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Edible Applications Technology Interest Area Technical Program Abstracts

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EAT 1: Lipid Gels

Chairs: A.R. Patel, Ghent University, Belgium; and C.C. Udenigwe, Dalhousie University, Canada

Importance of p-p Stacking in Urea and Sorbitol-based Gelators. M.A. Rogers and A. Singh, University of Guelph, Canada.

Dimethylurea, when modified to N,N' dibenzylurea changes the relationship between molecular structures and their nanostructures and supramolecular architectures. Through a series of synthetic modifications, we have probed: 1) what functional groups enhance self-assembly; 2) how intermolecular forces drive assembly into different supramolecular structures (e.g., fibers, ribbons, etc.); and 3) how changes in molecular structure and non-covalent interactions alter the solutions that are transformed into molecular gels. Using the new molecules derived from DS and N,N' dimethylurea, the importance and nature of hydrogen bonding, p-p stacking and electrostatic interactions on the modes of self-assembly and gel formation can be probed independently.

Revisiting Mixtures of Stearyl Alcohol/Stearic Acid as an Oleogelator System. C. Blach[1], A.J. Gravelle*[2], F. Peyronel[2], J. Weiss[1], S. Barbut[2], and A.G. Marangoni[2], [1]Food Physics & Meat Science, University of Hohenheim, Germany, [2]Dept. of Food Science, University of Guelph, Canada.

Mixtures of stearyl alcohol and stearic acid (SO and SA, respectively) were arguably one of the first identified oleogelators with potential for food applications. A synergistic enhancement in gel strength was identified at a ratio of 7:3 SO:SA, which was attributed to their needle-like crystal morphology. In the present study we have meticulously characterized this system with a variety of techniques at different gelator ratios. Large deformation tests and DSC results were consistent with previous studies, the latter suggesting a possible eutectic behavior near the synergistic ratio. X-ray analysis indicated there is a continuous change in solid state structure was observed from SO to SA, without any remarkable changes at the synergistic ratio. Light micrographs suggest the crystal network which forms the hardest gels (8:2, 7:3) and weakest gels (5:5, 4:6) are similarly structured. Accelerated oil loss tests show the stability of the gels mirror the mechanical strength with ~1wt% oil loss in the firm gels and >10wt% in the weakest formulations. We hypothesize that the observed differences in mechanical strength and stability can be explained by a combination of crystal morphology and the strength of crystal-crystal interactions. To this end, presently we are continuing to characterize crystal morphology and working towards determining the Hamaker constants of the different SO:SA formulations.

Stearyl Alcohol/Stearic Acid Mixtures in the Presence of Ethylcellulose: Creating New Oleogelator Systems to Diversify Functionality. A.J. Gravelle[1], M. Davidovich-Pinhas[2], S. Barbut[1], and A.G. Marangoni[1], [1]Dept. of Food Science, University of Guelph, Canada, [2]Technion-Israel Inst. of Technology, Israel.

It was recently reported that fatty acids and fatty alcohols greatly enhance the mechanical strength of ethylcellulose (EC) oleogels, and can impart plasticity on the gel network. Additionally, mixtures of stearyl alcohol and stearic acid (SO and SA, respectively) are also capable of structuring edible oils, particularly at 7:3 SO:SA, where a synergistic enhancement occurs. When used in combination, these two systems produce a new type of gelator with unique microstructure, rheological, and mechanical behavior. Rheological data demonstrated that the presence of SO:SA plasticizes the EC network, causing a depression in the crossover temperature during gelation. Microstructural analysis showed EC had a strong influence on the size and arrangement of the crystal network, producing smaller, oriented, fan-like needle structures, as opposed to a network of larger, randomly distributed needles in its absence. However, X-ray diffraction patterns suggest the polymorphism of the SO:SA crystals is unchanged in the presence of EC. Thermal analysis indicates a more rapid crystallization process of the SOSA molecules in the presence of EC, favoring nucleation and thus smaller crystals. This work demonstrates the potential to use EC as a means to influence crystallization processes in fat crystal networks, and may be applicable to other oleogelator systems.

