

# 2009 Annual Meeting Abstracts

## MONDAY

### MORNING

#### **FS&FF 1: Structuring at Nano Level for Food Applications**

Chair(s): D. Rousseau, Ryerson University, Canada; and J. Weiss, University of Hohenheim, Germany

**Control of Bioavailability of Lipophilic Components using Nanolaminated Coatings.** D. McClements, Department of Food Science, University of Massachusetts, Amherst, MA 01003, USA

There is a lack of effective delivery systems to encapsulate, protect and release bioactive lipid components (e.g.,  $\omega$ -3 fatty acids, carotenoids and phytosterols), which is holding back the development of functional foods that combat diseases such as coronary heart disease, diabetes and cancer. Delivery systems consisting of lipid droplets encapsulated by nano-laminated biopolymer coatings have great potential for use in the food industry, to improve the stability and performance of encapsulated lipids during storage, transport and utilization, but also to control their bioavailability after consumption. This presentation highlights how the physicochemical characteristics of nano-laminated biopolymer coatings can be rationally designed to control the bioavailability of encapsulated lipids. The use of *in vitro* and *in vivo* studies to provide fundamental mechanistic insights into the impact of specific coating properties on lipid digestibility are reviewed.

**Electrospun (Bio)polymer Nanofibers as Novel Functional Materials for the Food Industry.** J. Weiss, University of Hohenheim, Stuttgart, BW, Germany

Electrospinning is a technique that is used to fabricate ultrafine fibers with mean diameters ranging from several tens of nanometers to a few micrometers from solutions of synthetic polymers or naturally-occurring biopolymers. Ultrafine fibers or nanofibers have generated significant interest in a number of industries such as the food, pharmaceutical, personal care and chemical industries due to their unique mechanical, optical and thermal properties. In this presentation, we will provide an overview over recent results obtained in our laboratories that demonstrate the ability of electrospun nanofibers to act as novel controlled release vehicles. We will discuss typical fabrication set-ups, highlight the influence of process conditions on nanofiber properties, and emphasize emerging methods to further functionalize fibers for applications in the area of food science and technology. Specifically, we prepared synthetic and biopolymer nanofibers that contained both fine-disperse emulsion droplets and surfactant aggregates loaded with a lipophilic ingredient (eugenol; a potent food antimicrobial) to render the fibers antimicrobially active. We will present results of compositional, morphological and functional analysis of the generated fibers using FTIR, TEM and SEM, Release Studies and Antimicrobial Activity Assays.

**Forays into the Nanoscale of Fats.** N. Acevedo, F. Peyronel, A. Marangoni, Food Science Department. University of Guelph, Guelph, ON, Canada

In fat products, attributes such as spreadability, hardness and mouth feel are partly determined by the size and the shape of the individual fat crystals and the way in which they interact to form the network. Recent evidence suggests that the nanoscale plays a key role in determining the mechanical strength of fats and has the potential to affect many of these attributes. The objective of this work was to develop an adequate method for the determination of crystal size and morphology. The main goal was to elucidate the nano-structure of fat systems and discuss the arrangement and interaction of fat crystals and the consequences on higher levels. Blends of Fully hydrogenated canola oil and high oleic sunflower oil were prepared. Studies were conducted by microscopy, dynamic light scattering and X-ray diffraction and scattering. The results showed that X-Ray is a useful technique for ?see? individual domains of fat crystals. Using electron microscopy it was possible to observe the nanocrystal unit while polarized light microscopy allowed the observation of higher structural levels. The functional properties of fats are given by their molecular composition and nanostructure. The use of nanoscience to understand the structure in materials and the nanostructures induced upon processing will lead to rational selection of improved raw materials, enhanced processing and design of

novel foods.

**Sustained Release of Lysozyme from Corn Zein Micro- and Nanocapsules.** Qixin Zhong , Dept. of Food Science and Technology, University of Tennessee, Knoxville, TN, USA

Corn zein was researched as a carrier material for manufacturing particulate delivery systems with sustained release of antimicrobials to enhance microbial safety or reduce spoilage. Three techniques, i.e., liquid-liquid dispersion, supercritical anti-solvent, and spray drying, were applied to produce a range of lysozyme-loaded zein capsules with sizes at micro- and nanometer scales. Liquid-liquid dispersion was a simple and scalable process to produce zein nanoparticles with a diameter of 100-200 nm, and the encapsulated lysozyme showed sustained release in 2 days at neutral pH. Microcapsules were produced using supercritical anti-solvent and showed a continuous matrix with internal voids. Sustained release of lysozyme was observed over 36 days at room temperature, with slower release at higher pH between 2 and 8. At pH 4, release kinetics was further slowed by addition of sodium chloride. Spray drying was lastly studied as one commercially feasible process for various formulations. Although capsules were micrometer-sized, it was nanoscale structures of capsule matrix that were important to release profiles. Continuous capsule matrices enabled the sustained release of lysozyme over 49 days at neutral pH, while matrices composed of nanoparticles did not. For all capsules produced with the above three processes, more complete release of lysozyme was observed at lower pH because of stronger molecular repulsion between the carrier material and the encapsulated lysozyme. Findings from this work demonstrated the importance of molecular interactions and nanoscale structures for controlling the release rate of the encapsulated antimicrobials. Further research on this class of GRAS antimicrobial delivery systems may eventually reduce foodborne illness and food spoilage to enhance food safety and quality. This will be important to the wellbeing of consumers and the profitability of food manufacturers.

