2.5 MHz (Olympus NDT V323 and Sonatest PIM 3725 – Central frequency 2.25 MHz) and 0.85, 1 & 1.15 MHz (Sonatest PIM 501 – Central frequency 1 MHz). Suspension samples were taken at multiple depths for offline concentration analysis and comparison with the in-situ ultrasonic concentration profiles measured at several concentrations up to 133.7  $\text{kg m}^{-3}$ . A novel calibration routine, based on the method proposed by Rice *et al*<sup>5</sup>, was used to calculate the acoustic attenuation coefficient for each particle size to allow for the inversion of concentration profiles for the four particle systems. Once the critical backscatter and attenuation parameters were measured, parameterisation of the voltage-distance profile, converted from the backscattered acoustic signal, was performed to measure dispersion concentration. Both iterative-implicit single and dual-frequency inversion models<sup>5</sup> were applied and it was seen that accurate concentration profiles up to ~50  $\text{kg m}^{-3}$  could be produced (Fig. 1). Subsequent errors in particle concentration profiles were studied through sensitivity analysis on the transducer constant. Importantly, this work details the flexibility of using single transducers excited at multiple frequencies to enable dual-frequency inversions, while limitations in terms of particle size, insonification frequencies and solids concentration are discussed for each model, with a focus on theoretical limitations as particles approach the Rayleigh regime.

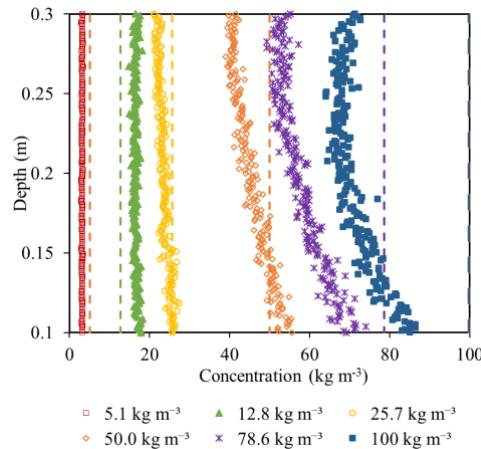


Figure 2. Backscatter concentration profiles produced from voltage profiles using experimentally determined attenuation constant for Honite-16 at 2.25 MHz (single probe) (dotted lines indicate nominal concentration)<sup>6</sup>

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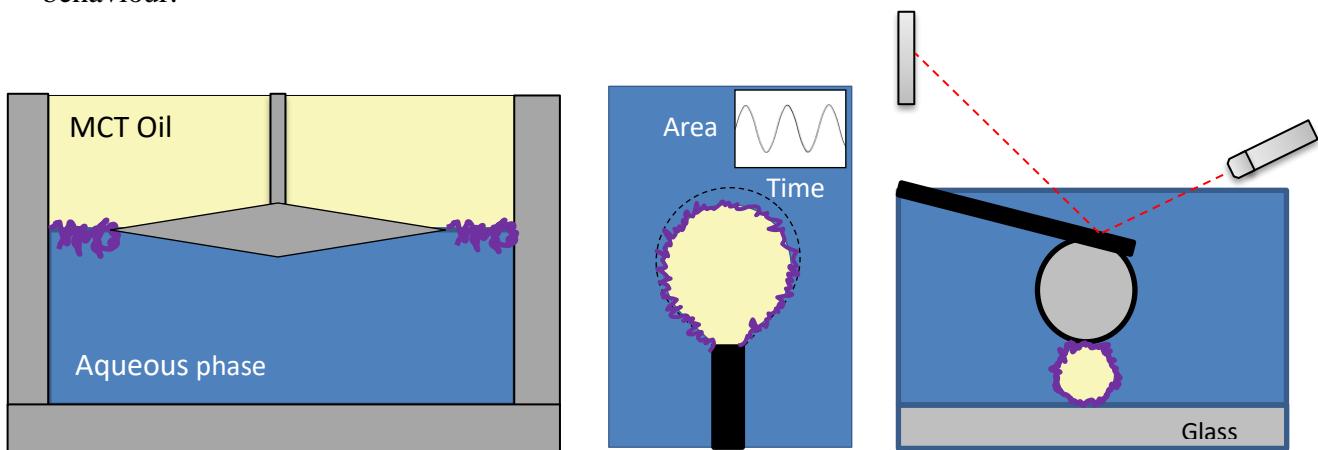
# Viscoelastic characterisation of an emulsion drops coating via capsule compression

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Characterising the viscoelastic properties of the interfacial films between oil and water has traditionally been done with interfacial rheological techniques such as bicone shear rheology<sup>1</sup>, and pendant drop dilatational rheology<sup>2</sup>. These techniques characterise the behaviour of the interfacial film independent of the drop geometry in an emulsion or the impact of the film properties on drop interactions. We use colloidal probe Atomic Force Microscopy (AFM) to directly compress and dwell on individual emulsion droplets with viscoelastic coatings. The response to deformation of these drops exhibit viscoelastic relaxation behaviour more complicated than traditional interfacial rheological techniques. Through numerical modelling of the compression and relaxation process, we extract viscoelastic parameters of these films on the microscale and correlate this with traditional rheological techniques. We analyse an MCT oil/water interface covered by the whey protein,  $\beta$ -lactoglobulin, then crosslink this interface with glutaraldehyde to observe the changes in the interfacial film and rheological behaviour.



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# Electrostatic Formation of Polymeric Stabilised Liquid Marbles

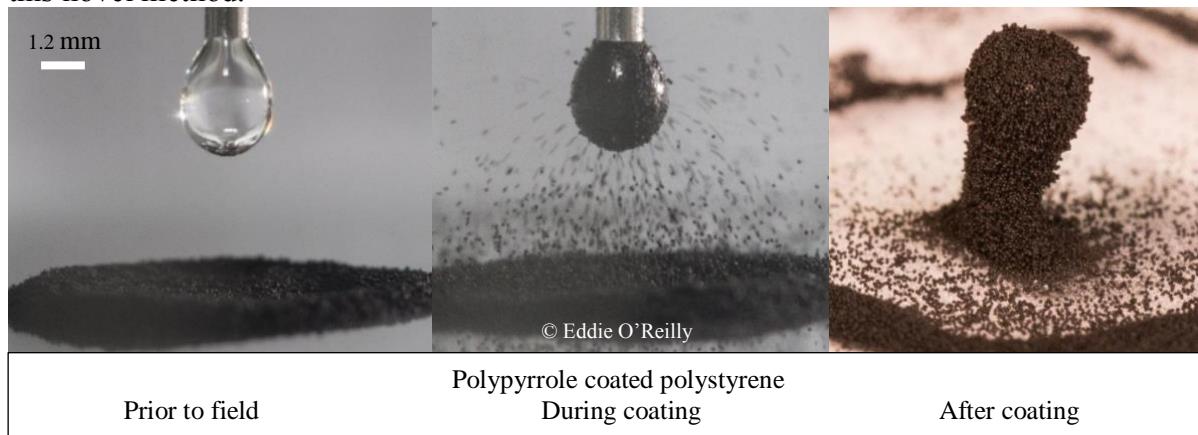
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Small liquid droplets encapsulated and stabilised by hydrophobic powders which exhibit low friction on surfaces, known as liquid marbles, have increased in research interest in the last 15 years. Traditionally, liquid marbles have been produced by rolling the liquid droplet on top of a bed of particles, resulting in the particles attaching at the gas-liquid interface<sup>1-3</sup>. More recently, we have shown that the production of liquid marbles is achievable using an alternate, non-contact method, involving electrostatics<sup>4</sup>. This method has also allowed the study of a range of particles, including polymer latexes which have lower contact angles than required for the direct contact formation method.

A 5 µL earthed water droplet was suspended above a bed of 85 µm diameter polystyrene particles, and a potential applied between 0.5 and 3.0 kV. Certain potentials allow for the electrostatic transfer of the particles to the droplet interface, which was monitored as a function of bed-droplet separation distances. Adding a shell of conductive polypyrrole to each particle alters the particles' charging and transport behaviour, resulting in differing bed-droplet separation transfer distances for a given applied potential. Further modification of the polypyrrole shell thickness and dopant properties allows for the particle hydrophobicity and conductivity to be tuned, thus changing conditions where liquid marbles can be produced using this novel method.



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# What Makes SLIPS Slippery? Measuring effective slip on lubricated surfaces with colloidal probe AFM

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Slippery Liquid Infused Porous Surfaces (SLIPS), inspired by the pitcher plant, possess self-cleaning and anti-fouling properties, with the potential for drag reduction.<sup>1</sup> They accomplish this via utilising micro- and nano-scale topography to trap a chemically compatible lubricant via capillary wicking, creating a customizable liquid interface. Though it is now a simple task to produce these materials,<sup>2</sup> understanding how liquid interacts with their interfaces on the nano-scale is yet to be fully understood, which is crucial for their practical use under shear.<sup>3</sup> In addition, the mechanism behind their lubricant retention is unclear, with the emergence of smooth surfaces capable of retaining a lubricant layer being reported in recent years.<sup>4</sup> Using colloid probe atomic force microscopy, we are measuring the flow of simple viscous liquids over smooth lubricated surfaces, with the aim to quantify effective interfacial slip.<sup>5</sup> This work will provide insight regarding liquid flow in confined geometries and on effective ways to minimise hydrodynamic drag.

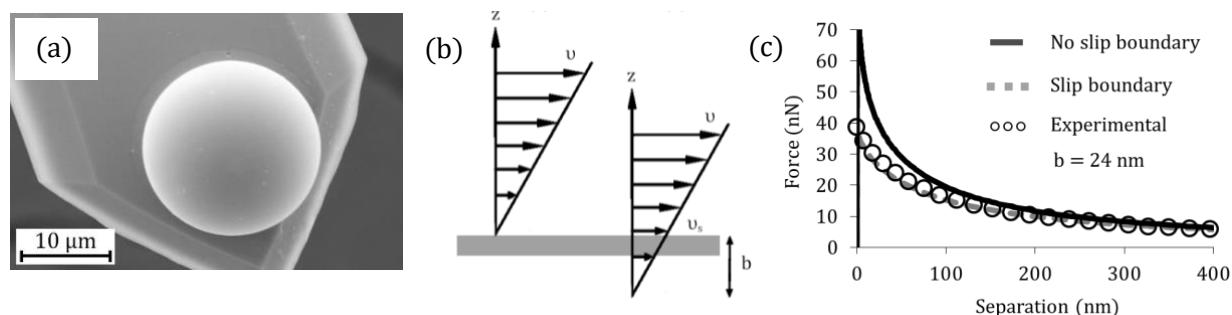


Figure 1 (a) Scanning electron micrograph of a colloidal probe used in hydrodynamic force measurements with diameter of approx. 17  $\mu\text{m}$ . (b) Schematic illustration of (LHS) the no-slip boundary condition, and (RHS) the partial slip boundary condition with corresponding slip length  $b$ . (c) Hydrodynamic Force vs Separation colloidal probe AFM measurement, experimental force approach curve fitted with theoretical slip length  $b$ .