Comparative Study on the Crystallization Behavior and Application of Beeswax, Carnauba Wax, Rice Bran Wax, and Oryzanol/Sitosterol Oleogels. Z. Meng, X. Zhu, and Y. Liu, Jiangnan University, China.

Oleogels offer an alternative to the use of "bad" fats such as saturated and trans fats. Two organogelling strategies were studied comparatively, namely crystal particles (beeswax, carnauba wax, and rice bran wax) and self-assembly (oryzanol/sitosterol mixture, 60/40, w/w), for gelling canola oil.

XRD analysis indicated that rapidly cooling process had a little effect on the contents of polymorphism. Isothermal crystallizations of crystal particle oleogels were tested under 30, 40, 50°C. $T_{1/2}$ varied between 0.09 and 1.00s indicated that the oleogels crystallized rapidly in an instantaneous nucleation, linear growth pattern. Non-isothermal crystallization of crystal particle oleogels analyzed by Mo method showed that the crystallization behaviors were relevant to cooling rates. The submicroscopic structure of self-assembly (10%, w/w) based oleogels was studied by SAXS and Cryo-SEM. Three general models for *q*-dependence of the scattering were built. The tubule with a radius of R_{out} = 4.0nm, R_{in} = 3.9nm were obtained with fitting test data into the model of a tubule with walls of a finite thickness. Application evaluation showed that carnauba wax and rice bran wax oleogels could be used as spreads, and beeswax oleogels could be used for produce the bread and cookie shortenings.

Mixed Component Oleogels Prepared Using Synergistic Combinations of Monoglyceride and Phytosterols.
M.D. Bin Sintang[1,3], A.R. Patel[1,3], A. Lesaffer[2], and K. Dewettinck[3], [1]Vandemoortele Centre of Lipid Science & Technology, Lab. of Food Technology & Engineering, University of Ghent, Belgium, [2]Vandemoortele R&D Centre, Belgium, [3]Lab. of Food Technology & Engineering, University of Ghent, Belgium.

Monoglyceride in combination with phytosterol have shown a promising synergistic combination especially at ratio of 8:2 (MG:PS), in which the rheological properties of combination outperformed the properties of single structurant. In order to have successful appeal, the oleogel must have comparable characteristic as commercial spread. Therefore, it is very important to investigate the gelation formation of crystal in the oleogels for further application. In this study, commercial phytosterol is incorporated into the system to reduce the dependency towards monoglyceride to structure the sunflower oil into solid-like properties. This approach may help to reduce crystal agglomeration that usually associate with monoglyceride. Due to that, we investigate the crystallization properties of oleogel by using DSC in pre-defined time interval by the mean of comparing the melting curve. Moreover, the oleogel is subjected to time sweep measurement for three hours period in rheometer. The crystallization and microstructure development could be elucidated by using the modulus and delta value obtained from that experiment. CRYO-SEM is used to define the crystalline structure of the oleogels. In addition, polarized light microscopy is use to monitor the crystal development, as previous study have shown the incorporation of phytosterol leading to disintegration of monoglyceride crystal.

Understanding the Oil Gelling Properties of Natural Waxes.
A.R. Patel, University of Ghent, Belgium.

Natural waxes have been receiving a lot of interest due to their capability of gelling vegetable oils at significantly lower crystalline mass fractions ($w_c << 0.1$). Although, a lot of work has been published in the last few years, a comprehensive understanding of the gelation mechanism of waxes is still lacking. The aim of this study was to use a detailed characterization to gain new insights into the gelation behavior of six natural waxes (differing in the relative proportion of chemical components). Flow and dynamic rheological properties of gels prepared at critical concentrations of waxes were studied and compared using drag and oscillatory shear tests. Although, none of the oleogels satisfied the rheological definition of a 'strong gel' (G''/G' (ω) < 0.1), the strongest gel (highest critical stress and dynamic and apparent yield points) was obtained not with a mono component wax but with wax containing wax esters along with a higher proportion of fatty alcohols suggesting a role of surface active fatty alcohols in strengthening of the microstructure. The yield-pseudo-plastic behavior of oleogels was evident from the dynamic and apparent viscosity plots. The rheological behavior was combined with microstructure studies to have a comprehensive understanding of the gelation behavior.