**Polymeric Particles of Antioxidant Properties Made with Novel Alpha-Tocopherol-Ascorbic Acid Surfactant.**

C.M. Sabliov<sup>1</sup>, C.E. Astete<sup>1</sup>, D. Dolliver<sup>2</sup>, <sup>1</sup>Louisiana State University Agricultural Center, Baton Rouge, LA, USA, <sup>2</sup>Southeastern Louisiana University, Hammond, LA, USA

Classical methods used to make polymeric nanoparticles from preformed polymers involve the use of a surfactant. It is hypothesized that nanoparticle physical properties and antioxidant activity could be controlled and manipulated by designing a custom-made bio-friendly surfactant (BFS) of desired characteristics. The aims of this research were: 1. to design and synthesize a BFS (derivative of vitamin E and vitamin C, EC) to be used in making polymeric nanoparticles and 2. to control and manipulate physical properties (size, size distribution, and surface charge) and antioxidant activity of poly(DL-lactide-co-glycolide) (PLGA) nanoparticles by applying the newly synthesized BFS. Ascorbic acid (vitamin C) and alpha-tocopherol (vitamin E) were coupled to yield the new EC surfactant of antioxidant properties. Polymeric nanoparticles were synthesized by nanoprecipitation in the presence of the EC surfactant. The particles measured 60 to 180 nm as a function of pH, ionic strength, polymer and surfactant concentrations, and exhibited superior antioxidant activity as compared to the EC surfactant alone. It was concluded that tailor-made bio-friendly surfactants could be successfully used to control the physical and antioxidant activity of polymeric nanoparticles specifically designed for food and drug delivery applications.

**AFTERNOON**

**FS&FF 2: Design of Successful Performing Interfaces**

Chair(s): K. Dewettinck, Ghent University, Belgium; and Y. Wang, Kraft Foods Inc., USA

**Freeze-thaw Stability of Water-in-Oil Emulsions.** Supratim Ghosh, D errick Rousseau, Ryerson University, Toronto, ON, Canada

The purpose of this research was to investigate the stabilization of water-in-oil (W/O) emulsions with two commonly-used food-grade emulsifiers ? polyglycerol polyricinoleate (PgPr) and glycerol monostearin (GMS). The isothermal (25  C) and freeze/thaw (25 to -80 to 25  C) stability of W/O emulsions stabilized with these two surfactants was evaluated as was the effect of two continuous phase lipids (canola oil and coconut oil) with different melting and solidification properties. Emulsion stability was determined with pulsed field gradient NMR droplet size analysis,

sedimentation, microscopy and DSC. During freezing, canola oil crystallized at a higher temperature than the dispersed aqueous phase, with the emulsion remaining stable irrespective of the emulsifier used. However, with coconut oil, the water droplets crystallized at a lower temperature than that of the oil phase. Depending on the emulsifier used, significant differences in emulsion stability were observed: with PgPr, the emulsion destabilized due to the rupture of the interfacial membrane surrounding the droplets. With GMS, the crystalline shell surrounding the dispersed droplets prevented coalescence and the emulsion remained stable after numerous freeze/thaw cycles. Overall, this work demonstrated that composition plays a significant role in the freeze/thaw stability of W/O emulsions.

**Fat Crystal Orientation at an Oil-Water Interface.** R. Ergun<sup>1</sup>, J. Grummer<sup>1</sup>, R. Hartel<sup>1</sup>, P. Spicer<sup>2</sup>, <sup>1</sup>University of Madison Wisconsin, Madison, WI, USA, <sup>2</sup>The Procter & Gamble Co., Cincinnati, OH, USA

Partial coalescence is caused by an extension of fat crystals through the oil/water interface from one globule that penetrates another fat globule to cause a clustering effect. The occurrence of partial coalescence is well known in various food applications, from thickening of whipping cream to fat destabilization in ice cream. However, the conditions that lead partial coalescence are not very well understood. In this study, partitioning of natural fat crystals at the oil/water interface in oil in water emulsions are being studied as a function of surfactant concentration, the rate of cooling and emulsion properties, including fat variety and content, droplet size and solid fat content. The occurrence of fat crystals extending through the interface and coalescing will be correlated with interfacial conditions (interfacial tension and contact angle) between the fat crystals and the oil/water phases. Preliminary studies with milk fat in water emulsions show that an increase in sodium dodecyl sulfate (SDS) and addition of co-surfactant, decanol, to the system increase the number of crystals emerging from fat droplets yet did not always lead to partial coalescence. A decrease in emulsion droplet size and an increase in cooling rate decrease the number of crystals emerging from fat droplets.

