

PolyMAC
The 9th Annual
PolyMAC
Conference

Including the Research Groups of:

Alex Adronov
 Michael Brook
 Emily Cranston
 Kari Dalnoki-Veress
 Carlos Filipe
 Tohid F. Didar
 Charles de Lannoy
 Gillian Goward
 Kathryn Grandfield
 Todd Hoare

Jose Moran-Mirabal
 Robert Pelton
 Marko Saban
 Kalai Saravanamuttu
 Heather Sheardown
 An-Chang Shi
 Leyla Soleymani
 Harald Stöver
 Ryan Wylie
 Shiping Zhu

December 13, 2016
 McMaster University, CIBC Hall



Schedule

8:30 am	<i>Registration</i>
9:00 am	Keynote Speaker Dr. Charles de Lannoy
10:00 am	<i>Coffee Break</i>
10:20 am	Cheryl Bachan
10:35 am	Fariha Mahmood
10:50 am	Kevin de France
11:05 am	Vladimir Kardelis
11:20 am	<i>Poster Session</i>
12:00 pm	<i>Lunch</i>
1:00 pm	Derrick Hastings
1:15 pm	John Nivan
1:30 pm	Yuan Gao
1:45 pm	Vincent Huynh
2:00 pm	<i>Coffee Break</i>
2:20 pm	Benjamin Macphail
2:35 pm	Sarah Dawson
2:50 pm	Trevor Gilbert
3:05 pm	Kevin Saem
3:20 pm	<i>Door Prizes, Awards and Concluding Remarks</i>



Plenary Lecture

Dr. Charles de Lannoy (Chemical Engineering)



Impact of Polymer Nanocomposites on Global Environmental Issues

Water is a resource we cannot take for granted. Increasingly, safe, potable, available water sources are dwindling. "Emerging" contaminants produced from industrialized nations, naturally occurring environmental contaminants, bacterial, algal, and viral contamination, widespread droughts, and salt-water intrusion are a few of the major stressors affecting water supplies around the globe. Improved separations technologies are critical for dealing with these widespread challenges. Such separation technologies include membranes, sorbents, and catalysts. Polymer nanocomposite materials have demonstrated great potential in the field of separations both as composite membranes and as sorbents. As such, polymer nanocomposite materials are leading the way towards effective solutions to global environmental challenges. Sustainable, efficient, and effective polymer nanocomposite separations technologies will become increasingly vital to our society as they represent long-term solutions to our global water problems.

My research has developed a suite of polymer nanocomposite materials with direct environmental applications. These polymer nanocomposite materials take the form of 1) mechanically reinforced membranes, 2) electrically conductive polymer nanocomposite membrane coatings and active separation surfaces, and 3) catalytic nanomaterial composites.

Effectively reinforcing polymer membranes with nanomaterials requires understanding the compatibility of nanomaterials with polymer chains. Optimizing this interaction can lead to great improvements in stress-strain characteristics, as well as changes to surface energies. We showed that reinforced polymer nanocomposite membranes had greater resistance to pressures, while also demonstrating reduced surface hydrophobicity. These characteristics are important for enhancing the efficiency of aquatic separations, while reducing the concern for damage and breakage during operation.



Next we developed a suite of electrically conductive membrane surfaces formed from polymer nanocomposites. Simple cross-linking and interfacial polymerization reactions were developed to form these composites. These porous electrically conductive nanocomposites act like typical membranes for water filtration and desalination, while also demonstrating high electrical conductivity. Our active membrane surfaces demonstrate superior biofouling resistance, as well as the ability to prevent mineral scaling and organic fouling. These are the three largest challenges for effective membrane operation, and by combating surface fouling these polymer nanocomposite membranes demonstrate the ability to greatly increase membrane efficiency and effectiveness. Furthermore, the electrical conductivity of these polymer nanocomposites allows charge particle separation, in-situ degradations of contaminants, and catalytic decontamination of aqueous systems.

Finally, when dealing with nanomaterials, one must be cognizant of their potential environmental impact, especially as these polymer nanocomposite materials will be used for environmental applications. To achieve long-term solutions requires the development of safe, sustainable, and stable materials. It is critical, therefore, to test the stability of nanomaterials in their polymer matrices. Additionally, it is important to strive for safe and sustainable practices in the development of these polymer nanocomposite materials. We tested possibility of nanomaterials leaching from their polymer matrices in each polymer nanocomposite material developed. Leach-testing is done by challenging these materials under accelerated usage, weathering, and treatment procedures. Some early materials failed these tests, and more stable chemistries were developed to minimize the leaching of nanomaterials.

With these advances, the polymer nanocomposite materials that we have developed have the potential to pave the way for broad-based solutions to dynamically evolving global environmental problems.



Abstracts of Oral Presentations

Cationic Nanogels for Potential Ophthalmic Gene Therapy Vehicle

Cheryl Bachan, Todd Hoare and Heather Sheardown

Wet age-related macular degeneration is one of the most common causes of blindness due to deterioration of the macula caused by leaky and poorly formed neovascularization. Current treatment requires intravitreal injections administered every 4-6 weeks. Propositions for siRNA targeting vascular growth factors to inhibit vessel growth show promise for extended administration periods but due to physiological recognition undergo rapid degradation upon application. The development of cationic nanogels using polymeric "smart" biomaterials to transport siRNA is explained in this study. N, N-(dimethylamino)ethyl methacrylate (DMAEMA) was crosslinked with di(ethylene glycol) methacrylate (DEGMA) by free radical emulsion-precipitation polymerization. Size, charge and morphology were analyzed to assess potential as a nanovehicle. The lower critical solution temperature (LCST) was determined at 538 nm to monitor temperature sensitivity. Cationic particles were synthesized when composition was 55:45 (DEGMA:DMAEMA), sized at approximately 160 nm. They had minimal impact on cell proliferation and exhibited spherical morphology when imaged by TEM at physiological pH and maintained their structure between pH 3.5-9. Sensitivity to pH was shown by DLS through swelling at physiological pH, which can be taken advantage of in future studies for loading and release. Degradation was exhibited by GPC suggesting promise of bioelimination. Future tests using gene therapeutics will be attempted with transfection studies to verify delivery of therapeutics in target cells.