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# **This Effect of Bacterial species and Calcium ions on Biofilm Adhesion forces on Silica based Construction materials at Atomic scale**

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Civil engineering constantly deals with the interface of construction materials and wide range of soil-water environments in which microorganisms especially bacterial biofilms are ubiquitous. Bacterial colonization at the liquid-mineral interfaces plays a crucial role in formation as well as stabilisation of soil aggregates. This property of bacterial biofilm adhesion to primary minerals has been extensively studied recently and has emerged as a potential technology with applications in various fields of civil engineering including Microbially Induced Calcite Precipitation (MICP) etc.<sup>1</sup>. The technology of MICP has fetched the greatest interest of Geotechnical engineers due to its wide applications as prevention of soil liquefaction, dust suppression, soil stabilisation, slope and embankments stabilization etc. The potential of this technology is dependent upon several physical and environmental factors including adhesion and fixation of bacterial cells to the granular substrate material. This adhesion is the first step in biofilm formation and the subsequent steps of mineral precipitation and biocement formation are dependent upon the efficacy of biofilm attachment.

A large number of pure bacterial cultures have been isolated from soils with potential ability of calcification in laboratory conditions but very limited information is available on variations in adhesion mechanisms by different soil bacteria. Surface charge and zeta potential of the bacteria as well as the substrate has also been found to play an important role in bacterial adhesion<sup>2</sup>. With respect to that, modification of the chemical heterogeneity in the substrate material can lead to change in the overall zeta potential, thereby altering the degree of adhesion<sup>3</sup>. Further information about selection of a potential bacterial culture and improving its adhesion via alteration of surface charge of the substrate can significantly improve the potential of MICP as well as other technologies exploiting the aggregation abilities by biofilms.

Keeping this in view, the aim of this study is to investigate the effect of a) variations in surface charges in different bacterial species and b) varying concentrations of CaCl<sub>2</sub> (which has been commonly used as a fixative of bacterial cells onto soil grains) on adhesion properties of bacteria with silica-based substrate. Biofilms of commonly used calcite producing bacterial cultures *Sporosarcina pasteurii* and *Bacillus megaterium* were grown and coated on the tips of Atomic force microscope (AFM) which provided the insights into electrostatic forces between the biofilms and the granular material at different time intervals.

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# High-refractive index silk-titanates nanocomposites for biophotonic and optical devices

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In the last decades a great interest is emerged in the development of a new class of nanocomposite materials obtained by the combination of natural polymers and inorganic nanoparticles, that integrate the advantages of the biopolymeric matrix, such as biocompatibility and biodegradability, with the functional properties provided by the inorganic fillers.<sup>1</sup>

Here we report the synthesis of easy-tailored high refractive index nanocomposite made of silk, a natural biopolymer, and titanate nanosheets (TNSs), a 2D precursor of TiO<sub>2</sub>.

Silk fibroin, derived from *Bombyx mori* cocoons, is a widely used and studied protein polymer for biomaterial applications, and, recently, it has been proposed as a platform for biocompatible optics, resorbable electronics, and implantable and biofunctional optical devices.<sup>2</sup>

To enhance the performances of silk as a material for optics while preserving all its specific properties, we combined silk fibroin with titanate nanosheets (TNSs). TNSs are 2D crystals of sub-stoichiometric TiO<sub>2</sub>, synthetized by sol-gel chemistry, that were chosen because of their small size, high refractive index and water dispersability, which makes the integration with silk processing very easy to implement.

The structural and functional characterizations of the new composite were performed to correlate functionality and structure. The different fabrication techniques already developed for pure silk were applied on the high refractive index nanocomposite demonstrating the possibility to fabricate optical devices with increased performances due to the increased refractive index.<sup>3</sup> In particular, a bioinspired multilayer optical structured was fabricated with the new material, showing a stimuli-responsive behaviour, which results in a reversible change of structural coloration in response to the surrounding environment. As the beetle *Hoplia coerulea* is able to modify its colour in the presence of moisture, thanks to the variation of the thin films stack responsible for the interference colour, in the same way our structure can sense the environmental humidity with a reversible mechanism and transduces the change in a colorimetric scale.<sup>4</sup>

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# Colloidal capsules from an emulsion route stabilized by graphene oxide composite materials and their delivery application

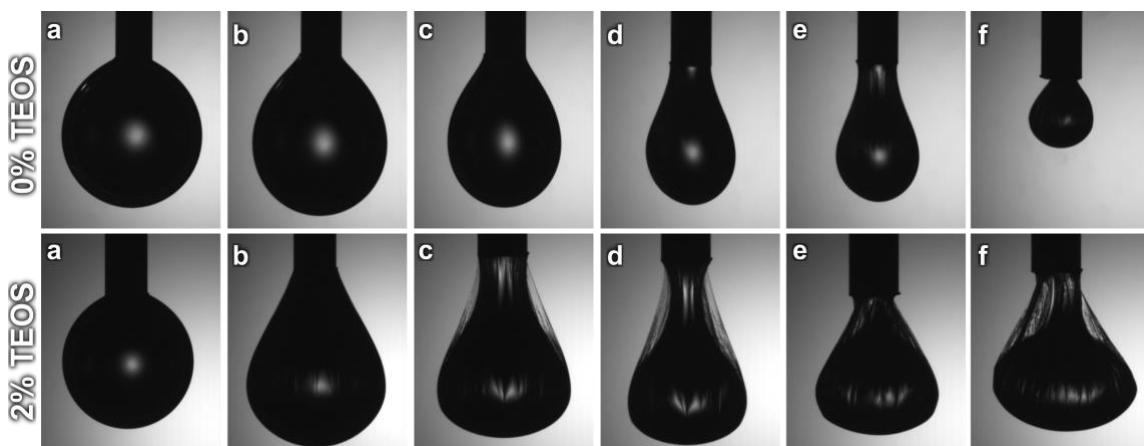
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Hybrid colloidal capsules are obtained using a Pickering emulsification route in which the interface is stabilised by graphene oxide (GO) and silica particle composite materials that form the droplet shell. Two integrated steps are proposed to fabricate these capsules using both pre-grown silica particles and solution phase growth of silica. The GO/Si/oil hollow capsules system is characterized using atomic force microscopy (AFM) and scanning electron microscopy (SEM).

Exploration of the mechanical properties using AFM, rheology measurements and compression-expansion techniques support the existence of hard shells surrounding the capsule, with the mechanical strength depending greatly on the concentration of the materials and the density of the shells. This capsule system has potential applications for the delivery of fragrances, pesticides and fertilizer materials. The capsule efficiency as a carrier has been determined by monitoring the release of loaded materials as a function of time using headspace gas chromatography and UV-visible spectrophotometry.



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# The study of foam film characteristics to improve the efficiency of foam dust suppression technology

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In many workplaces, air pollution caused by inhalable particles is not only a serious threat to the health of the staff, but also leads to the risk of catastrophic explosions.<sup>1, 2</sup> The spraying of foams is a potential technical approach to solve the dust problem in many industrial workplaces.<sup>3, 5</sup> The wetting ability of foam is much better than pure water, especially for fine particles as the gas-liquid interface of the bubbles has a molecular layer of surfactant.<sup>5, 6</sup> Further, since the volume of foam is dominated by gas, a small quantity of solution is needed to generate a large volume of foam. In order to improve the dust suppression efficiency of foam, knowledge of the effect of concentration and type of the foaming agent on the stability and elasticity of liquid films as well as the affinity between dust and the liquid film is required. Therefore, we are developing an experimental system to analyze the relationship between disjoining pressure and film thickness for different foaming agents.<sup>7</sup> Disjoining pressure isotherms will be measured with a thin-film balance, TFB<sup>8</sup> where the film thickness is measured by an interferometric technique.<sup>7,8</sup> The interaction of the liquid films with a particles in therespirable particle size range ( $<7 \mu\text{m}$ ) that mimic the surface properties of rock and coal will be studied to understand the stability of liquid films in the presence of particles and the interaction of particles with liquid films.

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# Development of novel, recombinant lubricin based coatings for implants.

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The field of bionics continues to advance and accomplish extraordinary feats by integrating electronic devices into the body to solve biological problems by mimicking a function. Despite the health benefits that implants offer, they initiate foreign body reactions that includes inflammation, infection, fibrosis, coagulation, and fibrous encapsulation<sup>1</sup>. Lubricin (LUB) is a natural, biological lubricant that works in synergy with hyaluronic acid (HA) within joints; LUB is a large, mucinous glycoprotein with a highly hydrated central domain that constitutes its natural, anti-adhesive properties, while its globular end domains are in contrast very adhesive and can self-assemble on almost any kind of surface in a carpet of molecular loops<sup>2</sup>.

Using recombinant LUB (R-LUB) and HA to coat gold mylar discs either by themselves or in a combined ‘synergy’ group, cellular attachment (fibroblast and chondrocyte cells) was investigated. R-LUB coatings show exceptional reduction in cell attachment on gold mylar discs compared to the control and in some cases, the HA group. HA alone reduced cell attachment, however it’s not observed as affecting vinculin as the R-LUB does; vinculin is a membrane scaffold protein with an actin binding site that helps attach cells to surfaces. The anti-adhesion by LUB affects vinculin by impeding its ability to form footholds on a surface. Experiments conducted under electrical stimulation (e-stim) mimicked typical electric signals sent from bionic implants to test LUB’s performance under an electric field. LUB significantly reduced cell attachment under both passive and e-stim conditions. LUB has demonstrated promising results in reducing cell attachment by impacting the vinculin proteins. It is a suitable candidate to develop coatings for any kind of medical implant to help reduce the attachment of proteins and cells during the first 6 weeks following implantation, when the immune system is at it’s most active, to enhance their longevity whilst also maintaining signal fidelity.