**Modulation of Interfacial Properties through Competitive Adsorption Kinetics between Two Different Polyelectrolytes onto Protein-coated Lipid Droplets.** Y.-H. Cho, E.A. Decker, D.J. McClements, University of Massachusetts, Amherst, MA, USA

Novel nutraceutical delivery systems can be created by coating bioactive lipid droplets with biopolymer layers. The objective of this study was to investigate whether biopolymer layer properties could be manipulated by controlled competitive adsorption of two different polysaccharides. Combinations of anionic polysaccharides with different charge densities and affinities for  $\beta$ -lactoglobulin ( $\beta$ -Lg) coated lipid droplets were studied. Protein-coated lipid droplets ( $\phi=1$  wt% oil, 0.045 wt%  $\beta$ -Lg,  $d_{32}=0.28\mu\text{m}$ ) were mixed with polysaccharide solutions at pH 7, then the pH was decreased to 3.5 to promote polysaccharide adsorption, and polysaccharide adsorption was monitored using electroacoustics (EA). The adsorption and stability of the emulsions containing different types of polysaccharides depended on their charge density and molecular structure due to electrostatic and steric repulsion, respectively. In a mixed system (pectin + carrageenan), carrageenan displaced pectin from the surfaces of BLg-coated droplets, but not vice versa. These studies facilitate the rational selection of optimum polysaccharide combinations and concentrations required to encapsulate, stabilize and release flavor and bioactive lipophilic components.

**Pickering Stabilization of Water-in-Oil Emulsions with Solid Lipid Nanoparticles.** Elham Hazfi, D errick Rousseau, Ryerson University, Toronto, ON, Canada

The purpose of this proof-of-concept study was to establish whether surface-active solid lipid nanoparticles (SLNs) can be used to kinetically stabilize water-in-oil (W/O) emulsions. SLNs were generated using microemulsions as nano-scale reactors. The phase behavior of mixtures of monostearin, Tween 20, ethanol and water was investigated using a pseudo-ternary phase diagram (PTPD) at 70 C. Oil-in-water microemulsion domains within the PTPD that yielded the highest proportion of solubilized monostearin were crash-cooled to 4 C to generate SLNs ~100 nm in size, based on dynamic light scattering and atomic force microscopy measurements. These particles were then concentrated and harvested before usage as emulsion stabilizers. To stabilize a mineral oil-based W/O emulsion, 0.5-2% (w/w) SLNs were added to the oil phase, and the oil, water and SLN mixture was homogenized using an impeller-type mixer. Characterization of sedimentation behavior, evolution in dispersed water droplet size distribution (with pulsed NMR) and microstructure showed that emulsion stability was most improved by addition of 2% (w/w) SLNs to the continuous phase. Fluorescence and polarized light microscopy showed that interfacially-adsorbed (Pickering) particles

as well as continuous phase crystals were responsible for stabilization of the W/O emulsions.

## TUESDAY

### AFTERNOON

#### FS&FF 3: New Processing Approaches for the Creation of Novel Food Structures

Chair(s): S. Metin, Cargill, Inc., USA; and G. Yang, Kelloggs North America Co., USA

**Characterization of Lipid Nanostructures Using SWAXS/DSC.** Daniel Kalnin<sup>1,2</sup>, Michel Ollivon<sup>2</sup>, <sup>1</sup>YKI Institute for Surface Chemistry, Stockholm, Sweden, <sup>2</sup>UMR CNRS 8612, Chatenay Malabry, France

Lipids are self-assembling molecules responsible for compartment formation in animal cells. Beside bilayer membranes, they also form all kind of aggregates and mesophases thanks to their aptitude to modulate interface curvature. Thus, lipid-based structures such as Solid Lipid Nanoparticles (SLN), liposomes, cubosomes, etc. which are potentially interesting for drug delivery in pharmacy can be also employed in food science for instance for controlled release. Our research focuses mainly on structural properties of lipid self-assemblies at a nanometric scale. Triglycerides (TAGs), the main constituents of fats, exhibit a complex monotropic polymorphism that frequently forecloses the study of thermal and structural properties of the fats. Mono and Diglycerides that are lyotropic and thermotropic show phase transitions of different orders. Naturally lipid structures self-organize into complex structures whose periodicity spans from a few nanometres up to hundreds of nanometres. As the range of organization is variable, it may affect both nanometric and macroscopic properties at the same time using lipids as a molecular building block. Mainly small and wide angle x-ray scattering coupled with DSC are used to investigate the thermal and structural properties of emulsified lipids.

**Crystallisation Mechanisms and Microstructure of Milk Fat - Effects of Cooling Treatments and Addition of Phospholipids.** L. Wiking<sup>1</sup>, V. De Graef<sup>2</sup>, E. Fredrick<sup>2</sup>, K. Dewettinck<sup>2</sup>, <sup>1</sup>University of Aarhus, Department of Food Science, Tjele, Denmark, <sup>2</sup>Ghent University, Department of Food Safety and Quality, Ghent, Belgium

Using DSC, synchrotron time-resolved X-ray diffraction and pulsed NMR, the crystallisation mechanisms of milk fat were elucidated. Under same crystallisation conditions the microstructure of the milk fat was analysed with confocal laser scanning microscopy and oscillatory rheology. The milk fat was cooled to 20°C by two different cooling rates, 0.1 and 10°C/min, respectively. Hereafter, the isothermal crystallisation at 20°C was monitored. The fast cooling resulted in two-step crystallisation, and a microstructure that consisted of smaller and more uniform crystals compared with the slower cooling. Consequently, fast-cooled milk fat showed a higher complex modulus, indicating a stiffer network, compared with the slower cooling. X-ray diffraction showed that the two-step crystallisation involved a polymorphic transition from  $\alpha$  to  $\beta'$  phase. Furthermore, oscillatory rheology was demonstrated to be very useful to detect polymorphic transition in crystallising anhydrous milk fat. Addition of a small amount of milk phospholipids (0.2 wt%) delayed the onset of crystallisation upon both isothermal and non-isothermal crystallisation. Small angle X-ray diffraction analysis revealed that the initial formation of a lamellar structure was inhibited by the phospholipids.