Photoreactions in an Optical Vortex

F. Mahmood, O. A. Herrera Cortés, A. V. Arzola, K. Volke-Sepúlveda, and K. Saravanamuttu

Optical vortices are beams of light that exert torque perpendicular to their direction of propagation. This angular momentum originates from the helix-like phase structure of these beams. The topological charge corresponds to the number of twists in the helix and determines the size and direction of angular momentum—i.e. the beam may exert a force clockwise or counter-clockwise with varying strength. We study the interactions between optical vortices and nonlinear media, which are materials that change refractive index in the presence of light. In our materials, this change occurs due to light-induced chemical reactions. Our first system uses the photopolymerisation of acrylate moieties on organosiloxane oligomers. The second involves a photoreaction between a Ru-based complex and sodium iodide. Our experiments reveal complex interactions between the materials and optical vortices. The most frequent response was breakup of the annular beam profile into two or more self-trapped filaments. Both systems also show rotational responses, where the sense of motion (clockwise or counter-clockwise) switched upon reversal of topological charge. These results allow us to understand the impact of angular momentum at both the molecular and macroscopic structural level.



Anisotropic Cellulose Nanocrystal Composite Hydrogels

Kevin De France, Emily Cranston and Todd Hoare

While injectable hydrogels have several advantages in the context of biomedical use, their generally weak mechanical properties often limit their applications. By physically incorporating cellulose nanocrystals (CNCs) into hydrazone cross-linked poly (oligoethylene glycol methacrylate) (POEGMA) hydrogels, macroscopic properties including gelation rate, swelling kinetics, mechanical properties, and hydrogel stability can be readily tailored. Furthermore, by utilizing different preparation techniques including freeze-casting and magnetic alignment, hydrogel network structure and anisotropy may be readily controlled. In the case of freeze-casting, macroscopic network pore morphology can be tailored via changing the composition of the CNC/POEGMA dispersion and the freeze casting temperature, resulting in hydrogels with fibrillar, columnar, or lamellar morphologies. In the case of magnetic alignment, CNC orientation can be controlled via application of an external magnetic field, whereby CNC rods align perpendicular to the field direction due to their diamagnetic susceptibility. Due to the structural hierarchy and directionality of many biological tissues, the fabrication of anisotropic hydrogels exhibiting direction-dependent structure and properties have attracted great interest in biomimicking, tissue engineering and bioseparations applications.

Post-Polymerization Cycloaddition of a Cyclooctyne-Containing Polyimine

Vladimir Kardelis, Ryan C. Chadwick, Alex Adronov

A conjugated polyphenyl-co-dibenzocyclooctyne Schiff-base polymer was synthesized via condensation of dibenzocyclooctyne bisamine (DIBO-(NH₂)₂) and bis(hexadecyloxy)dialdehyde. The resulting polymer exhibited high molecular weight ($M_n > 30$ kDa, $M_w > 60$ kDa), good solubility, and good stability. Strain-promoted alkyne-azide cycloaddition reactions with a series of aryl azides allowed efficient functionalization of triazole units at the polymer backbone and the rapid synthesis of a small polymer library with a consistent degree of polymerization (DP). GPC and density functional calculations suggest a substantial change in polymer conformation after cycloaddition. Kinetic studies show second order reaction rate constants consistent with rate constants for monomeric dibenzocyclooctynes. Grafting with polystyrene and polyethylene glycol monomethyl ether azide-terminated polymers allowed the efficient syntheses of a series of graft-co-polymers with M_n values up to 800 kDa.



Investigation of Cationic Charge-Shifting pBDMAPA in Cell Encapsulation Systems

Derrick Hastings and Harald Stöver

Polyelectrolytes are of great interest in the biomedical field because of their wide range of chemical properties and their ability to be engineered to meet specific criteria.¹ Polycations, in particular, are useful in alginate-polycation-alginate cell encapsulation systems, however standard encapsulating polycations, such as poly-L-lysine, evoke an immune response due to patches of positive charge on the surface of the capsule.² One possible solution to this problem is to use charge-shifting polycations to reduce the net positive charge of the polymer due to hydrolysis, such as poly-N,N-dimethylaminoethyl acrylate (PDMAEA) or poly-1,3-bis(dimethylamino)propyl acrylate (PBDMAPA).^{3,4} This presentation will discuss the (co)polymerization, hydrolysis, and other properties of pBDMAPA, as a charge-shifting polycations to potentially replace poly-L-lysine in cell encapsulation. Experimental results of monomer synthesis, homopolymerization, and copolymerization with 3-aminopropyl methacrylamide in various monomer feed ratios by conventional free radical polymerization will be presented. Reactivity ratios of BDMAPA and APM will be presented as well as hydrolysis studies of the charge-shifting polycations. Future considerations of the project such as coating experiments, crosslinking, and cell toxicity experiments will be discussed.

1. Nair, L.; Laurencin, C. *Progress in Polymer Science* 2007, 32, 762-798.

2. Orive, G.; Tam, S.; Pedraz, J.; Halle, J. *Biomaterials* 2006, 27, 3691-3700.

3. McCool, M.; Senogles, E. *Eur. Polym. J.* 1989, 25, 857-860.

4. Ros, S.; Burke, N.; Stöver H. *Macromolecules* 2015, 48, 8958-8970.

Capillary Levelling of Holes in Freestanding Polymer Films

John Niven and Kari Dalnoki-Veress

Studying nano-scale flow in thin viscous films is of both practical and theoretical interest. Here, bilayer polymer films with thicknesses thousands of times less than the diameter of a hair were prepared freely-suspended in air, with one of the two films having micrometer scale cylindrical holes. When heated, the films transition to the molten state, and minimize surface energy by flowing to fill in the holes. Because of the free interface, such films flow without interfacial friction at either surface. The temporal evolution of the holes can be studied using atomic force microscopy, and shows three distinct regimes: an early time regime where the films undergo an elastic response; an intermediate regime where the films symmetrize to equilibrate internal Laplace pressure; and a late time regime where the films flow to fill in the holes.