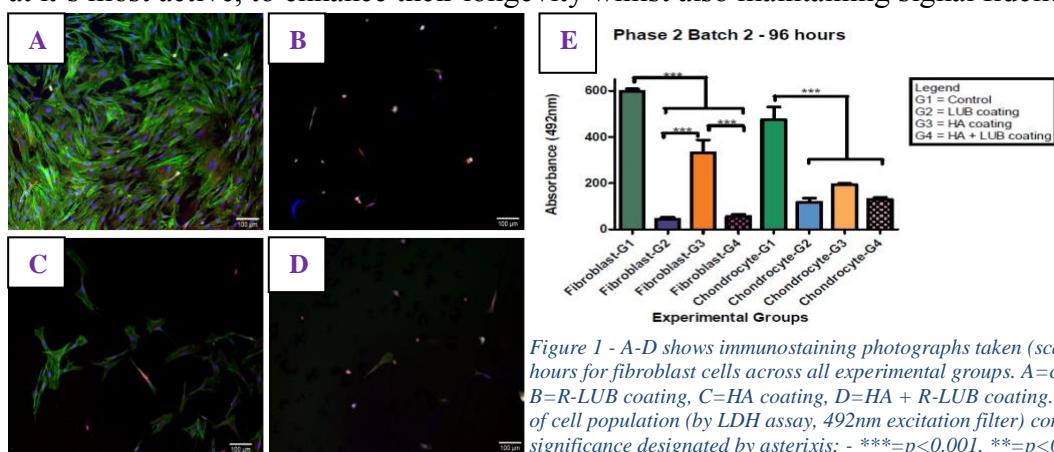


Figure 1 - A-D shows immunostaining photographs taken (scale=100μm) at 96 hours for fibroblast cells across all experimental groups. A=control (no coating), B=R-LUB coating, C=HA coating, D=HA + R-LUB coating. E=statistical analysis of cell population (by LDH assay, 492nm excitation filter) conducted at 96 hours, significance designated by asterisks: - \*\*\*=p<0.001, \*\*=p<0.01, and \*=p<0.05.

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# Characterization of Amphiphile Self-Assembly in Neat and Non-Stoichiometric Protic Ionic Liquid Solutions

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Protic ionic liquids (PILs) are increasingly being investigated as solvents for a wide range of applications due to their desirable properties, such as their ability to promote amphiphile self-assembly and form H-bonds, being highly tailorabile. However, their use in many applications requires a greater understanding of how additional chemical species affects their properties. To address this, we are investigating the effect of modifying the pH of PIL solutions through the addition of precursor acid or base, and the ionicity through addition of water. The eight PILs were used in this study containing alkyl ammonium cations combined with 3 different anions. A Latin Hypercube Sampling (LHS) approach was used to select 25 different non-stoichiometric PIL-water combinations for each PIL, as shown in Figure 1, in order to evaluate the effect of non-stoichiometry and the presence of water. Key physicochemical, thermal and solvent properties of the resulting PIL solutions have been obtained, including density, surface tension, thermal properties and Kamlet Taft parameters. We have previously reported that some of the neat PILs used in this study are able to promote amphiphile self-assembly [1]. Cross Polarized Optical Microscopy has been used to determine the liquid crystal phases of a cationic, non-ionic and anionic amphiphile supported in each of these PIL-solvent combinations, as shown by the representative image in Figure 2 for CTAB-Ethylammonium nitrate (EAN) system. For both exploring the liquid nanostructure of PIL-solvent combinations and determination of liquid crystalline mesophases of amphiphiles, Small and Wide Angle X-Ray Scattering (SAXS/WAXS) measurements have been carried out. These enable us to determine the mediating role of PIL-solvents in solvent-hydrocarbon interactions, and how the pH and ionicity of the solutions affects them. Consequently, this work will lead to the development of design rules for promoting amphiphile self-assembly and with implications for stabilising functional biomolecules in PIL based solvents.

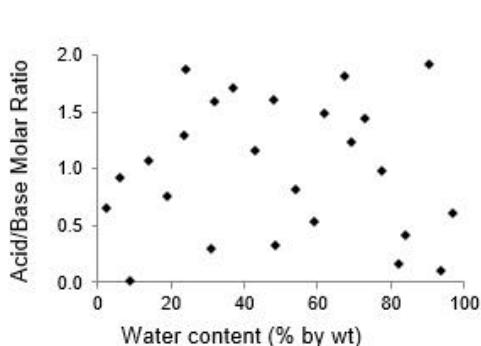


Figure 1. PIL-Solvent combinations according to LHS design methodology



Figure 2. CTAB-neat EAN penetration scan @ 85 °C (lamellar, cubic and hexagonal phases from left to right)

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# Bulk and interfacial properties of mixed systems comprising surfactants and carbon nanomaterials

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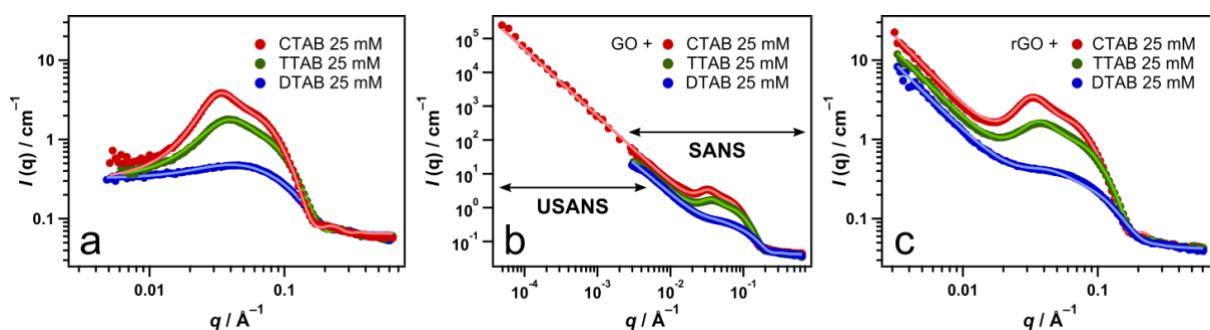
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Despite the enormous amount of research interest surrounding graphene oxide, a fundamental understanding of its thermodynamics, molecular interactions and stability as a colloidal material are lacking. By incorporating specific surfactant molecules into aqueous dispersions of GO and assessing the interactions between the two, it is possible to explore dispersion and adsorption characteristics to inform more effective utilisation of these materials.

A systematic analysis of the bulk properties of these mixtures has been conducted using combined SANS and USANS, to arrive at a more complete understanding of their aggregation and self-assembly. Crucially, the combination of SANS and USANS is required in order to explore scattering at both high  $q$  (surfactant aggregate region) and low  $q$  (fractal region). Results show that cationic surfactants have the strongest affinity for graphene oxide, compared to only minor interactions for nonionics and little to no interactions for anionics. Surfactant adsorption induces fractal-type morphology of the sheets as evidenced by USANS.

Lastly, neutron and x-ray reflectometry are used to explicitly examine the synergistic enrichment of GO/surfactant complexes at the air-water interface, providing valuable information on interfacial properties that complements the bulk scattering data obtained from SANS and USANS.



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# **Preparation of Novel Film Forming Polymer Latexes using Colloidal Nanoparticles as Emulsion Stabilisers**

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Polymer latex particles of diameter <300 nm have been prepared in the absence of surfactants through a Pickering-type emulsion polymerisation process stabilised in part by silica nanoparticles. Control of the silica wettability through modulation of reaction pH or by reaction of the nanoparticles with a hydrophobic silane resulted in silica-covered latex particles. Conditions were investigated to maximize adsorption of nanoparticles onto the surface of the latex such that the resultant latex was completely armoured in nano-silica. The oil-in-water emulsion polymerisation process used methyl methacrylate (MMA) and n-butyl acrylate (BA) as co-monomers, along with a water-soluble initiator, in the presence of a commercially available colloidal nano-silica (Ludox®- TM40). It was found that precise pH control before polymerisation using methacrylic acid (MAA) facilitated the formation of armoured latexes, and mechanistic features of this process are discussed. An alternative and more robust protocol was developed whereby addition of vinyltriethoxysilane (VTES) to control wettability readily resulted in latexes completely armoured in colloidal nano-silica. The latexes were characterised using SEM, cryo-TEM and AFM imaging techniques and the mechanism behind the adsorption was investigated through surface pressure and contact angle measurements in order to understand the factors that influence this irreversible adsorption. Results indicate that nanoparticle attachment (but intriguingly not latex size) is dependent on particle wettability, providing new insight into the formation of nanoparticle-armoured latexes, along with opportunities for further development of diversely functionalized inorganic/organic polymer composite particles

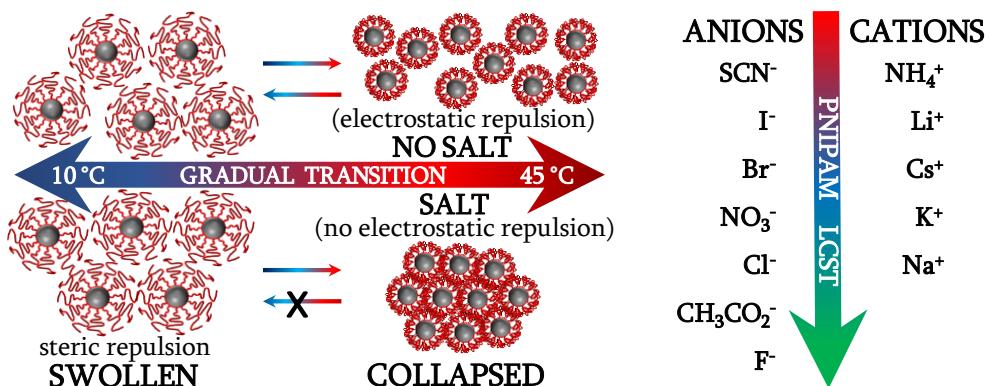
# Effect of Ionic Strength and Salt Identity on PNIPAM Brush Modified Colloidal Silica Particles

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The thermoresponse of core/shell poly(*N*-isopropylacrylamide) (PNIPAM) brush modified colloidal silica particles was investigated via dynamic light scattering between 5 and 45 °C. The intensity average diameter of the modified particles exhibited a broad transition between swollen and collapsed conformation between 10 and 35 °C with minimal hysteresis observed between increasing and decreasing temperature. The electrophoretic mobility ( $\mu$ ) of the modified particles increased in magnitude as the brush collapsed from 15 to 45 °C. Specific ion effects were observed for the lower critical solution temperature (LCST) of the modified particles in the presence of 11 different salts solutions between 50 and 1000 mM. The magnitude of brush layer LCST reduction was found to follow the order  $F^- > CH_3CO_2^- > Cl^- > NO_3^- \sim Br^- > I^- > SCN^-$  for the potassium series and  $Na^+ > K^+ > Cs^+ > Li^+ \sim NH_4^+$  for the chloride salts. The electrophoretic mobility of the modified particles was examined in the presence of 100 mM potassium salts, and was found to increase in magnitude as the brush layer collapsed and also as a function of the chaotropic nature of the salt.<sup>1</sup> The thermoresponse of the core/shell particles was also probed using small angle neutron scattering (SANS).