**A Microstructural Approach on the Isothermal Crystallization of Palm Oil.** V. De Graef<sup>1</sup>, L. Wiking<sup>2</sup>, F. Depypere<sup>1</sup>, S. Cabus<sup>2</sup>, M. Rasmussen<sup>3</sup>, B. Goderis<sup>2</sup>, K. Dewettinck<sup>1</sup>, <sup>1</sup>Faculty of Bioscience Engineering, Ghent University, Ghent, Belgium, <sup>2</sup>Department of Chemistry, Catholic University of Leuven, Leuven, Belgium, <sup>3</sup>Department of Food Science, Faculty of Agricultural Sciences, Aarhus University, Tjele, Denmark

A multi-methodological approach was used to study the isothermal crystallization of palm oil at four crystallization temperatures (18, 20, 22 and 25°C). Differential scanning calorimetry (DSC) and pulsed nuclear magnetic resonance (pNMR) were used to gain insight in the primary crystallization, while oscillatory rheology provided information on both primary crystallization and microstructural crystal network development and strength. Polarized light microscopy (PLM) and confocal scanning laser microscopy (CSLM) in particular revealed that differences in microstructure have to be taken into account besides the amount of solid fat to explain the higher network stiffness for samples that

crystallized at a lower temperature. At lower temperatures the fat crystallizes quickly, leading to a large number of small crystals. When these crystals start to aggregate, a very compact network structure is formed, giving rise to high dynamic moduli. At higher crystallization temperatures, fewer but larger crystals are generated that grow more slowly. As a result, a looser network structure is formed, leading to a lower complex modulus. In addition, CSLM revealed that at lower crystallization temperatures, more protruding crystalline filaments exist that bridge the crystalline aggregates through the remaining liquid fraction.

**Modifying Lipid Crystallization using Acoustic Waves.** Silvana Martini, Andreia Suzuki, Jiwon Lee, Utah State University, Logan, UT, USA

The functional properties of shortenings and lipids can be altered by changing processing conditions such as cooling rate, crystallization temperature, agitation and the addition of emulsifiers. High intensity ultrasound (HIU) can be used as a novel processing condition to modify the functional properties of lipids by either inducing or delaying their crystallization. Our study applies high intensity ultrasound (HIU) to lipid systems such as anhydrous milk fat (AMF), palm kernel oil (PKO) and cocoa butter (CB) to alter their crystallization behavior and therefore their functional properties. Results show that HIU induces the crystallization of AMF and generates small crystals. On the other hand, HIU slightly induced the crystallization of PKO while it delayed the crystallization of CB. This study shows that the effect of HIU on the crystallization behavior of lipids depends on the chemical composition of the lipid system, the crystallization temperature, the duration of the ultrasound signal and the time at which HIU is applied. This data suggests that HIU can be used as a novel processing tool to modify the functional properties of lipids.

**How Fundamental Rheology Can Predict Bread Volume.** F. Van Bockstaele<sup>1,2</sup>, I. De Leyn<sup>2</sup>, M. Eeckhout<sup>2</sup>, K. Dewettinck<sup>1</sup>, <sup>1</sup>Ghent University, Ghent, Belgium, <sup>2</sup>University College Ghent, Ghent, Belgium

Dough rheological properties are a key factor in the processing of bread products. An optimal balance between elastic and viscous properties is required to obtain bread with a desired volume and crumb structure. To study the viscoelastic properties of bread dough, fundamental rheological measurements are extensively applied. However, up till now, no convincing relationship between fundamental rheological measurements and bread volume could be established. In our research, the rheological properties of seventeen pure European wheat cultivars were analyzed and evaluated in relation to the bread volume. A relationship between fundamental dough rheological properties and bread volume was found. Of all rheological parameters under study, dynamic oscillation parameters showed the highest correlations with bread volume. Also, a clear relationship between creep-recovery and dynamic oscillation results was observed.

**Monoacylglycerols as Crystallization and Stability Modifiers of Recombined Cream.** E. Fredrick<sup>1</sup>, P. Walstra<sup>3</sup>, H. Zijtveld<sup>2</sup>, S. Fischer<sup>2</sup>, K. Dewettinck<sup>1</sup>, <sup>1</sup>Ghent University, Belgium, <sup>2</sup>Friesland Foods Corporate Research, The Netherlands, <sup>3</sup>Wageningen University, The Netherlands

Whipped cream can be defined as a destabilized fat rich dairy product in which the air bubbles and the serum phase are immobilized with a continuous network of fat globules. These fat globules are capable of building a structure due to the occurrence of partial coalescence during the whipping process. The presence of fat crystals is thereby indispensable. Monoacylglycerols are small molecule surfactants which can affect a dairy oil-in-water emulsion against partial coalescence and thereby the whipping properties. The effect of these monoacylglycerols largely depends on the length and the degree of saturation of the fatty acid chains because they probably differently affect the nucleation, the crystal growth, the solid fat content of milk fat and the polymorphism, the morphology and size of the milk fat crystals. In this research the effect of monolaurin, monostearin and monoolein is investigated on the crystallization mechanism and kinetics of dairy creams. DSC, NMR and XRD are the main techniques applied.