Development of Thermoplastic Reversible and Multiple Shape Memory Polymer Systems

Yuan Gao, Weifeng Liu and Shiping Zhu

Reversible shape memory polymers (RSMPs) have made tremendous progress in recent years, shifting between two temporary shapes with or without external force. We present a general strategy of fabricating polyolefin RSMPs via engineering methods. Here a diblock ethylene/1-octene copolymer was synthesized with designed chain structures and tunable distribution of hard segments and soft segments, resulting in a continuously wide range of crystal sizes. In the current thermoplastic semicrystalline polyolefin system, a reversible shape memory effect was observed upon heating and cooling cycles, both under stress and stress-free conditions by DMA measurements. A reversible tangle change and multiple shape changes were also observed in RSMP films. These behaviors were driven by a mechanism of oriented crystallization, and a further crystallization-induced elongation upon cooling of the semicrystalline shape memory polymer. The first temporary shape was fixed by large crystals, which formed the "skeleton", so that the newly formed small crystals could be oriented along the skeleton and induce an elongation on a macroscopic level. The second temporary shape is fixed with further cooling, and melts with heating to recover the first temporary shape. This platform for RSMP design and synthesis would lead to a series of new kinds of potential products.

An Injectable and Degradable Hydrogel for Long Term Drug Delivery to the Posterior Segment of the Eye

Vincent Huynh and Ryan Wylie

Current treatments for many retinal diseases such as macular degeneration, diabetic retinopathy, uveitis and retinitis pigmentosa, require frequent and invasive intravitreal injections of anti-VEGF inhibitors. To decrease injection frequency, increase patient compliance and reduce potential side effects from frequent invasive procedures, we propose the use of an injectable and degradable hydrogel for long term drug delivery. The proposed two-step drug delivery system (DDS) utilizes the reversible and irreversible complexation of streptavidin to desthiobiotin and biotin respectively. Using sparingly soluble biotin derivatives, a streptavidin conjugated drug would be released from the hydrogel matrix at a rate determined by the solubility of the biotin derivative, resulting in release >5 months. Incorporation of the DDS in PEOGMA hydrogels with in situ gelling capabilities and tunable degradation profiles, an elegant and simple method for the controlled release of a therapeutic anti-VEGF inhibitor will be developed.



Silicone Boronic Acid Elastomers: Interfacial Phenomena Inducing Elastomer Reinforcement via Saccharide Binding

Benjamin Macphail, Michael A. Brook

Silicone elastomers are normally crosslinked through covalent linkages. Boronic acids form complexes with a variety of ligands including other boronic acids and saccharides [1]. We were interested in examining the use of this complexation as a new way to make silicone elastomers. Both pendant-arranged and telechelic-arranged tartrate ester-protected silicone-boronic acid polymers with different molecular weights and boronic acid densities were synthesized via hydrosilylation[2]. These polymers were exposed to moisture, which led to hydrolysis of tartrate protecting groups and an immediate and dramatic change in physical properties [3]. Low viscosity oils were transformed into viscoelastic films even in the absence of coordinating Lewis bases; Lewis acid/Lewis base complexation provides a different route to crosslinking. Although the protected oil spreads readily, upon water exposure, very rapid boronic ester hydrolysis occurs at a water-silicone interface and the free boronic acids adhere both to water and other boronic acids forming 1:1 complexes which facilitate crosslinking. The stability of these films were tested to better understand the impact of silicone molecular weight, density of boronic acid appendages and boronic acid-saccharide complexation on mechanical properties. The surprisingly high level of control over mechanical properties that is possible simply by changing these parameters will be discussed.

1. Springsteen, G. and B. Wang, *Tetrahedron*, **2002**, 58(26): p. 5291-5300.
2. Dodge, L., Y. Chen, and M.A. Brook, *Chemistry--A European Journal*, **2014**, 20(30), 9349-9356.
3. Zepeda-Velazquez, L., B. Macphail, and M.A. Brook, *Polymer Chemistry* **2016**, 7, 4458-4466.

Heterogeneous Nucleation in Block Copolymers

Sarah Dawson and An-Chang Shi

The microphase separation of block copolymer mixtures offers an exciting opportunity to design materials with tunable properties. The self-assembly of the material into ordered microphases typically proceeds through nucleation. In particular, the rate of nucleation and subsequent growth of an ordered phase can be modified by the presence of an interface. It is important from a theoretical perspective to understand this process. The free-energy landscape describing the system is quite complicated, so we focus on the most probable transition pathway: the minimum free-energy path (MFEP). By studying this path we can visualize the free-energy landscape during the transition. Furthermore, by looking at how the MFEP changes in the presence of an interface we can design an optimized substrate to produce defect-free materials. We demonstrate how an interface alters the MFEP in a model system, and describe how this work can be extended to block copolymer systems.



Injectable Interpenetrating Network Hydrogels

Trevor Gilbert and Todd Hoare

Interpenetrating polymer networks (IPN's) are formed by interlocking two cross-linked networks without covalent bonding between them. Previously reported IPN biomaterials were either implanted (requiring traumatic surgical intervention) or polymerized *in situ* (only viable in the limited subset of biologically benign chemistries). Alternatively, we have developed a method to produce IPN hydrogels through reactive mixing of functionalized polymer precursors, injectable as solutions. This is accomplished using hydrazone bonding to gel a thermosensitive poly(N-isopropylacrylamide) (PNIPAM) network and orthogonal thiosuccinimide (fast) or disulfide (slow) chemistry to cross-link a highly hydrophilic poly(vinylpyrrolidone) (PVP) network. The resulting IPN's are mechanically reinforced (assessed by shear storage modulus G' , most extensively using thiosuccinimide cross-links in the PVP networks) and retain the LCST thermal response of PNIPAM (although LCST transitions are more reversible, slower and smaller; potentially useful for mitigating drug burst release) with the addition of adhesive behaviour from the PVP network. The nano-scale structure of the composite (assessed by contrast matched small angle neutron scattering and dSTORM single-molecule fluorescence labeling) combines features attributable to each constituent network type, with PNIPAM-enriched domains in a PVP-rich field of fractal polymer clusters, possible useful for differential partitioning of drug cargo.