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# High Pressure Dewatering Rolls - Applying Shear and Compression Simultaneously to Enhance Dewatering

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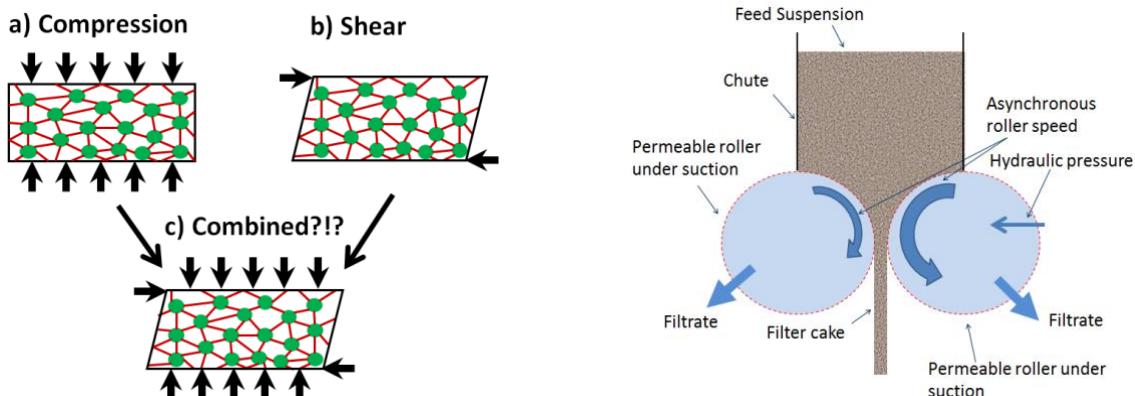
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The need for dewatering solid-liquid mixtures or suspensions spans across a variety of industries handling mineral suspensions, such as iron ores, tailings or coal and biological suspensions as paper pulp, algae or waste water sludges. Dewatering is often costly due to initial capital costs and ongoing process costs. For example, dewatering paper pulp suspensions is not only a crucial step for paper recycling, but also a major contribution to the recycling costs. In order to recycle paper more sustainably, new dewatering devices have to be considered. One pathway is to combine several forces synergistically to enhance dewatering, e.g. vacuum pressure, compressional and shear forces.

The dewatering of suspensions aims to remove water and increase the solids concentration. A particulate network forms at a material specific concentration, called the gel point. At concentrations greater than the gel point, suspensions exhibit a resistance against deformation. In compression, it is the compressive yield stress  $p_y$  (Figure 1a) and, in shear, this resistance is called the shear yield stress  $\tau_y$  (Figure 1b). For further dewatering of a suspension, an applied force has to be greater than the compressive yield stress  $p_y$ . However, dewatering devices in industry apply a combination of shear and compression (Figure 1c). Combining the two mechanisms seems to speed up the dewatering processes and is of great interest. However, the effects of combined shear and compression in dewatering are still unclear and need further investigation.

A novel dewatering device, called High Pressure Dewatering Rolls (HPDR), has been developed at the University of Melbourne that combines shear and compressional forces with an applied vacuum pressure. The operating principle of the HPDR is depicted in Figure 2. With this device, the extent of shear and compression can be adjusted and thus interrelation of the two can be investigated. This presentation will discuss the compressional rheology, followed by an overview of the HPDR and its scaling capabilities. Furthermore, experimental results of dewatering paper pulp with combined shear and compression and the HPDRs throughput will be presented.



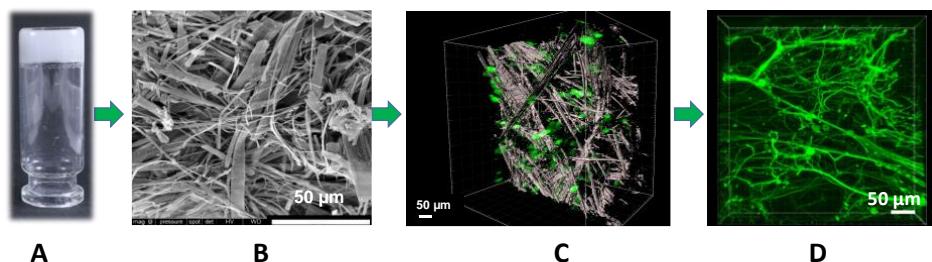
# Sugar-based supramolecular gelators as scaffolds for neuronal cell growth

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Currently, most of the scaffolds for 3D-cell growth are based on synthetic or natural polymers. Colloidal soft materials such as low molecular weight (LMW) supramolecular gelators, which are not polymers, are not widespread for these applications, but however are emerging in this field [1-3]. LMW gels can display a variety of supporting fibre morphologies, with various size, shapes, helicity, roughness, local stiffness and cross-link topology (Fig A-B). The aim of this work was to assess the biocompatibility of simple sugar-based supramolecular gelators for neuronal cell growth and to explore the effect of the gel structure and properties on this growth. For this purpose, three gelators differing only by a one-carbon increment on their alkyl chain length have been prepared. The gels were analysed by optical, electronic microscopy, rheology and uniaxial compression tests (Fig. A-B). The gels have remarkable different morphologies and mechanical properties, despite their tiny chemical structure difference.



After studying quantitatively cell viability and growth of a neuronal cell line on the different hydrogels, we demonstrated by confocal microscopy that the cells settled inside the gels, in 3D, until a depth of around 200  $\mu\text{m}$  (Fig. C). Adult human neuronal stem cells directly isolated from patient biopsies were also grown with success on the sugar based hydrogel. Immunostaining experiments specifically adapted to those extremely soft hydrogels highlighted the differentiation of the hNSCs into glial and neuronal cells and the formation of a really dense neurofilament network. It demonstrated that this hydrogel could support neural tissue regeneration in 3D. The original structure of the network - made of both very wide, straight and long micrometric fibres, linked together by a second network of narrower flexible nanometric fibres - makes it suitable for 3D neuronal cell culture [4]. This work also highlight how a tiny difference in chemical structure can have a strong impact on the scaffold morphology and the cell viability and growth.

The authors acknowledge the ANR ("Neuraxe" project) for financial support.

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# **Growth-related biophysical characteristics of microalgae cells and their impact on settling**

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Microalgae are photosynthetic organisms that are relevant to a variety of chemical industries including biofuel production, isolation of bioactive molecules, and water and wastewater treatment. The use of these organisms as industrial feedstocks encompasses a chain of unit operations that aims to generate the biomass (growth), to concentrate the suspension (harvesting/dewatering), to weaken the cells (incubation), to expose the intracellular products (rupturing), and to isolate the commercially-interesting compounds (extraction).

Industrially grown microalgae cultures are highly dilute ( $\sim 0.1 - 1.0 \text{ g L}^{-1}$ ) and very stable colloidal suspensions, which require extensive dewatering for economical downstream processing. Efficient dewatering is limited by the small size of the microalgae, the similar densities of the cultivation medium and the cells, the high ionic strength of seawater, and the release of metabolites which act as natural stabilizers. These facts pose technical challenges to concentration and dewatering, which represent up to 30% of the total production cost.

Size, density, ionic strength, and the presence of dissolved organics play an important role in the stability of suspensions. For microalgae, all these characteristics are dynamically regulated by elaborate physiological processes that change along the growth cycle, especially during lipid accumulation triggered by nitrogen deprivation. However, despite the relevance of these characteristics and their impact on dewatering via gravitational settling, a detailed quantitative characterisation and comparison is currently lacking.

In this project, the biophysical properties of microalgae suspensions were characterized across a diverse range of species and physiological states to evaluate how the growth stage influences subsequent dewatering that commences with gravitational settling. By understanding the influence of intrinsic cell properties on the settling behavior and subsequent dewaterability of suspensions, it will be possible to develop novel strategies including the use of natural low-cost coagulants and ultrasound harvesting to target reductions in harvesting costs and improved dewatering efficiency.

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# Laser incubation for blood typing

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For safe blood transfusion, blood group typing and antibody screening are mandatory to check for compatibility between patient and donor. Incompatibilities can lead to complications with varying degrees of severity including death. Red blood cell (RBC) express molecules called antigens on their membrane. The immune response occurs when antigens bind with their specific antibody. This results in the agglutination of the RBCs.

A certain kind of antibody (IgG) are checked for antibody screening. They required an incubation at 37°C [1, 2] for at least 5 minutes using gel technology [3]. This is usually done in an incubation oven.

Here, we use a laser to heat the blood, which has the potential to decrease incubation time and to easily control the blood's temperature. We find that laser incubation gives results as good as oven incubation. This work leads towards a more rapid blood typing test useful for urgent blood transfusion.

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# Decreasing the wettability of cellulose nanocrystal surfaces using wrinkle-based alignment

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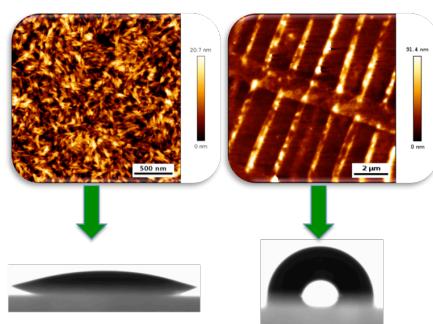
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Cellulose nanocrystals (CNCs) are a particularly appealing format of the natural biopolymer due to their exceptional strength, nanoscale dimensions and needle-like shape anisotropy [1]. However, CNCs are hydrophilic and hence their wettability makes them impractical for many coating applications [2], with various approaches using chemical functionalisation to overcome this. Here we show that CNC-coated surfaces can be rendered apparently hydrophobic by alignment of the native CNCs using a wrinkled template-mediated printing process [3]. We present a novel and simple method allowing full release of the CNCs from the template and their permanent adsorption into fine patterns onto the surface, thus preventing CNC repositioning during wetting. The aligned CNCs induce strong pinning effects that capture and retain water droplets with high contact angle and large roll-off angles, without becoming susceptible to oil contamination. The fabrication process for these coatings could be achieved by large-scale printing, making them a practical and cost-effective solution to hydrophobic coatings from raw cellulosic materials.



**Figure 1** The wettability of a water droplet changes from hydrophilic (bottom left) to hydrophobic (bottom right) when the cellulose nanocrystals are aligned (top right) compared to a random state (top left).