**Compositional Effects on the Occurrence of Fat Bloom in Filled Chocolates.** Nathalie De Clercq, Frédéric Depypere, Koen Dewettinck, Ghent University, Ghent, Belgium

The by far most important reason for the shelf life limitation of chocolate products is the occurrence of fat bloom. A bloomed chocolate is characterized by a change in colour and loss of gloss giving a greyish white appearance to the chocolate surface. In filled chocolate like pralines this problem is even more pronounced since the oils in the filling

tend to migrate through the chocolate to the surface. Bloom depends on the structure resulting from formulation of the fat, emulsifier and dispersed solid component compositions and the crystallization processing conditions. The exact mechanism behind this phenomenon is not yet completely understood and there is also no thorough insight in the influence of chocolate and filling composition and processing variables. In this set-up the composition of the chocolate and filling in model systems was varied and the influence on the appearance of fat bloom was evaluated by applying different methods: image analysis, HPLC-ELSD and DSC.

**Effects of Fat Crystal Network Structure on the Physical Properties of Interesterified and Non-interesterified Fully Hydrogenated Canola Oil and High Oleic Sunflower Oil Mixtures.** Latifeh Ahmadi, Alejandro Marangoni, University of Guelph, Guelph, ON, Canada

Interesterified fully hydrogenated fats (FHF) and oleic acid rich blends are being considered to create semi solid fats with particular physical properties and improved nutritional characteristics. Crystallization kinetic parameters (Avrami index and rate constant) were similar for the non-interesterified (NI) and interesterified (IE) blends when compared as a function of SFC. X-ray analysis revealed a predominance of the  $\beta'$  form in the IE blends. A higher Db in the CI-sample relative to the EI sample, at an equivalent area fraction, was attributed to the homogeneity in the distribution of crystalline mass. The storage modulus ( $G'$ ) and yield force increased with FHF concentration in the blends and was affected strongly by interesterification, but it showed a weak correlation with SFC content. An increase in  $G'$  at 30°C upon interesterification was attributed to a decrease in the fractal dimension of the fat crystal network which translated into a decrease in crystal cluster size. The higher  $G'$  in the CI-samples relative to the EI-samples was due to a higher  $\lambda$  parameter which is directly proportional to the strength of interparticle interactions and inversely proportional to crystal size.

## WEDNESDAY

### MORNING

#### **FS&FF 4: Advanced Multi-Disciplinary Capabilities to Allow Identification and Characterization of Key Structural Mechanisms**

Chair(s): D. Kittleson, General Mills Inc., USA; and J. Webb, Reading Scientific Services Ltd., UK

#### **Coherent Anti-Stokes Raman Scattering Microscopy for Rapid Chemically Selective Imaging of Food Samples.**

E.O. Potma<sup>1</sup>, M. Paques<sup>2</sup>, <sup>1</sup>Department of Chemistry, University of California, Irvine, CA, USA, <sup>2</sup>FrieslandCampina Corporate Research, Deventer, The Netherlands

Non-invasive, rapid microscopic identification of basic compounds in food samples, such as water, lipids, protein and carbohydrates, is a crucial part of full structure characterization and the understanding of structure - function relationships. This insight is key for controlling the functional properties of food, overcoming product structural failures, and engineering new product concepts. As such, knowledge of structure - function relationships has a direct impact on consumer products, products used by food services and professional users (bakerys), and ingredients used by manufacturers of consumer products. Chemical identification on a microscopic level can be achieved by optical microscopy in combination with fluorescent labeling techniques. Nonetheless, introducing fluorescent probes to the sample is often undesirable in terms of interfering with the intrinsic properties of the specimen. Coherent anti-Stokes Raman scattering (CARS) microscopy is an alternative imaging method, which avoids the use of fluorescent labels while maintaining chemical selectivity. Unlike in fluorescence microscopy, contrast in the CARS microscope is derived from the vibrational properties of molecules. CARS microscopy offers a direct means to visualize the distribution of water, lipids and proteins in food samples at high sub-micron resolution in real-time. In this talk, the principles and applications of the CARS technique in the food sciences will be discussed and illustrated with some examples.

**The Impact of Lipid-based Fillings on Early Fat Bloom Development on Chocolate.** Hanna Dahlenborg, Birgit Brandner, Anna Fureby, Daniel Kalnin, Fredrik Johansson, YKI, Institute for Surface Chemistry, Stockholm, Sweden

The objective was to compare fat bloom development between plain milk chocolate and milk chocolate with two

different lipid based fillings. The study focussed on the evaluation of surface bloom using LVSEM for its capacity in detection of fat bloom in early stages. The chocolate samples were stored at three different temperatures: 18, 22,5 and 26°C. LVSEM showed a complex surface with numerous imperfections. After analysis with Confocal Raman Microscopy it could be envisaged that the observed as bubbles and pores, are most likely voids that continue inside chocolate surface at least 20 µm. We speculate that this is where liquid oil could be transported to the chocolate surface. All samples stored at 26°C developed fat bloom in form of needle like crystals. The time dependence of fat bloom development differed between plain and filled chocolate. This was connected to the filling and chocolate composition. Formation of fat bloom crystals was accelerated on chocolate with a softer filling, while a harder filling with CBE added indicated on retardation of development of fat bloom. Some of the observed 'bubbles' on the chocolate surface could be connected to the formation of fat bloom detected by LVSEM. Therefore, a reduction in quantity of bubbles could lead to a reduction in quantity of fat bloom crystals.