Single-Walled Carbon Nanotube:PDMS Devices - A Promising Avenue in Flexible Electronics

Sokunthearath (Kevin) Saem, Daryl Fong, Alex Adronov, Jose Moran-Mirabal

The use of elastomeric substrates in the field of flexible electronics presents an opportunity to develop conductive materials that can be stretched and compressed without altering their electronic properties, requirements that traditional conductive materials cannot fulfill. Single-walled carbon nanotubes (SWNTs) are promising materials for flexible electronics as they offer high conductivity, optical transparency at low loadings, and mechanical flexibility. In this work, we have used supramolecular functionalization of SWNTs with poly(phenylene ethynylene)-alkene (PPE-A) to confer solubility in organic solvents and vinyl group reactivity. Stretchable electrodes were fabricated via hydrosilylation reactions between SWNT-PPE-A and PDMS precursors during the vulcanization step. The resulting devices exhibited good electrical conductivity (sheet resistances as low as 6.5 $M\Omega/sq$) and optical transparency, and remained conductive when subjected to strains of up to 170%. The durability of the electrodes was tested through 100 stretch-and-release cycles (0-100% strain) resulting in no significant resistance change for the devices at 0% strain. Standard peel tests (10 cycles) showed excellent electrode-PDMS adhesion due to the covalent linkage of PPE-A-SWNTs to the PDMS surface. Through this study, we demonstrate the potential of SWNTs covalently grafted onto PDMS as electronically active and flexible substrates that can be useful in applications, such as flexible displays, pressure and biochemical sensors, and implantable medical devices.



Abstracts of Poster Presentations

(by first author first name)

Pancreatic Beta Cell Encapsulation in Alginate-based Hydrogels

Ali Abu Helal, Nicholas A.D. Burke, Nicole Latchminarine, Alison C. Holloway, Harald D.H. Stöver

Type 1 diabetes mellitus (T1DM) is a metabolic disorder characterised by autoimmune-mediated destruction of pancreatic beta cells. A promising therapeutic strategy for this disease involves the transplantation of functional beta cells encapsulated in alginate-based hydrogels. These biocompatible hydrogels prevent immune rejection and facilitate glucose-triggered insulin release *in vivo*. Here, we demonstrate a simple air-jetting system for encapsulating INS1E cells (a rat pancreatic beta cell line) in calcium alginate beads, as well as calcium alginate beads coated with different types of semi-permeable shells. Specific examples include poly-L-lysine/alginate shells, as well as novel, cross-linked, poly-L-lysine/poly(methyl vinyl ether-*alt*-maleic anhydride). Beads were characterised using fluorescence microscopy, cell viability assays, and sodium citrate/sodium hydroxide challenge tests. Preliminary results suggest that cell viability immediately after encapsulation is consistent across all three types of beads, and can be as high as 95%.

Snakes & Ladders: Instability Driven Pattern Formation in Thin Elastic Films

Ben Davis-Purcell, Kari Dalnoki-Veress

Intricate patterns are abundant in nature, from the stripes of a zebra, to the formation of snowflakes, to the wavy peaks and valleys on a beach. Instabilities often drive this pattern formation where two competing interactions result in a frustration that is alleviated through the development of these beautiful patterns. One such instability occurs when a soft elastomeric film bonded to a rigid substrate deforms to adhere to an upper rigid surface brought into contact with the film. In this system, the balance of interfacial surface energy and elastic strain energy leads to distinct labyrinth patterns. We study the formation of this adhesion-induced instability by placing small glass indenters on thin elastic films, observing the labyrinth pattern that forms upon contact. We investigate the characteristic wavelength of this instability as a function of film thickness, and also observe how the pattern changes when there is an anisotropic tension in the film.



Stabilizing Various Bicontinuous Morphologies via Polydispersity of Diblock Copolymer

Chi To Lai, An-Chang Shi

Diblock copolymers are composed of two chemically distinct homopolymers, covalently bound end-to-end. One of the most notable features of block copolymers is their ability to self-assemble into a wide range of ordered periodic structures as a means of minimizing the free energy. There are many factors affecting the equilibrium morphology for diblock copolymers, one of which is polydispersity. The rich phase behaviour of polydisperse diblock copolymers offers an opportunity to realize exotic phases that are not found in monodisperse systems. In particular, we explore the possibility of using polydispersity, in the form of binary mixtures of diblock copolymers, to stabilize the various bicontinuous structures, such as the double-diamond morphology. The principal methodology of our study is self-consistent field theory (SCFT). Solutions of the SCFT equations corresponding to the bicontinuous phases are numerically computed. The relative stability of the different ordered phases is then determined through a comparison of their free energy. From the study, we obtain optimal sets of parameters that stabilize the double-diamond or other bicontinuous morphologies, as well as examine the mechanisms responsible for the formation of these novel phases.

Self-Crosslinking Structured p(APM-co-AA) Films and Nanofibers for Use as 3D Biomimetic Scaffolds

Christal Zhou, Harald D. H. Stöver, and Jose Moran-Mirabal

In tissue engineering, synthetic or biological materials are designed to present biochemical and/or physical cues to cells to proliferate and regenerate lost or damaged tissue. Nearly all cells in the body reside within an extracellular matrix (ECM), consisting of intricate 3D fibrous networks of collagen and elastin. A well-characterized polymer system, poly(N-(3-aminopropyl)methacrylamide-co-acrylic acid), or p(APM-co-AA), was used to construct two types of 3D scaffolds to mimic the natural ECM: 1) structured films and 2) nanofibrous scaffolds. Attenuated total reflectance infrared spectroscopy suggests that p(APM-co-AA) scaffolds can self-crosslink under heat without the use of small molecule cross-linkers. The films and fibers were subjected to solubility tests under Dulbecco's Modified Eagle Medium and 1 mM NaOH to confirm their structural integrity in solution. The films and fibers were visualized using scanning electron and confocal microscopy. Surface wettabilities of the substrates were successfully tuned by functionalizing the films with decylamine or D-glucamine. NIH 3T3 fibroblasts were seeded onto films prepared with different weight percentages to introduce different structural environments. In this way, we aimed to manipulate and study cell behaviour by developing tunable environments that mimic the ECM.



Brush Polymer Analogues of PLA-PEG for Nanoparticle Drug Delivery

Daniel H. Luo, Lukas P. Sadowski, Meghan Rothenbrocker, and Todd Hoare

Poly(lactic acid)-block-poly(ethylene glycol) (PLA-*b*-PEG) polymers have found widespread use within the biomedical field owing to their demonstrated biocompatibility in a range of applications and favourable *in vivo* stability/degradation profiles. However, functionalization of such block copolymers is somewhat challenging, as only the polymer chain ends are available to add targeting moieties or other chemical groups to (for example) alter chain hydration, tune degradation, or optimize drug-polymer interactions. Instead of incorporating PLA and PEG in the polymer backbone, our approach was to design well-defined block copolymers with PLA and PEG on the side-chains of a methacrylate backbone polymerizable by controlled free radical polymerization, creating a “block-comb” polymer with significantly higher potential for tunability. A library of polymer analogues with various molecular weights were prepared and used for nanoparticle (NP) fabrication using a confined impinging jet device yielding NPs of 100-500nm in diameter. These NPs have prolonged stability, typically greater than 14 days at 37°C in PBS and negligible *in vitro* cell toxicity. Drug loadings of >96% and 41% was achieved with Taxol and the hydrochloride salt of doxorubicin, respectively. Given the clinical relevance of PLA-PEG polymers, the aforementioned brush polymers will be the subject of *in vivo* studies in the near future.