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# The effect of SDS on PNIPAM brushes examined with neutron reflectometry: results and methodology

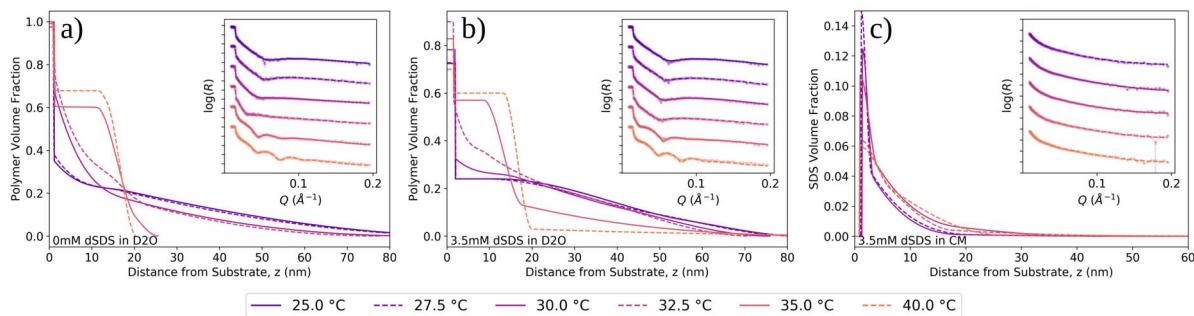
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Surfaces decorated with densely-tethered polymer chains – referred to as polymer brushes – have been shown to possess desirable anti-biofouling and lubricating properties that are directly dependent on their structure.<sup>1</sup> Additionally these surfaces exhibit a structural response to stimuli such as pH and temperature. Ionic surfactants are also known to elicit a response from polymer brushes, as they adsorb to the polymer, causing the brush to swell.<sup>2</sup> Surfactant and adsorbed polymer are often employed together in product formulation to stabilise active ingredients – where product efficacy depends on the interfacial structure of these components.

Here we present a study of the structure-stimulus relationship between a thermoresponsive grafted-from poly(*N*-isopropylacrylamide) (PNIPAM) brush and the common anionic surfactant sodium dodecyl sulfate (SDS). We use neutron reflectometry to determine the volume fraction profiles of the adsorbed polymer layer (a, b) and the molecular stimulus of interest (c) which elucidates the supramolecular structure of these interfaces. Our novel data fitting procedure utilises a Markov Chain Monte Carlo ensemble sampling method, allowing us to quantify the uncertainty and uniqueness of our final fit. Using selective deuteration we examine both the effect of SDS on brush structure and the effect of structure on the adsorbed amount of SDS, controlling structure via the system temperature. We find that SDS swells the polymer brush and that brush collapse has a negligible effect on surfactant adsorption.



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# Agglomeration of magnetite using a high internal phase water-in-oil emulsion

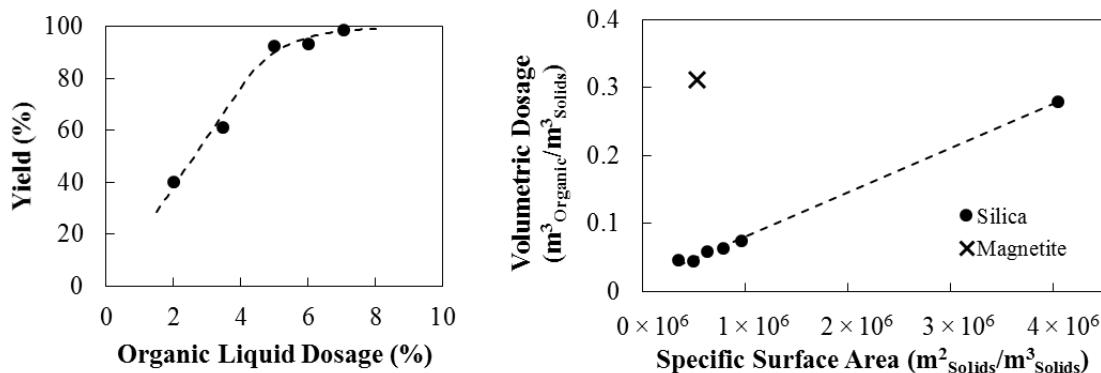
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A novel technique for recovering ultrafine hydrophobic particles from an aqueous suspension has been applied to magnetite particles with a Sauter mean diameter of 11  $\mu\text{m}$ . Previously, coal<sup>1</sup> and hydrophobised silica<sup>2</sup> particles have been recovered. The technique, originally inspired by oil agglomeration, utilises a binder consisting of a high internal phase water-in-oil emulsion to aggregate the hydrophobic particles. In a high shear field, the binder, 95% aqueous phase, and an organic phase of 2.5% kerosene and 2.5% emulsifier, is fragmented and thus presents an expanded hydrophobic interface of thin permeable oil films. The permeability of the films allows for particles as fine as 0.5 microns in size to be recovered in seconds due to the elimination of the viscous hydrodynamic resistance.

In this study, the agglomeration of magnetite was investigated to determine the relationship between the solid specific surface area and the organic liquid dosage required to achieve complete recovery for an alternative material. It should be noted that the emulsifier, sorbitan monooleate, acts as a collector for the magnetite, and no further surface modification was required. Firstly, the minimum organic liquid dosage required to completely recover the magnetite was determined and found to be ~ 5.5 wt% organic liquid. This result was then compared to the results for the silica system examined previously<sup>2</sup>. It was found that roughly 6 times more binder was required to agglomerate the magnetite than a similar sized silica powder. This significant difference between the binder requirements for the two materials was unexpected and current investigations are exploring the reasons for this variation



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# ***In-situ* small angle X-ray scattering of cellulose nanocrystal dispersions in a confined geometry**

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Aqueous dispersions of cellulose nanocrystals (CNC) produced by acid hydrolysis from plants and bacteria, could form a chiral nematic liquid crystalline phase. While the CNC rod packing in aqueous dispersions has been investigated at concentrations up to *ca.* 7.0 vol% by small angle X-ray scattering (SAXS) and laser diffraction,<sup>1</sup> the self-organization at high CNC concentrations (above 10 vol%) and effect of counterions on anionic CNC assembly has not been probed in nanoscale. Here, we will present our recent work where time-resolved SAXS was used to explore evaporation-driven assembly of CNC in a confined geometry. Time-resolved SAXS studies on shrinking levitating aqueous droplets show how the average spacing, *d*, between the CNC rods varies as the volume fraction increases from 1 to 25 vol%. The CNC dispersions in the shrinking droplets are able to attain near-equilibrium structures and phase at low and intermediate concentrations. At higher concentrations (above 10.0 vol%) *d* could be reduced to almost 1.4 nm before the structural features are lost. We have also used *in situ* SAXS to study the effect of different counterions (hydrogen, H<sup>+</sup> and lithium, Li<sup>+</sup>) on the structural behavior of aqueous CNC suspensions during drying and reswelling of films. It is found that CNC-Li films can readily swell and redisperse while CNC-H films swell much less and do not redisperse without intense agitation. The significant differences of the time-resolved structural evolution during drying suggests that the CNC-H particles readily forms a chiral nematic phase while the CNC-Li particles aggregate and form a poorly ordered gel.

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# The effect of asphaltene dispersion on characteristics of heavy crude oil in water (O/W) emulsions

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Emulsions or dispersions of heavy or extra-heavy crude oil in water (O/W) have been an alternative to exploitation of high-viscosity crudes because of viscosity reduction. However heavy or extra-heavy crude oil always have a characteristic of high concentration of asphaltene which is described as heavy, polar, and surface-active components in crude oil, they tend to gather at the oil/water interface and hinder the adsorption of surfactant molecules at the interface, which make it very difficult to form a stable O/W emulsion. Therefore, a further understanding of the effect of asphaltene dispersion on characteristics of crude oil in water emulsions could lead to improvements in heavy oil viscosity reduction.

In this work, we applied strong oleophilic polar group to disperse asphaltene aggregation in the oil phase first, then hydrophilic group with long main chain was applied to promote the formation of a stable O/W emulsion. An experimental test shows that, a low oil/water interfacial tension (IFT, 1.38 mN/m) can be achieved when 0.90 wt.% hydrophilic group and 0.30 wt.% oleophilic polar group were added into the heavy crude oil-water system. This IFT is significantly lower than the 18.42 mN/m IFT obtained by a 1.10 wt.% oleophilic polar group, as well as the 13.34 mN/m IFT achieved by a 0.70 wt.% hydrophilic group. The droplet size distributions and emulsion morphology at different mass ratios of hydrophilic and lipophilic group and aging time were measured to investigate the phase-transformation points from W/O to O/W. The emulsion rheological property with different mass ratios of hydrophilic and lipophilic group were measured to investigate emulsion stability. Finally, a mass ratio 3:1 of hydrophilic and lipophilic group was decided to be the optimal proportion for dispersing asphaltene and formation of stable heavy oil in water (O/W) emulsion.

# Examining the interaction of mesoporous silica particles with lipid digestion components

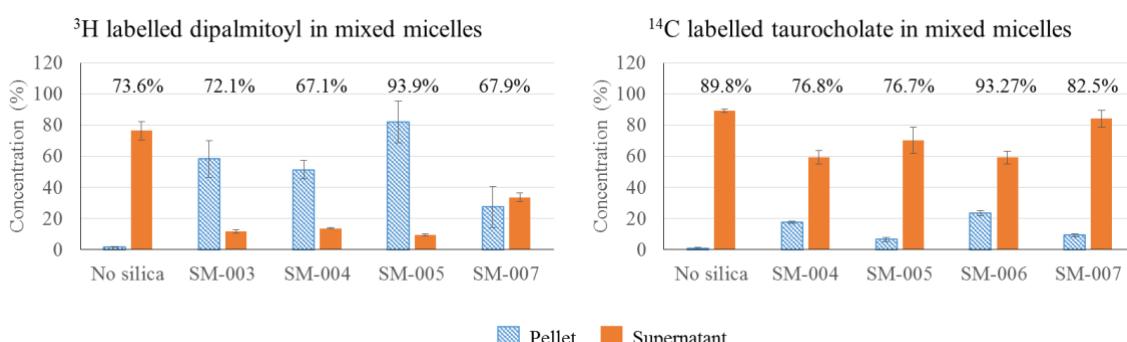
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Mesoporous silica particles (MSPs) have been proposed as a potential treatment for obesity. MSPs are composed of SiO<sub>2</sub> with highly ordered pores ranging from 2-50 nm in size. They have been reported to successfully reduce the weight of obese mice when incorporated into the diet<sup>1</sup>. Kupferschmidt et al. hypothesised that the weight loss was due to the MSPs disrupting the digestion of triglycerides, and hence hindering the absorption of calorific components. The properties of the MSPs, e.g. pore size and surface chemistry, have been shown to influence the adsorption of lipids into the pores of MSPs. Their properties also influenced the kinetics of the resulting *in vitro* digestion of the loaded lipid<sup>2</sup>. This project aims to explore why MSPs with specific properties alter the weight of mice, by investigating the interaction between the MSPs and various components of lipid digestion.