**Pre-bloom Topographical Roughening of Dark Chocolate Observed with Optical Profilometry.** Sopark Sonwai<sup>1</sup>, Rizwan Khan<sup>2</sup>, D errick Rousseau<sup>2</sup>, <sup>1</sup>Department of Food Technology, Silpakorn University, Nakornprathom, Thailand , <sup>2</sup>Department of Chemistry and Biology, Ryerson University, Toronto, Ontario, Canada

The aim of this investigation was to ascertain the early microstructural stages of fat bloom development in dark chocolate. The surface morphology and polymorphic behavior of dark chocolate subjected to thermal cycling between 20 and 28, 30, 32 or 34°C were examined using optical profilometry (OP) and differential scanning calorimetry (DSC). Cycling to any of these temperatures did not lead to visual bloom, though notable effects on microstructure and polymorphism were noted. The surface of the control chocolate was glossy and consisted of small, evenly distributed crystals. DSC confirmed the presence of form V crystals in all controls, with form VI crystals appearing with cycling to 32 and 34°C. Based on OP, the samples cycled between 20 and 30, 32 or 34°C consisted of jutting crystals interspersed with valleys of smooth areas consisting of smaller crystals. As a result of cycling, surface roughness increased for all samples. Decomposition of the roughness into low and high-frequency components revealed a significant contribution of waviness (the low-frequency component) to overall roughness. High-frequency features (i.e., crystals) also contributed, but less so than waviness, which dominated with larger temperature gradients. This study demonstrated that significant changes take place within chocolate prior to the visible onset of surface fat bloom.

### **Coupled DSC and X-ray Study of the Mesomorphic Phases of Monostearin and Stearic Acid in Water.**

Alexander Zetzi<sup>1</sup>, Michel Ollivon<sup>2</sup>, Alejandro Marangoni<sup>1</sup>, <sup>1</sup>Department of Food Science, University of Guelph, Guelph, Canada, N1G 2W1, <sup>2</sup>Universite Paris Sud, UMR CNRS 8612, Chatenay-Malabry Cedex, France

Differential Scanning Microcalorimetry coupled to Synchrotron Small Angle X-ray Scattering (SAXS) were used to characterize the mesomorphic phases of monostearin. A series of dispersions of monostearin and stearic acid (95:5 w/w) in alkaline water were prepared in the range 5 to 90% (w/w). Samples were subjected to both melting and cooling regimes from 45 to 81degC at 2degC/min in a Microcalix machine, while X-ray spectra and the microcalorimetric trace were being collected simultaneously. Using these two techniques, 2D phase diagrams of the mesomorphic phases (lyotropic and thermotropic liquid crystalline phases as well as the crystalline, hydrated gel phases) of monostearin were constructed, one for heating and one for cooling, and the typical X-ray diffraction pattern for each of the phases was identified. Polarized light microscopy was also used in an attempt to differentiate and identify the mesomorphic phases. Furthermore, the largest d-spacings (which includes the thickness of the water domain and the lipid bilayer) for each of the different monostearin concentrations was plotted at 73degC during heating and when cooling in order to better understand the water binding capacity of the different mesomorphic phases. This information will provide guidance for the use of these unique phases in food and pharmaceutical applications.

### **Phase-Contrast X-ray Tomography of Food Products to Enable the 3 Dimensional Visualisation and**

**Measurement of the Internal Structure Without Mechanical Sectioning.** L.A. Brownlow<sup>1</sup>, C.J. Sheffield-Parker<sup>1</sup>, S.C. Mayo<sup>2</sup>, <sup>1</sup>XRT Limited, Port Melbourne, VIC 3207, Australia, <sup>2</sup>CSIRO Material Science and Engineering, PB 33, Clayton South, VIC 3169, Australia

Absorption contrast has been the normal X-ray imaging modality for more than 100 years, recently though phase contrast has become well recognised as being strong where absorption contrast is weak. This is particularly useful for imaging of low density materials with included secondary components and for the visualisation of cracks, pores and bubbles, all features commonly found in food products. We discuss the functional requirements for phase-contrast imaging in a laboratory environment together with arrangements for tomography (microCT) and procedures for phase retrieval to produce projections for the reconstruction, visualisation and 3D measurement of food product structures. A number of practical examples will illustrate the techniques used and form the basis for a brief discussion of the benefits and the issues.