Cellulose Aerogels for Bone Scaffolding Applications

Daniel Osorio, Emily Cranston, and Kathryn Grandfield

Cellulose nanocrystals (CNCs) are versatile nanoparticles produced from renewable resources – they are strong, chemically inert and non-toxic. By grafting surface groups onto CNCs using water-based chemistry, CNCs gain the ability to form covalent bonds. Covalent bonds are formed when two CNCs are physically forced together through freezing or increased pressure. A CNC aerogel can then be formed through a drying technique. CNC aerogels have characteristics such as a bimodal pore morphology, high porosity, shape recovery, and stability in aqueous environments, which can be used for osteogenic scaffolds. The goal of this work is to understand the fundamentals of CNC aerogel formation in order to optimize them as a scaffold for new bone growth.

1. X. Yang, et al., *Chem. Mater.*, **2014**, 26, 6016 – 6025
2. Seifried, B. (2011). Supercritical Treatment of High Molecular Weight Biopolymers. WO 2011/120155 A1



Switchable Self-trapping of Laser Light in a Spiropyran Functionalized Hydrogel

Derek R. Morim, Ankita Shastri, Andy Tran, Fariha Mahmood, Joanna Aizenberg, and Kalaichelvi Saravanamuttu

Careful selection of a dynamic material can enable exquisite control of light for applications in responsive optics. We show unique, reversible optical self-trapping in a reconfigurable spiropyran-functionalized hydrogel and spatially contain the light along a cylindrical channel. We monitor self-trapping of a continuous wave, visible laser beam ($\lambda = 532$ nm) through changes in its spatial intensity profile that arise from dynamic refractive index increases in the material. This occurs from light-induced isomerization of open-chain merocyanine to closed-ring spiropyran, which locally contracts the polymer. Removal of the optical field allows spiropyran to revert back to the merocyanine form, and the system relaxes to its original state. Exploiting this reversibility, we can switch “on” and “off” the self-trapped beams in the medium over 45 trials with minimum relaxation times of 40 seconds.

Characterization of Boron Coordination Environment in Silicone Polymers via Solid State ^{11}B NMR

Gabrielle Foran, Ben MacPhail, Mike Brook, Gillian Goward

Silicone polymers are used in biological applications due to high biocompatibility and low toxicity.¹ Functionalizing silicone polymers with boronic acids results in materials whose properties can be tuned via boron loading and biologically-relevant temperature and pH changes.² This work investigates the effects of boronic acid loading and sample temperature on boron coordination environment in boronic acid-functionalized silicone polymers. ^{11}B is a spin 3/2 quadrupolar nucleus; the boron coordination environment can therefore be characterized by fitting the quadrupole lineshape.³ This method has been used to characterize amorphous glasses but has not been applied in the analysis of polymer systems.³ Preliminary experiments on simple boron-containing samples showed that these methods could be applied to more complex systems. Crosslinking between boron sites was found to occur regardless of boron loading. High boron loading resulted in the formation of structures with three- and four-coordinate boron centers that were not sensitive to temperature changes in the -12 to 28 °C temperature range. Polymers with lower boron loading were found to exhibit Gaussian lineshapes which were indicative of 4-coordinate environments. Boron-oxygen cross linking was found to be more prevalent at the boronic acid functionalized end than at the polymer backbone.

1. Brooks, W. And Sumerlin, B. *Chem. Rev.* **2016**, 116, 1375-1397
2. Dodge, L., Chen, Y. and Brook, M. *Chem. Eur. J.* **2014**, 20, 9349-9356
3. Kroeker, S. and Stebbins, J. *Inorg. Chem.* **2001**, 40, 6239-6246



Immobilized DNAzyme on Flexible Epoxy Surfaces as Real-Time Bacterial Indicators For Packaged Foods

Hanie Yousefi, M. Monsur Ali, Carlos D.M. Filipe, Tohid F. Didar

Infectious diseases caused by microorganisms through contaminated food can cause serious health care problems. Foodborne illness can be caused by microbial pathogens that enter the food supply before consumption. Therefore, real-time sensing mechanisms are much needed to examine food safety. We need simple biosensors that can be installed inside the food package and trace the presence of pathogenic bacteria inside the food. We are motivated to take advantage of deoxyribozymes (DNAzyme), which are artificial single-stranded DNA molecules, to generate a detectable fluorescent signal in the presence of a specific bacterial CIM (Crude Intracellular Mixture) in food or water. To generate a sensing layer located between the packaging and food material, we chose to covalently bind DNAzyme probes to epoxy-coated surfaces. Transparent, flexible and thin COP (cyclo olefin copolymer) slides, coated with epoxy were used for this purpose. Subsequently, amine terminated fluorogenic DNAzyme probes were covalently immobilized on transparent and flexible epoxy surfaces. This method can be applied to food wraps or water bottles. These DNAzymes cleave in the presence of pathogenic bacterial CIM and generate a fluorescent signal. The change in fluorescent signal can be reported as proof of the presence of bacteria.

Probing the Rearrangement of 2D Clusters of Soft Colloids Under Compression

I.-C. Ono-dit-Biot, S. Barkley, E. Weeks, T. Salez, E. Raphaël, K. Dalnoki-Veress

A crystal and a glass (an amorphous solid) are different at a molecular level which has strong consequences at the macroscopic scale. Observing these structures at the nano-scale directly remains challenging, thus we have developed an ideal system to experimentally model such structures on length-scales that are accessible with optical microscopy. Glasses and crystals are modeled using 2D clusters made of an emulsion - approximately 20 stabilized oil droplets ($R \approx 10 \mu\text{m}$) in water - assembled droplet by droplet. We study the response of the cluster when it is compressed between two thin glass rods ($R_c \approx 10 \mu\text{m}$). One glass rod is used as a force transducer in order to measure the forces within the cluster as the droplets spatially re-arrange under compression. Coupling the optical microscopy images of structural rearrangements within the 2D cluster with the direct force measurements provides insight into the failure mechanisms. Perfectly ordered crystals (highly monodisperse droplets) show well-defined transitions. As the number of defects (substitution of a droplet by a smaller one) is increased in the crystal, we can study the transition toward a glassy system (bidisperse cluster).