Mixed micelles containing taurocholic acid (TC) and dipalmitoyl phosphatidylcholine (DPPC) were investigated for their adsorption behaviour to the MSPs using radioisotopes as tracers. Labelled mixed micelles were incubated with MSPs of various pore sizes for 4 hours. The systems were centrifuged to separate the MSPs from the supernatant. Radioactivity of the pellet and the supernatant were measured to determine the bound and unbound components respectively. The results demonstrate that the DPPC adsorbed to the MSPs significantly while TC remained unbound in the supernatant. This indicates that there is an interaction between the components of lipid digestion and the MSPs.



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# Interfacial interaction of ruptured microalgae slurry and nonpolar solvent in a wet lipid extraction method

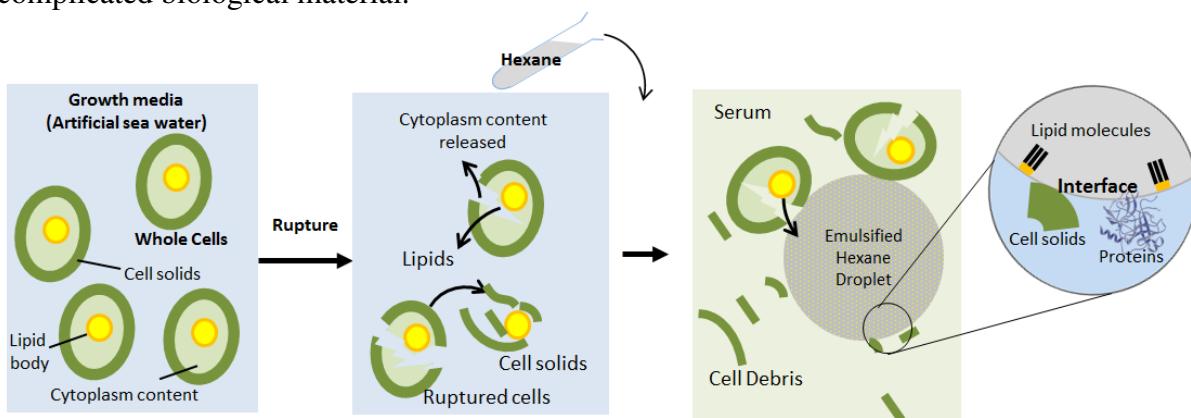
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Efficient lipid extraction from wet oleaginous microalgae biomass slurry (15-25% w/w solids concentration) is still a major technological challenge for biofuels application. A wet lipid extraction typically requires a cell rupture process to break the cell wall barrier to access the intracellular lipid bodies<sup>1</sup>. As the lipid bodies (3-8% w/w of the slurry) do not coalesce and cream even after the rupture process, the use of nonpolar solvent (e. g. hexane) is necessary to contact the ruptured biomass in order to dissolve and concentrate the lipids. Once saturated with lipid, the lipid-rich hexane can be separated via centrifugation. Mixing of the hexane and the biomass forms a tight emulsion that makes processing difficult<sup>2</sup>. As there are more than one type of potential emulsion stabilizer in the biomass, the identity and the role of these potential emulsion stabilizers are not known.

In this study, a marine microalgae species with commercial potential, *Nannochloropsis* sp., was used as a model of study. It was cultured and concentrated into a slurry form (10-20% w/w), before a cell rupture process using high pressure homogenization. The homogenized-ruptured biomass was then fractionated into cell debris, serum/supernatant, lipid, and delipidated biomass. The interaction of each fraction on the hexane interface was studied and characterized via various techniques such as interfacial rheology, pendant drop measurements, and emulsion stability (LUMifuge<sup>2</sup>). This fractionate-and-study approach allowed us to develop a better understanding of the potential role of each biomass fraction in stabilizing the emulsion and to elucidate the potential interaction between them in this seemingly complicated biological material.



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# Rheological effects on bacterial production of biofilm materials

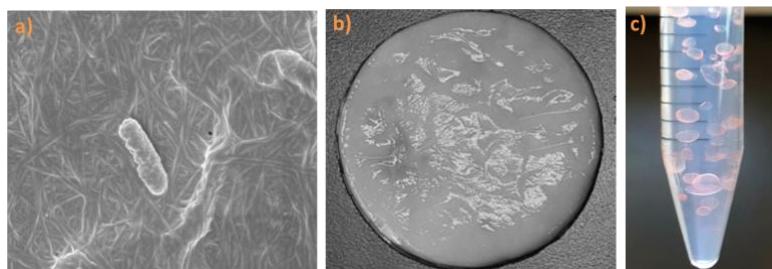
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Biofilms form when microorganisms grow on surfaces and produce different polymeric and biological compounds that make the immediate environment sticky. An example of a medically-relevant biofilm is bacterial growth in cystic fibrosis mucus, forming a complex gel. Although the biofilm rheology is complex and can limit transport of antibiotics used to treat the infection, it can also affect the growth of bacteria. Model systems used to study biofilms often modify the rheological environment so that common growth media like alginate can be used.<sup>1,2</sup> The effects of alginate and other polymer rheology on bacterial growth are not well-studied. A more systematic understanding of varying rheology effects on bacterial growth is relevant to experimental design as well as understanding of growth of biofilms in widely varying natural systems like mucus.

This work examines the formation and growth of biofilms in different rheological environments using microscopic observations and *in situ* study of rheological changes caused by bacterial metabolism. Water-soluble polysaccharide is used to modify the rheological properties of the growth environment for various encapsulated organisms.



a) Bacterium sitting on biofilm fibres, b) Bacterial cellulose pellicle, c) Dispersed bacterial capsule

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# Molecular insights into amyloid toxicity using model lipid membranes

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We will be focusing on how the mechanism of action of irreversible (toxic) fibrillogenesis e.g. in 'Alzheimer's Disease' differs from that of reversible (functional) fibrillogenesis which is observed in many hormones. Due to the delicate and heterogeneous nature of the cell membrane, we concentrated on lamellar phase as a model lipid bilayer system to simulate few of the properties of the biological systems (Conn & Drummond, 2013). Circular Dichroism (CD), a robust technique to study the conformational changes of these amyloid forming peptides in the liposomes and Synchrotron-CD (SR-CD) at ASTRID2 was used to obtain conformational integrity at lower wavelengths compared to conventional CD instruments. SR-CD is used to investigate the secondary structure content and also the conformational change that takes place when these amyloid peptides bind to the liposomes and cubosomes. According to the literature it is proven that amyloid fibrillation process is rapidly accelerated when it binds phospho choline (PC), amount of cholesterol in the membrane and anionic lipid phospho serine (PS). Liposome structural characterization information in the presence of amyloid peptide was obtained using High-throughput (HT) auto-loader Small-Angle X-ray scattering (SAXS) beamline at the Australian Synchrotron.

The aim is to investigate the mechanism of interactions between functional amyloid-forming peptides and cell membranes. Specifically, we are investigating how the lipid bilayer environment of the cell membrane affects fibrillisation, and conversely, how amyloid fibrils and intermediate oligomeric species affect the structure of the lipid bilayer mainly focusing on the lamellar phases to achieve insight into how these interactions affect fibril assembly kinetics and membrane phase behavior. In particular, we will focus on the effects of charge, lipid composition and curvature as these aspects can strongly disturb membrane interactions in a series of amyloid disease models (Reynolds, 2011). Synchrotron SAXS will be the main technique used to analyze the structure of the lipidic lamellar phase in the presence of oligomers and fibrils of model amyloid forming peptides.

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# Single-particle orbit tracking of nanoparticles within triblock terpolymer membranes

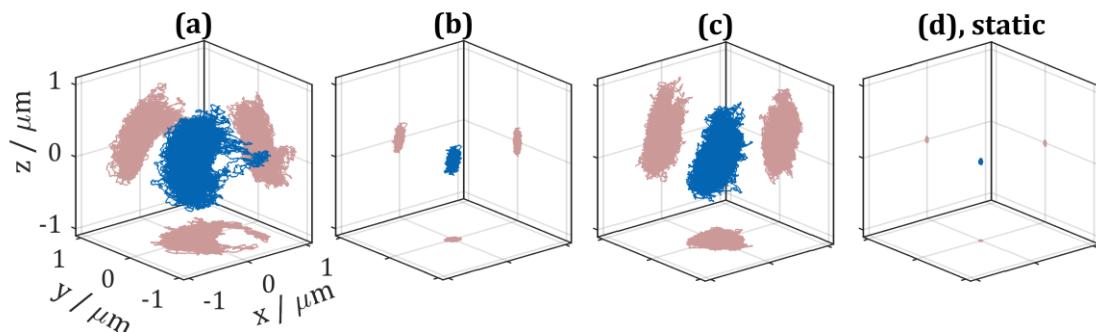
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We apply single-particle orbit tracking<sup>1</sup> (SPOT) for investigating the three-dimensional pore size distribution of nanoporous polystyrene-block-polyisoprene-block-poly(N-isopropylacrylamide) ( $\text{PS}_{43}\text{-b-PI}_{40}\text{-b-PNIPAAm}_{17}$ ) triblock terpolymer membranes under liquid filled conditions. In order to do so, we monitor the diffusion behaviour of single dye-labeled particles of different diameter with a spatial precision<sup>2</sup> of about 10 nm, thereby mapping out the cavity structure of the membrane. Our results are compared with environmental scanning electron microscopy (ESEM) data obtained from the same type of membrane.

We observe deviations between the morphological and the accessible volume, which is an important information for the application of such membrane materials. Consequently, we suggest our method as a high—resolution supplementary tool to standard techniques used in this field.



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# **Self-assembly of polydisperse sodium lauryl sulphates in solution**

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Surfactants are key components in many applications such as detergents, emulsions and surface modifiers, *etc.* Surfactants in solution will self-aggregate to form micelles provided their concentration is higher than their critical micelle concentration (cmc). The micellization of uniform sodium lauryl sulphates surfactants is well studied, however, the synergy effects when the head groups of sodium lauryl sulphate surfactants are polydisperse in size is less well known, but of great interest. In comparison to micelles formed by sodium lauryl sulphate surfactants of a uniform size, it is expected that the introduction of head group size variability will change the structure and properties of the micelle, where the mixed surfactant micelle may be more complex, but offer more capability or function for the use of surfactants in real world applications.