#### **FS&FF 4.1: Food - Body Interactions**

Chair(s): I. Appelqvist, CSIRO, Australia; and M. Paques, Friesland Foods, Netherlands

**Lubrication of Food Emulsions Between Compliant Substrates.** J.R. Stokes<sup>1</sup>, D. Rossetti<sup>2</sup>, E. Wantling<sup>2</sup>, C. Zhang<sup>3</sup>, A. Taylor<sup>3</sup>, A.-M. Williamson<sup>2</sup>, <sup>1</sup>University of Queensland, Division of Chemical Engineering, Australia, <sup>2</sup>Unilever Research Discovery, UK, <sup>3</sup>Division of Food Sciences, University of Nottingham, UK

The sensorial response generated during and after consumption of food/beverages results from the interaction of food components with saliva and the soft tissues of the oral cavity during oral processing. It has become increasingly important to understand this process due to the need to develop foods and beverages that are low in fat, sugar and salt but high in bioactives (so-called phytonutrients). The transformation of the food/beverage to very thin films and its confinement between deformable rubbing surfaces (i.e. the tongue and palate) coated with a salivary film is considered to influence mouthfeel, taste and aroma. We explore this process by utilising our recently developed techniques in narrow-gap rheometry and soft-contact tribology to predict the response of complex multiphase fluids during oral processing. We demonstrate that during confinement, the dynamic response of a range of systems (including biopolymer solutions, food emulsions, microgel suspensions, as well as model tea beverages) no longer necessarily depend on their bulk rheological properties, but also on the local dynamics and micro/nano-structure of the individual phases, the adsorption of surface active constituents onto the biosubstrates, and the interaction with an pre-adsorbed salivary film. We also demonstrate how these measurements translate to a sensory response. In particular, we show how sensory perception of flavoured biopolymer solutions depends on their tribological properties and the apparent viscosity under thin film conditions and at high shear rates ( $10^5 \text{ s}^{-1}$ ). Our studies also indicate that astringency perception is not necessarily related to the aggregation of tea catechins with salivary proteins that cause saliva film depletion from oral surfaces and an increase in friction, and that astringency is unlikely to be a purely tactile percept as often stipulated. These new thin film techniques are offering valuable new insights into the oral processing and perception of foods.

**Studies of Food Oral Processing in Relation to Food Structure, Rheology, and Sensory Perception.** Jianshe Chen, Department of Food Science, University of Leeds, Leeds LS2 9JT, UK

Food oral processing serves two main purposes: to break down food particles for easy swallowing and to have sensory appreciation of food texture, taste and flavour. The (micro)-structure and mechanical properties of food play a key role in this process. In this work, oral behaviour of a wide range of foods have been studied in relation to food properties and sensory perception. The formation and properties of food boluses were found to be highly food-dependent. For dry biscuits, the number of chewing cycles tends to increase with food hardness. However, it was found that a minimal number of chewing was needed however brittle a biscuit is. This suggests that a minimum oral resident time is needed in eating solid foods probably for the purposes of producing enough saliva and for sensory pleasure. The amount of saliva in food boluses varies, dependent on the properties of the food. For wet solid food, the participation of saliva was small (17 % for raw carrot), but for dry solid foods, the contribution of saliva was much higher (44 % for roasted peanut and 36 % for roasted macadamian nut). Activities of facial muscles have also been investigated. It was observed that food hardness and stickiness were the two key parameters influencing activity pattern of facial muscles.

Chair(s): D. Rousseau, Ryerson University, Canada

### **Effects of Addition of a Palmitic Sucrose Ester on Low-*trans*-Fat Blends Crystallization in Bulk and in Oil-in-Water Emulsions.**

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The effect of addition of a highly hydrophobic emulsifier, the sucrose ester P-170, on isothermal crystallization behavior of a high-melting fraction of milk fat (HMF) and its mixtures with 20 or 40% sunflower oil (SFO), both in bulk and in emulsion systems, were studied by nuclear magnetic resonance (NMR) and by time-resolved in-situ small angle synchrotron X-ray diffraction (SAXS). NMR studies showed that the effect of P-170 on the overall isothermal crystallization kinetics in bulk phase was acceleration or delay depending on supercooling. In emulsion systems, however, the effect was always acceleration for all temperatures selected. With the aid of synchrotron X-ray diffraction, it was possible to establish that the different effects caused by P-170, as described by SFC curves, were strongly related to the effects of P-170 on fat polymorphism, specially to the value of the time interval of co-existence of the alpha and beta forms.

### **Physical Chemical Stability of Emulsions Formulated with Marine Oil.**

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The development of an effective strategy to prevent undesirable changes in the properties of a particular food emulsion depends on the dominant physicochemical mechanism. It is therefore important for food scientists to identify the relative importance of each mechanism. The n-3 fatty acids, such as  $\alpha$ -linoleic acid (ALA), eicosapentaenoic acid (EPA, 20:5 n-3) and docosahexaenoic acid (DHA 22:6 n-3) have many health benefits. Oil enriched in these fatty acids is of great importance for supplement makers and functional food formulators. Stability of emulsions formulated with commercial fish oil, trehalose, and sodium caseinate (NaCas) as emulsifier was studied using a TMA 2000 analyzer. The reading head is composed of a pulsed near-IR light source ( $\lambda = 850$  nm) and two synchronous detectors. The samples were put in a cylindrical glass measurement cell and the backscattering (BS) and transmission (T) profiles as a function of the sample height (total height = 70 mm) were studied in quiescent conditions at 22.5°C. Five concentrations of NaCas were used: 1, 2, 3, 4 and 5 wt%. Droplet size diminished as sodium caseinate concentration increased. For 1 wt% NaCas concentration migration was the most important mechanism of destabilization. As the concentration increased coalescence occurred and became the main mechanism.