Injectable Nanocomposite Hydrogels with *in situ* Aligned Cellulose Nanocrystals

Kevin J. De France, Kevin G. Yager, Katelyn J. W. Chan, Emily D. Cranston, and Todd Hoare

Poly(oligoethylene glycol methacrylate) (POEGMA) hydrogels are thermoresponsive, non-cytotoxic, and biodegradable; all advantageous properties for various tissue engineering applications^[1]. Cellulose nanocrystals (CNCs) are colloiddally-stable rod-like nanoparticles that demonstrate a high Young's modulus, low cytotoxicity, and diamagnetic properties¹. Reactive coextrusion of aldehyde- and hydrazide-functionalized POEGMA, reinforced with CNCs, results in covalently cross-linked hydrogels with superior mechanical properties^[1]. However, these hydrogels are relatively isotropic and may be limited in biomedical applications since tissues such as muscle, cartilage, and nerves naturally require a high degree of alignment. This research aims to exploit the diamagnetic susceptibility of CNCs to enable facile *in situ* alignment within POEGMA hydrogels. Characterization with transmission electron microscopy (TEM) and small angle x-ray scattering (SAXS) gives evidence of CNC alignment within POEGMA networks, using a magnetic field strength of 0.56 T. The resulting hydrogels demonstrate decreased storage modulus and increased compressive modulus, attributed to planar alignment of CNCs within the hydrogel nanocomposites. Furthermore, preliminary mouse myoblast adhesion studies show an enhancement in cell elongation when cultured on aligned hydrogels. Therefore, anisotropy can be induced and tailored *in situ* in injectable CNC-POEGMA hydrogels to form aligned and mechanically-enhanced matrices for promoting directional cell growth and other tissue engineering applications.

1. De France *et al.*, *Biomacromolecules.*, **2016**, *17* (2), 649-660.

A Thermoresponsive Injectable Delivery System for Therapeutic Cells to the Retina

Megan Dodd, Graeme Prospero-Porta, Heather Sheardown

Degenerative conditions that affect the retina are the leading cause of blindness for adults in North America. The available treatments for these conditions are unreliable, expensive, invasive, and by no means curative. Previous approaches to deliver stem cells to the retina repeatedly demonstrate poor survival and integration of implanted cells, resulting in ineffective treatments. N-isopropylacrylamide (NIPAAm)-based copolymers have the unique property of being a liquid at room temperature and gelling above physiological temperatures, and have been optimized in our lab as a cell delivery vehicle. Modification of the thermogelling NIPAAm-based copolymer to incorporate bovine fibronectin protein did not inhibit previously assessed characteristics of transparency, LCST at physiological temperatures, or degradation. The proliferation and viability assays of ARPE-19 cultured in pNAND 11:10 prepared in 20% w/v PBS both 2D and 3D indicated that the copolymer presents a tolerable environment with some room for improvement. The pNAND copolymer with its attractive thermogelling characteristics presents a potential solution to low engraftment and survival of implanted cells in the eye. This material has the potential to be applied to a variety of cellular niches to repair and recover cellular damage.



Silicone Elastomers and Foams for the Synthetically Challenged

Mengchen Liao, Yang Chen, and Michael A. Brook

Silicone elastomers and foams are widely used in a large range of applications from automotive to cosmetic industries owing to their unique properties, such as electrical conductivity, hydrophobicity, skin feel, and bio-compatibility. Conventionally, hydrosilane functional groups are used in the synthesis of silicon-based polymer by condensation of hydrosilane with silanol groups, a process normally catalyzed by Pt, Sn or other metals or, more commonly, via hydrosilylation. We report that $B(C_6F_5)_3$ catalyzes both the hydrolysis and condensation processes to give silicone foams and elastomers whose properties can be tailored simply by judicious choice from readily available, commercial hydrosilane starting materials. A study of the reactivity of model compounds of pentamethyldisiloxane and bistrimethylsilyloxymethylsilane proved that the mechanism of the reaction involved participation of water in the reaction as a reagent rather than the alternative metathesis reaction. Polymer studies using tetramethylcyclotetrasiloxane and hydride-terminated polydimethyldisiloxane separately on or in combination showed that either elastomers or foams could be targeted. The mechanical properties of silicone elastomers and foams will be discussed.

Thin Film Swelling to Probe Cellulose Nanocrystal Particle-Particle Interactions in Solvents and Aqueous Media

Michael S. Reid, Stephanie A. Kedzior, Marco Villalobos, and Emily D. Cranston

Cellulose nanocrystals (CNCs) are sustainable and biocompatible nanoparticles, which have exciting mechanical, rheological and optical properties. Their interaction with water and solvents remains poorly understood but is of fundamental importance for the commercial development of CNCs and other cellulose-based materials. This work uses model films to investigate particle-particle interactions by thin film swelling in aqueous solutions (with sodium chloride) and organic solvents. Film thickness and solvent uptake were continuously monitored via surface plasmon resonance and the kinetics of liquid uptake were quantified. In aqueous media, CNC films with different surface chemistries and charge densities were evaluated to better understand the contribution of double layer forces to film swelling. Overall, swollen film thickness was observed to be independent of ionic strength, suggesting that for CNCs in the aggregated state (i.e., in a film) van der Waals forces dominate electrostatic repulsion, prohibiting CNC redispersion. Similarly, in organic solvents, attractive van der Waals forces maintained film stability however, solvents with hydrogen bonding and polar components more effectively interrupted particle-particle interactions allowing for more film swelling. This work highlights that CNC particle-particle interactions are dependent on the state of aggregation and these differences must be accounted for when developing cellulose-based materials.