The aim of this work was to investigate how polydispersity of surfactant head group can affect micelle structure and its behaviour in solution, which in turn can be used to improve products formulation. To study this synergy effect, surface tension measurements were used to determine the cmc's of the mixtures of sodium lauryl sulphates surfactants with different head group lengths where the polydispersity of the surfactants was systematically varied. The effects of added salt were also evaluated. These measurements were done in parallel with small angle neutron scattering measurements to characterize the shape, aggregation number and dimensions of the polydisperse micelles.

# Particle-scale studies of ultrasound and temperature effects on cubosome and hexasome formation

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Soft particles of cubic and hexagonal liquid crystalline phase, cubosomes and hexosomes, form different shapes based on phase symmetry but are also affected by processes used to make them. Liquid crystalline particles are nanoscale, made by a top-down method like sonication.<sup>1</sup> Particles show a lot of variations in shape even though they are the same phase. For example, hexosomes can form as spheres, flat hexagonal prisms, and three-dimensional bicones for samples with different compositions made by different processes.<sup>2</sup> Systematic study of the shape variation is hindered by limited EM sample size and an inability to study the formation *in situ*.

In this work, an ultrasonic sample cell previously used to study particle adsorption<sup>3</sup> device was used with high-speed imaging to study formation of hexosomes and cubosomes (Figure 1a). Ultrasound-generated bubbles oscillate and create micro-streaming flows that break down the bulk phase and form nano and microparticles as shown in Figure 2b. This process can then be observed by microscopy, as well as subsequent changes to the particles. Ordered particles form immediately in some cases and particle shapes sometimes depend on rheological properties of the bulk phase. The phase with the lower elastic modulus can more easily form ordered shapes, allowing better particle design. Heat from the high-energy process causes phase transition from liquid crystal to L<sub>2</sub>, and later recrystallization can form more ordered shapes (Figure 1c). This study gives a new perspective on a much-used technique for creating liquid crystalline particles.

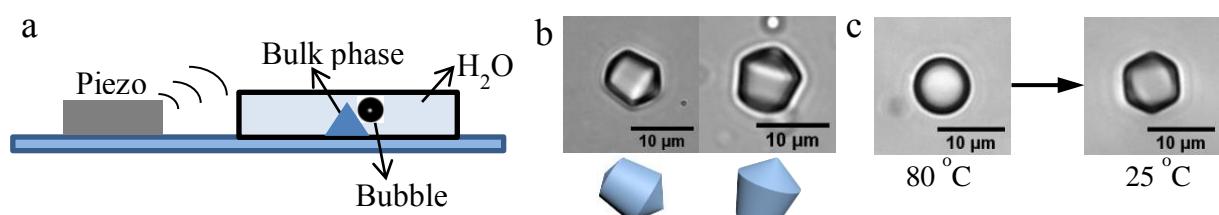


Figure 1. (a) Device schematic; (b) hexosomes via sonication; (c) cooling after dispersion.

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# Studies on adsorption kinetics of PiBSA-based emulsifiers at oil--water interface using dynamic interfacial tension measurements

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In this work, we explore adsorption kinetics of polyisobutylene succinic anhydride (PiBSA) based emulsifiers, at oil–water and toluene–water interfaces. The dynamic interfacial tension measurements were performed using pendant drop tensiometry<sup>1</sup>. Using the dynamic interfacial tension data, the dynamic adsorbed amounts of the emulsifier at solvent–water interfaces were estimated using the Gibbs adsorption isotherm, and Langmuir-Szyszkowski equation. It was seen that the adsorption kinetics of these emulsifiers follow a non-standard adsorption process, possibly because of competition between the molecules present in the emulsifier, as shown in the schematic (Figure 1). An attempt is therefore made to fit the experimental data using ‘two adsorbing species model’, discussed in our previous work<sup>2</sup>. We report that, close to the critical micelle concentration, the adsorption kinetics of such emulsifiers can be well predicted by this model.

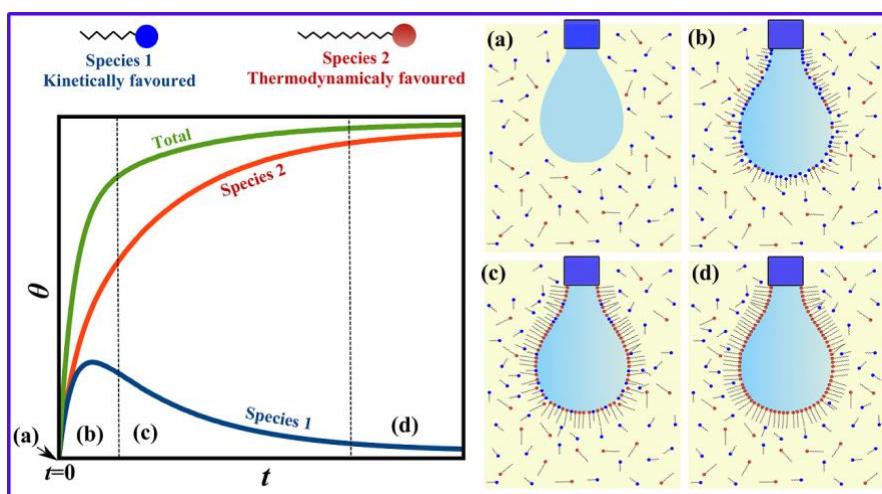


Figure 1: Schematic of competitive adsorption between two species

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# Biofabrication of Spider Silk Scaffolds for Biomedical Applications

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One great challenge in modern medicine is the regeneration of damaged tissue. A promising approach is the tailor-made design and manufacturing of scaffolds, which can be hierarchically structured and implemented with biological function. Spider silk is a suitable candidate for biomaterial applications, as it shows no immunogenicity, good biocompatibility and biodegradability. Recently, we developed engineered recombinant spider silk proteins, based on *A. diadematus* dragline silk, which can be processed into distinct morphologies. For instance, hydrogel preparation was examined in detail<sup>1,2</sup> as a basis for 3D bioprinting<sup>3</sup> to gain scaffolds for biomedical applications.<sup>4</sup> Further, negatively charged spider silk variants are capable of binding cations and can be biomimeticized in a two-step process.<sup>5</sup> As a result, specialized scaffolds for tissue regeneration applications of soft and hard tissue development are the aim of this study.

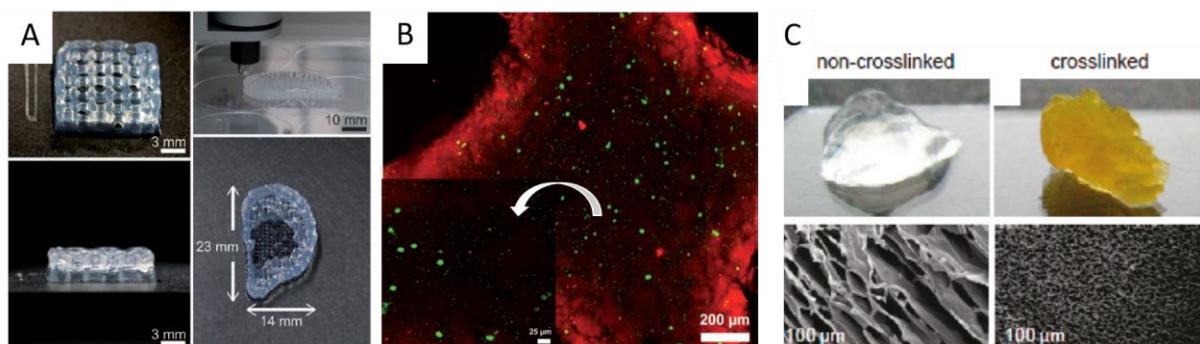


Fig. 1: A) 3D printed spider silk scaffolds; B) Confocal laser scanning microscopy images of human fibroblasts encapsulated in a printed 2-layer eADF4(C16) hydrogel after 48 h of incubation<sup>4</sup>; C) Macroscopic and SEM images of eADF4(C16) hydrogels<sup>1</sup>.

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# Novel Approaches for the Characterization of Bulk Nanobubbles: A Step towards Solving the Puzzling Existence of Nanobubbles in Aqueous Solution

Muidh Alheshibri, Vincent Craig

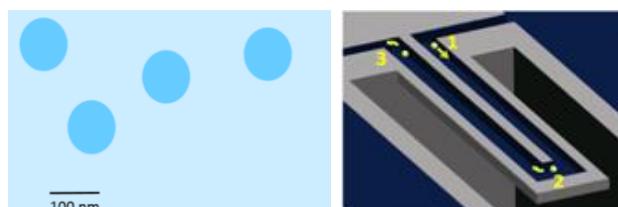
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Surface nanobubbles have been widely studied in the last 10 years. Their acceptance as nanoscopic gaseous bubbles is widespread, even though their stability is not fully understood. In contrast, reports of bulk nanobubbles<sup>1–3</sup> are controversial as they are expected to dissolve on a timescale of 1 – 100  $\mu$ s, as dictated by the widely accepted theory of bubble dissolution<sup>4,5</sup>. Despite this, nanobubbles reportedly have applications in water treatment<sup>6</sup>, froth flotation<sup>7</sup>, and ultrasound imaging<sup>8</sup>. It is therefore important to develop methods to test if nanoparticle dispersions contain nanobubbles.

The aim of this work is to investigate model systems that are reported to contain bulk nanobubbles in order to determine if the nanoparticles in these systems are indeed nanobubbles.

I will describe two novel approaches to investigate the nature of the nanoparticles. Firstly we develop a method to determine the density of nanoparticles using the resonant mass method. Secondly we develop the equipment to measure the influence of external pressure on the size of nanoparticle dispersions. As the density and compressibility of a gas is very different to the density and compressibility of liquids and solids these methods are able to determine whether nanoparticles are gaseous or otherwise.



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# **Engineering Cellulose Fiber Inorganic Composites for Depth Filtration Adsorption**

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Liquid filtration is widely used in food and beverage applications. Depth type composite filter mediums, which are porous materials heavily loaded with adsorbents, find a range of applications in liquid filtration. These adsorbent filled filter products remove contaminants from liquids by both mechanical entrapment and adsorption. Even though existing products are commercially available, there is still a need of development for high performance filter products with selective adsorption.

In this study, performance of products with nanocellulose as a potential partial substitute for the fibre matrix was investigated. Filters were fabricated by embedding perlite particles into cellulose fibre matrix and structured into flat sheet configuration by papermaking technique. Different composites were prepared by the same proportion of fibre and inorganic fillers, where nanocellulose fraction of the fibre component was varied. Mechanical properties, pore size distribution, surface morphology and 3D structure were characterised. Adsorption and filtration characteristics were quantified by using a laboratory benchmark system on removal of suspended particles and solute molecules from aqueous solution. Furthermore, relationship between permeation flux, porosity and adsorption/filtration characteristics was quantified. Results indicated that nanocellulose is an outstanding material with increased surface area to achieve high performance filter products.