### **Formation of Core-Shell Model for Encapsulation of Bioactives in Solid Lipid Nanoparticles (SLN).**

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Solid lipid nanoparticle (SLN) suspensions are potential delivery systems that can be used for the incorporation of bioactives into food. However, particle aggregation and the migration of bioactives towards the SLN interface, where oxidation is accelerated, limit their industrial utilization. A possible strategy to overcome these problems is to confine bioactives in the particle core surrounded by a solid shell. In this work, we utilize highly hydrophobic emulsifiers (HHE) to explore possibility of the formation of core-shell model in SLN that are suitable for the encapsulation of bioactives. Preliminary results indicated that high-melting HHE modified the crystallization behavior mainly by increasing the crystallization temperature due to freezing of the surface layer. This suggests the formation of a solid shell via a mechanism of template-assisted nucleation at the interface. Optical observations indicated that the type and concentration of emulsifiers are quite influential on stability against aggregation. Additional experiments were performed to confirm the formation of core-shell structure in SLN. Results from this work provided important information about the potential use of specific emulsifiers, and introduced a new strategy for the design of stable

delivery system that can be applied in the food industry.

### **Polymer Gelation of Oil.**

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With the increasing focus of the market on the reduction of saturated fat in the diet, the demand for unsaturated alternatives with the same functionality in food applications has seen a similar increase. Small molecule organogelators are capable of achieving this to some extent, but their cost can be prohibitive for the food industry. Polymers may offer a promising and more economical alternative to other gelators with similar functionality. There is little to no precedent for the use of polymers as gelators of edible oils. The present research surveyed a variety of polymers for their potential, including maltodextrin, cyclodextrin, and cellulose derivatives. Of the aforementioned, ethylcellulose seems to be the most promising option. A study of the effect of temperature, time, surfactant addition, cooling rate and shear on gelation behaviour is included. Small deformation controlled stress rheology was used to characterize gelation kinetics, while differential scanning calorimetry was used to examine the thermal transitions of the polymer in the oil. Scanning electron microscopy was used to inspect the microstructure of these polymer gels.

### **Effects of Oil Type and Surface Coverage on Physical Properties and Stability of Solid Lipid Nanoparticles (SLN) Suspensions.**

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The main purpose of this research was to investigate whether varying the oil type and surface coverage would affect the crystallization behavior and stability of a model octadecane solid lipid nanoparticle (SLN) suspension that is encapsulated with omega-3 rich fish oil. The effects of the oil type and ratio as well as the type and concentration of emulsifier(s) on the thermal behavior, particle size, optical properties and stability were investigated. We also tested the possibility of using mixtures of short and long chains emulsifiers. Particle size and appearance of SLN was dependant on the type and ratio of emulsifiers. DSC showed that the crystallization and melting behavior were strongly dependent on the oil type. The presence of high-melting surfactant molecules initiated nucleation active sites at the droplet interface. Attempts were made to change the surface properties by using a mixture of hydrophobic emulsifiers and varying the ratio of the high- to low-melting emulsifiers. The research results obtained from this work highlighted the potential use of specific emulsifiers to control the formation of highly stable SLN suspensions. Selected systems could be used as potential delivery systems in functional food.

### **Microencapsulation of Carotene: Effect of Process Variables and Wall Materials on Product Characteristics and Carotene Stability.**

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The purpose of this study was to investigate the physical characteristics of encapsulated powder of carotene produced by using various wall materials. Microencapsulation of carotene was done by using gum arabic, maltodextrine and modified starch. Encapsulation efficiency and entrapment efficiency was found for various parameters such as temperature, solid percentage and core percentage. These microcapsules were evaluated for encapsulation efficiency, surface oil, total oil, peroxide value, moisture content and carotene stability. 5% oil loading, 40% solid loading at 1400C temperature showed highest i.e. 92% encapsulation efficiency. Gum Arabic showed highest encapsulation efficiency i.e. 86% while maltodextrin and modified starch showed 68% and 83 % respectively. In binary blends GA:MS blend provided 1.5 times better protection than GA alone. Ternary blend (GA:MD:MS); (4/6:1/6/:1/6) showed highest encapsulation efficiency than any other blend. It was concluded that above blend provided better retention and gave highest encapsulation efficiency. Hence, gum arabic can be partially substituted by maltodextrin and modified starch and can become an economical and better matrix for carotene.

### **The Influence of Fat Crystallization on Structural Partial Coalescence in Ice Creams Containing Palm Kernel Oil and High Oleic Sunflower Oil.**

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Structure formation in ice cream, specifically partial coalescence of fat, is of great importance as it determines the sensory attributes and quality of the final product. Various ratios (40-100%) of palm kernel oil (PKO) diluted with high oleic sunflower oil (HOSO) and two different emulsifiers, glycerol monostearate (GMS) and glycerol monooleate (GMO), were chosen to investigate how solid-to-liquid fat ratios effect the structure, texture and quality of ice cream. Particle size distributions of fat globules, turbidity, overrun, meltdown resistance and air cell distributions were used to characterize the samples. Oscillatory thermo-rheology (OTR) was also used to correlate temperature and rheological properties with the microstructure of the ice creams. All experimental approaches agreed that the addition of a saturated emulsifier encouraged an increase in structural stability, overrun and a more rapid meltdown as solid fat content increased. Characterization of the samples containing GMO showed more variation, introducing the possibility of additional structural interactions between the unsaturated emulsifier and the fat crystal network. Rheological properties as a function of temperature agree with these results, emphasizing the influence of the solid-to-liquid fat ratio on frozen foam microstructure and its effect on overall quality characteristics of ice cream.