Super-Resolution Imaging of Cellulose

Mouhanad Babi, Jose Moran-Mirabal

Cellulose – a major and critical component of plant cell walls – constitutes the largest component of Earth's biomass and is an attractive raw material to exploit in the production of biodegradable and renewable products, such as biocomposites and biofuels. Manufacturing these products often entails the depolymerization of cellulose, a process that is limited by its crystalline structure. To improve the efficiencies of these processes, we require insight into the nanoscale structure of cellulose and the mechanism of its depolymerization. In this work, direct stochastic optical reconstruction microscopy (dSTORM) was used to study the structure of fluorescently-labelled bacterial microcrystalline cellulose (BMCC) at the nanoscale. Super-resolution imaging unveiled regular repeating patterns of high and low fluorophore density regions on BMCC microfibrils, which were persistent despite labelling using different dyes or grafting reactions. The length of the dark regions was measured and their distribution was also found to correlate with the length of cellulose nanocrystals produced by a short hydrolysis treatments, as observed using transmitted electron microscopy. These results strongly suggest that the observed labelling patterns represent the differently-ordered regions of cellulose microfibrils, and for the first time are being observed and directly characterized in terms of their prevalence and length.

Thermal Stability of Cellulose Nanocrystals via Hydrolysis with Sulfuric and Phosphoric Acids

Oriana Vanderfleet, Emily Cranston

Cellulose nanocrystals (CNCs) are rod-shaped nanoparticles extracted from natural cellulose sources through a hydrolysis procedure which most commonly uses sulfuric acid. CNCs are charged with sulfate half-ester groups and demonstrate high colloidal stability; however, they are not stable at high temperatures. High thermal stability is a requirement for many applications, thus the focus of this research is the fabrication of thermally stable cellulose nanocrystals. CNCs were generated using a combination of sulfuric and phosphoric acids in the hydrolysis procedure. The resulting CNCs were characterized to determine colloidal and thermal stability. It was found that CNCs hydrolyzed with phosphoric acid showed increased thermal stability, yet lacked colloidal stability. Moreover, by introducing small amounts of sulfuric acid to this hydrolysis, CNCs with both high thermal and colloidal stability could be fabricated. These CNCs are an attractive option for many applications in oil and gas extraction fluids, where they could act as stabilizers and rheological modifiers at high temperatures.

Photochemistry in Optical Vortices

O. A. Herrera Cortés, F. Mahmood, D. R. Morim, A. V. Arzola, K. Volke-Sepúlveda, and K. Saravanamuttu

Optical vortices display an azimuthal singularity due to their helical phase front, which is described by $\exp(i\ell\phi)$ where ϕ describes the angular coordinates and the integer ℓ , the vortex topological charge. We experimentally study the dynamics of optical vortices launched into three different photochemical systems with the objective of understanding the impact of orbital angular momentum ($\hbar\ell$). Our preliminary findings show that the vortex spontaneously collapses



into filaments as it propagates through a nascent photochemical medium. The filaments then rotate about the propagation axis – clockwise and anticlockwise for a negative and positive topological charge, respectively. The speed of rotation and the number of filaments increases with the incident optical intensity.

Epoxy Functional Silicone Elastomers for 3-D Printing Applications

Robert Bui and Michael A. Brook

3-D printing is gaining interests for the application of additive manufacturing, particularly for developing prototypes or specialized products. Silicone polymers are currently employed in manufacturing for their heat resistance, moldability, and biocompatibility; which lends itself well to applications in implantable devices which can be fabricated by 3-D printing. Current 3-D printing technologies employ either photo-curable liquid resin polymers or deposition of thermoplastics which is restricted to uniform materials throughout the manufactured product. A new method of extrusion-based 3-D printing in which silicone polymers are synthesized through the mixing of multiple components (i.e. resin, crosslinker, catalyst) in tunable proportions. Polydimethylsiloxane functionalized with glycidyl- and cyclohexyl-based epoxides were synthesized and cured into elastomers with hydroxyl- and amino-based crosslinkers. The cure times and the skin-over times were fine-tuned to give partial cure upon deposition, and full cure after treatment with heat. It is shown that by varying the molecular weight of the components and using accelerators the cure time can be controlled to match the application of 3-D printing.

Degradable Polymer Microgels from Precipitation Polymerization

S. Sinjari, NAD Burke, HDH Stöver

The precipitation polymerization of 4-methylstyrene, maleic anhydride and divinylbenzene (DVB) in marginal solvents to form monodisperse polymer microspheres has been previously reported by our group^[1]. Hydrolysis of the anhydride groups can turn the particles into swellable hydrogels with anionic surface charges, while size and stiffness of the particles can be adjusted by changing crosslinker loading and solvent composition. We realized that these particles could be useful for tissue engineering applications because of the ability to tune particle size, surface chemistry and stiffness, but also that it would be advantageous to replace DVB with a more labile crosslinker. Here we report the synthesis of a series of microspheres prepared by precipitation polymerization of maleic anhydride, 4-methylstyrene and varying amounts of degradable crosslinkers such as 1,4-butanediol diacrylate (BDDA) and a redox-labile disulfide containing crosslinker. Physical characteristics of the particles such as swelling and size will be described. Hydrolytic cleavage of the BDDA particles will be discussed, and preliminary studies on the redox-degradation of the disulfide crosslinked particles will be presented.

1. Frank, R.S.; Downey, J.S.; Yu, K.; Stöver, H.D.H. *Macromolecules*. **2002**, *36*, 2728-2735.



3D Printable, Rapid Cure Silicone Elastomers Prepared Using Thiol-ene "Click" Chemistry

Sijia (Scarlett) Zheng and Michael A. Brook

Silicone elastomers are of commercial interest in a number of areas because of their distinctive properties. Although simple extrusion has been used for the preparation of silicone structures using 3D printing, methods to print objects with mixed moduli or other properties are not well developed. Although there are several methods for silicone-elastomer synthesis, the most common methods (high-temperature cure or platinum-catalyzed hydrosilylation) are not favorable to 3D printing application either because relatively harsh reaction conditions are required or the reactions are too slow. In our research, we are exploring the possibility of using photoinduced thiol-ene click chemistry as an efficient method to produce crosslinked networks, thus allowing for relatively short printing period and high fidelity in 3D printing manufacturing. Results show that this procedure allows for very fast cure times (<1 min) under UV radiation. Physical and mechanical properties of the elastomers can be modified by control of the chain length of the starting materials, the functionality and the concentration of chain extender. An initial examination of the ability to 3D print using this protocol, and of our ability to control the odor of our final product will be presented.