## **Biography**

Aysu completed her Bachelor's degree in Chemical Engineering department at Istanbul Technical University in 2013. She received a Master's degree in Engineering Management from Deakin University in 2015. Aysu is currently working on a GRIP (Graduate Research Interdisciplinary Program) PhD project- Novel structured adsorbents for liquid filtration, under supervision of A/Prof Warren Batchelor and Prof Gil Garnier, with 3M industry partnership.

# **Protein sorption into polysaccharide polyelectrolyte multilayers studied by ATR FTIR**

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Wound dressing design is an active area of research where surface and materials chemistry has the potential to improve healing outcomes for patients, through understanding the interactions between biological molecules and surfaces and materials.

Polyelectrolyte multilayer (PEM) films have been shown to be an effective method of constructing coatings that can be applied to different surfaces to encourage tissue repair and growth. Naturally occurring biopolymers show great promise as biocompatible, bioactive components for PEM systems in wound care. Fucoidan, a sulfated polysaccharide mainly extracted from seaweeds, has the ability to bind and protect biomolecules in wound fluids to promote healing. This work investigates polyelectrolyte multilayers formed by layering fucoidan and chitosan chloride, (a well-studied biopolymer for biological applications) and the interactions of the film with lysozyme (LYZ) as a model protein.

Lysozyme is an anti-bacterial and degradative enzyme that is found in secretions such as tears, saliva and mucus, but not in a wound environment. However, from a physical chemistry perspective it is similar to fibroblast growth factor-2 (FGF-2) which acts to signal migration of cells to a wound bed and boost cell proliferation to promote healing. Both LYZ and FGF-2 have similar molecular weights (14.7 kDa and 17.2 kDa), similar hydrodynamic radius (19.5 Å, 28 Å) and are the same charge at physiological pH (pI 10.7 and pI 9.6) respectively.<sup>1, 2, 3</sup> LYZ is also much more cost effective for initial studies. Lysozyme is a glycoside hydrolase and by enzymatic hydrolysis has been shown to degrade chitosan-alginate-fucoidan scaffolds when incubated for 1 – 3 days.<sup>4</sup>

The layer-by-layer (LbL) build-up of the PEMs, and their interactions with LYZ, have been monitored using Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR). Build-up was shown to be reproducible and protein was shown to adsorb into the PEM as if it were a sponge. The PEM was then exposed to PBS for 8 hours to observe if lysozyme is released over time. The adsorption, interaction and entrapment of lysozyme will be discussed as well as how this relates to FGF-2 in a wound bed. In addition, the effect of lysozyme as a degradative enzyme will be shown.

# Radial Wicking in Paper: Effect of Biological Fluids

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The wicking of fluids is of fundamental importance in printing, textiles and more recently in the medical field because of its applications to paper based blood diagnostics.

The radial wicking of fluid through paper has been studied previously and some models have been proposed. These models involve several empirically derived constants and predict wicking to continue indefinitely until all fluid has evaporated. New results show that in the case of a sessile drop on paper, this is not the case, where instead an equilibrium final stain size is reached well before significant evaporation occurs. It has also been found that the size of the equilibrium stain is not significantly affected by surface tension or viscosity, both of which were found to be relevant to the previously studied non-equilibrium radial wicking situations.

The effect that drop volume, fluid properties and composition have on the equilibrium stain size and shape have been analysed, with particular attention given to the effect of biological fluids.

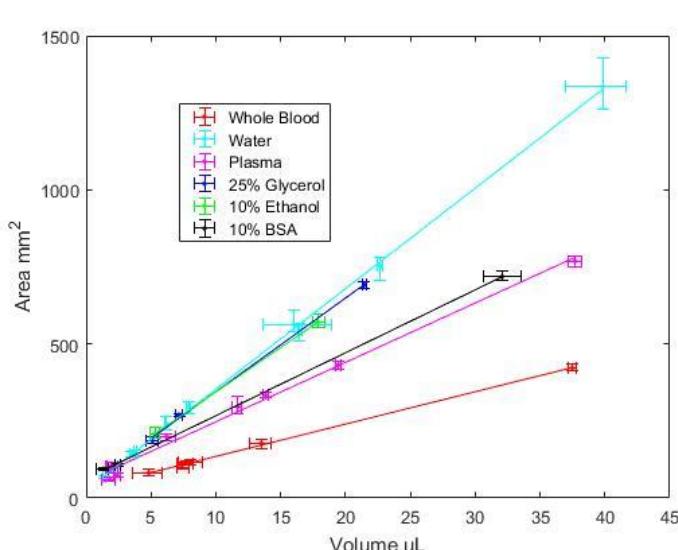


Figure 1: Variance of stain area with drop volume for a range of fluids

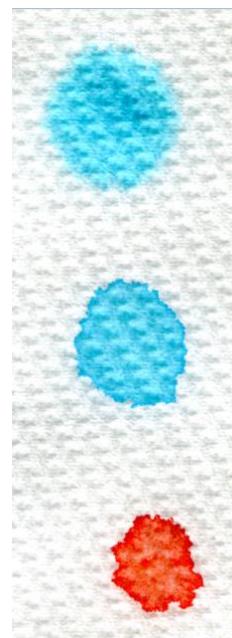


Figure 2: Drops of constant volume show high variability based on fluid properties.

# Degradation of Organosilica Nanosensors in Biological Media

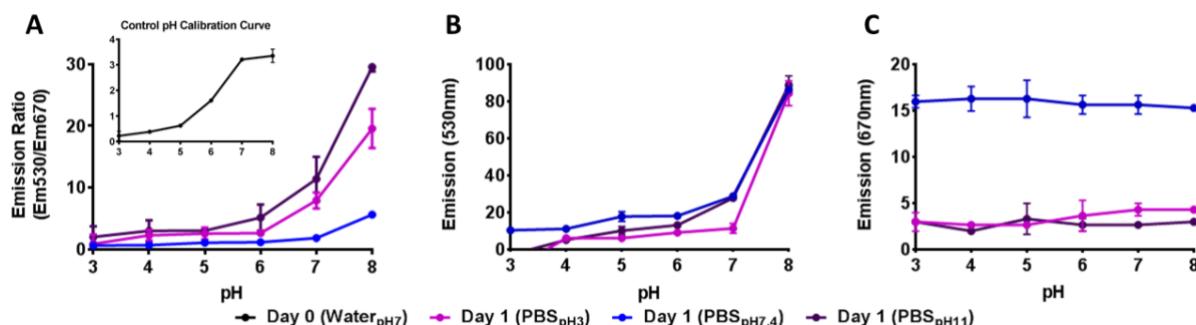
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In recent years, *in vivo* biosensing has become a key field in nanomedicine as a means of improving the quality of life for patients. Although there are commercially available biosensors, such as enzymatic blood glucose sensors, these *ex vivo* sensors lack the dynamic functionalities to accurately monitor key biological analytes continuously, often involve invasive sampling, and are unable to detect fluctuation between sampling points.<sup>1</sup>

Our group has recently developed a ratiometric organosilica nanosensor, displaying high sensitivity within a biologically relevant pH range. Although the dissolution of amorphous and mesoporous silica nanoparticles have been widely reported in literature, there has been little work surrounding organosilica nanoparticles.<sup>2</sup> Previous work has been limited to mesoporous organosilica particles,<sup>3</sup> but to our knowledge there has been no work conducted on amorphous organosilica nanoparticles. In this presentation, we will present our latest results on the particle and signal stability of our nanosensors in biologically relevant conditions (Figure 1).



**Figure 1:** A) pH nanosensor calibration curve, B) Fluorescein and C) Cy5 emissions after one day under accelerated conditions - incubated at 50°C. Insert: pH nanosensor calibration curve at Day 0.

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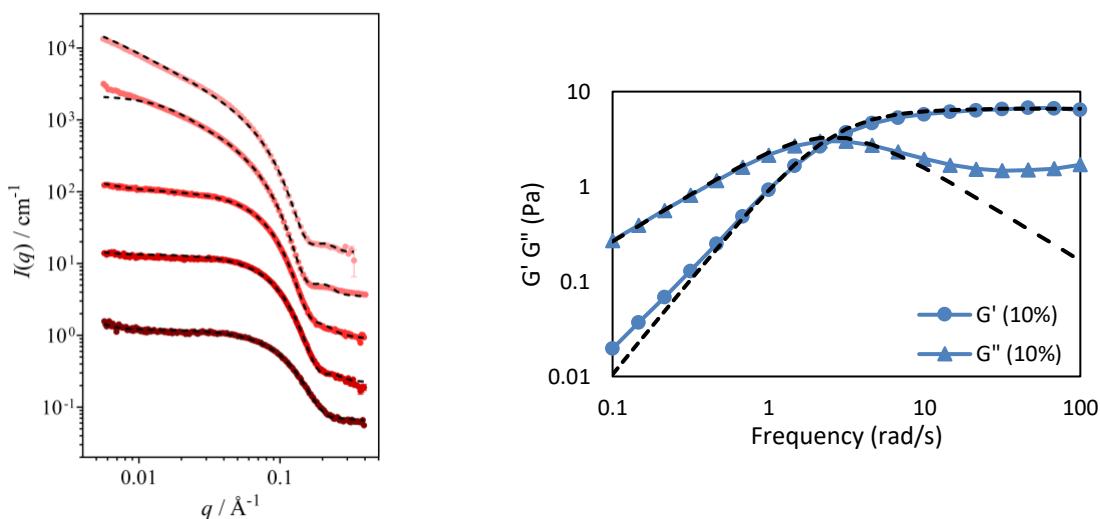
# Structure-function relationships and wormlike micelle formation of novel carbohydrate-based surfactants

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The use of carbohydrates as non-ionic surfactant head-groups is advantageous as they are naturally abundant, generally biocompatible and biodegradable, and readily functionalized. Here, seven novel carbohydrate based surfactants (CBS) have been synthesized with varying alkyl chain length. The aqueous self-assembly of these surfactants was explored using small- and ultra-small-angle neutron scattering (SANS and USANS). We observed elongation from spherical to cylindrical micelles with increasing alkyl chain length. Longer alkyl chains exhibited pronounced Krafft points, yet formed worm-like micelles as single components upon heating to 45–50°C. A slight structural modification facilitated production of similar worm-like micelles at room temperature. These worm-like micelles were further characterized using rheo-SANS and rheology, revealing that they exhibit near ideal Maxwell behaviour. These results demonstrate a promising molecular candidate for the formation of viscoelastic worm-like micellar solutions.



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