Beyond Buckling: Humidity-Independent Measurement of the Mechanical Properties of Green Nanocomposite Films

Urooj Gill, Travis Sutherland, Sebastian Himbert, Maikel C. Rheinstädter, Emily D. Cranston, Jose M. Moran-Mirabal

Cellulose nanocrystals (CNCs) have been targeted as environmentally friendly nanomaterials for thin films due to their biocompatibility, biodegradability, and high tensile strength. However, the humidity-independent characterization of the mechanical properties of green CNC-nanocomposites remains a challenge. We thus introduce a shrinking approach to irreversibly structure layer-by-layer (LbL) biocomposites, as a humidity-independent approach for quantifying the mechanical properties of these films. LbL films of CNCs and a complementary polymer were deposited onto pre-stressed polystyrene (PS) substrates, where the polymer type (xyloglucan, XG, or polyethyleneimine, PEI), and polymer concentration (0.1 wt% or 1 wt%) were modified. PS substrates were then shrunken by heat, which generated compressive stress that structured the films. Films had elastic moduli of 37 ± 2 GPa (CNC-XG_{0.1} films, [XG] = 0.1 wt%), 48 ± 2 GPa (CNC-PEI_{0.1} films, [PEI] = 0.1 wt%), and 25 ± 2 GPa (CNC-PEI_{1.0} films, [PEI] = 1 wt%). The elastic modulus, topography, and morphology of structured biocomposites were also tunable through simple changes in the LbL process. For proof-of-concept, RAW264.7 cell morphology was assessed on structured biocomposites, where cells extended less protrusions on films with larger topographies. These results open up potential applications of green nanocomposites in cell-based biosensors and extracellular matrix mimics.



Generation and Characterization of Microstructured Cell Scaffolds through Optical Waveguide Formation

Wan-Chi Chang, Alexander D. Hudson, and Kalaichelvi Saravanamuttu

Interest in the generation of microstructured cell scaffolds has grown in recent years due to their ability to facilitate controlled migration and alignment of particular cell cultures. These structures can be formed in a nonlinear material via modulation instability (MI), where intensity fluctuations in incoherent white light are amplified by refractive index changes in the material. Experiment studies of structured cell scaffolds based on photopolymerizable poly(ethylene glycol) diacrylate (PEGDA) hydrogel were conducted. The hydrogel is biologically friendly and can contain up to 90% of water; this makes it an ideal candidate for replicating the extra-cellular matrix. Waveguide formation via MI was optimized for the PEGDA hydrogel to increase transparency and minimize sample depth to 2 mm. Additionally, optical and physical properties of the structured hydrogel material were characterized to determine the viability of using it as a dynamic lens. Mouse fibroblast 3T3 cells will be introduced to the hydrogel and the migration of the cells will be analyzed.

Influence of Regiochemistry in the Selective Dispersion of Metallic Carbon Nanotubes Using Electron Poor Conjugated Polymers

William Bodnaryk, Alex Adronov

The incorporation of single walled carbon nanotubes (SWNTs) into electronic devices requires electronically pure samples of either semiconducting or metallic SWNTs. Selective extraction of SWNTs by wrapping with electron-rich conjugated polymers has proven an effective method for producing samples enriched in semiconducting SWNTs. However, large-scale purification of metallic SWNTs with conjugated polymers has proven elusive. In this poster, I will present SWNT dispersions prepared with three structurally similar poly(flourenco-phenylene) derivatives that possess varying degrees of nitration on the fluorene monomers. Differentiation of semiconducting and metallic SWNT populations was carried out by a combination of UV-Vis-NIR absorption, Raman, and fluorescence spectroscopy. We found that copolymers with *meta*-substituted nitro groups (with respect to the phenylene component) exhibit minimal inductive effects on the overall polymer backbone. When the nitro groups are *ortho*-substituted, a significant inductive effect occurs on the polymer backbone, resulting in a polymer that is more selective toward metallic SWNTs. The assessment of the inductive effects on the copolymer species was confirmed using Density Functional Theory (DFT) calculations. These results provide new insight into polymer design features, leading to the eventual goal of a conjugated polymer capable of selectively dispersing metallic SWNTs.



Nozzle Clogging Mechanisms in Inkjet Printing of Aqueous Silicates

Yuanhua (Zela) Li, Robert Pelton

Piezoelectric inkjet printing is an attractive technology for applying expensive reagents in roll-to-roll biosensor manufacture; however, printing silica sol-gel precursor solutions results in short duty cycles because of blocked inkjet print heads. Based on an extensive series of experiments, we have concluded that there are two major problems. First, and most important, thin layers of deposited silica near jet orifice induces the attachment of air bubbles generated by pulsing meniscus. Second, entrained air in the ink reservoir also contributes to attach air bubbles blocking flow. Freshly made sol-gel precursor solutions at low pH do not generate particles large enough to mechanically clog the jets. Printing performance can be improved by the inclusion of an inline filter to trap air bubbles. Surface treatment of the silica print head with a hydrophilic may also increase the printing efficiency. Ongoing experiments will determine if the ultrasonic field induces silica deposition or gelation.

Cellulose Nanocrystal Pickering Emulsions, Emulsion Gels, and Dried Emulsions

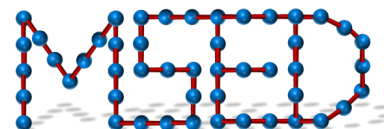
Zhen Hu, Robert Pelton, Emily D. Cranston

By tuning interactions between colloidal particles and surfactants/polymers, we can engineer sustainable high-performance materials with improved functionality. Cellulose nanocrystals (CNCs) are new commercially available nanoparticles which are derived from natural cellulose sources. Due to their "green" nature and non-toxicity, CNCs show great promise as emulsifiers, stabilizers and gelation agents in formulated chemical products such as foods, pharmaceuticals, and cosmetics. Herein we report emulsions stabilized by cellulose nanocrystals (CNCs) at low nanoparticle concentrations (0.25 wt. %) combined with either cationic surfactants or cellulose-adsorbing polymers. The resulting emulsions are extremely stable over long time periods (> 1 year) and can be tailored to be oil-in-water or water-in-oil. Surfactant and polymer adsorption to CNCs modifies their interfacial tension reducing abilities and wettability which is directly linked to emulsion droplet size and stability to coalescence. Polymer-enhanced CNC Pickering emulsions can be transitioned to emulsion gels at high temperatures (70 °C) by using thermally responsive polymers and can be dried to give CNC-stabilized oil solids with oil contents as high as 94 wt. % through freeze-drying. This work may extend the potential of CNCs to new liquid formulations as well as oil solid materials that are low-cost, green and potentially edible.



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