Edible Liquid Oil Structuring via Protein-stabilized Emulsions. I. Tavernier, J. Goemaere, K. Dewettinck, and A.R. Patel, Lab of Food Technology & Engineering, Ghent University, Belgium.

The traditional way of structuring oil using high amounts of saturated fats is being perceived as unhealthy by modern consumers. This resulted in a considerable amount of research in structuring oil-based products with materials different from these high saturated fats. In this research, we report for the first time on the use of an emulsion-templated approach using proteins to prepare oil gels (or oleogels).

The approach consists of the production of highly concentrated oil-in-water emulsions ($\phi_{oil} > 0.6$) stabilized with soy protein isolate, followed by the evaporation of the water phase which results in the trapping of the oil in a physical network of soy protein.

The microstructure of the concentrated emulsions and the oleogels was elucidated using advanced microscopic techniques such as cryogenic scanning electron microscopy and confocal scanning laser microscopy. Follow-up of the oil leakage revealed no destabilization during 4 weeks. The rheological properties of the oleogels were determined by subjecting them to both oscillatory and flow measurements.

The prepared oleogels have a unique structure and are promising for pharmaceutical and food applications since only food-grade materials were used to transform liquid oil into a solid-like matrix.

EAT 1.1/IMG 1: Structure Effects on Oil Binding

This session developed in conjunction with the Agricultural Microscopy Division (Imaging Techniques Interest Area).
Chairs: G. Cherian, Kellogg North America Co., USA; and M. Willson, LipoLogic Consultancy LLC, USA

Palm Based Structured Oils for Trans-free and Reduced Sat Solution. G. Wang, L. Liu, and G.P. McNeill, IOI Loders Croklaan LLC, USA.

Structured fats and oils provide the desirable physicochemical and sensory attributes to food items. With FDA's announcement to ban PHO last year, formulation strategies based on palm are becoming commercially viable because they are capable of delivering the structure without altering sensory and shelf life stability. Three types of structured technologies are reviewed for different food applications, e.g. fractionation, oil blending, and fat modification. Fractionated palm oils satisfy clean label requirement for variety applications, such as peanut butter stabilizer, microwave popcorn with no wick issue. Oil blending is another way to create structured oils with reduced saturated fat content. These trans-free, no hydro, reduced Sat formulations are important to industry with clean label demand. Reduced saturation can also be achieved through fat modification. This approach creates new fats with fast set-up crystallization profile. Customers can apply these approaches to replace pho but still maintain current shelf life stability and sensory acceptance.

Effect of High Intensity Ultrasound, Agitation, and Crystallization Temperature on the Crystallization Behavior of Interesterified Soybean Oil. J.V. Kadamne and S. Martini, Utah State University, USA.

Interesterified soybean oil (M.P. 41.9±0.5°C) was crystallized at 29 and 34°C without and with the application of high intensity ultrasound (HIU). Two crystallization set-ups were studied: (i) continued agitation for entire crystallization process (60min) (CA), and (ii) agitation for 10min (A10). Sonication and agitation did not affect induction times of nucleation while crystal morphology, crystallization kinetics, and viscoelasticity were all affected by these processing variables. For e.g., HIU reduced crystal sizes and increased viscosity (5.7±1.9 to 3,057.5±318.5Pa.s) and elasticity (86.7±4.0 to 95,955±2735Pa) of crystalline networks obtained at 29°C under A10 condition. Similar increases in viscosity and elasticity were observed when samples were crystallized at 34°C under A10 condition. However, this effect was not as significant for samples crystallized under the CA condition. HIU also increased crystallization rates for all conditions tested. Kinetic constants obtained from an Avrami fit increased from 1.73 $x10^{-5}$ to 7.28 $x10^{-5}$ min^{-n} for samples crystallized at 29°C A10 without and with sonication, respectively, and up to 7.9 10^{-9} to 2.1 10^{-7} for samples crystallized at 34°C A10 without and with sonication, respectively. This rise in crystallization rate was also observed for samples crystallized under the CA condition.

Effect of Sugars on the Crystallization Behavior of Confectionery Fat-hydrogenated Palm Kernel Oil. H. Zhang[1,2], R.F. He[2,1], Q. Shen[1], Y.L. Bi[2], and X.B. Xu[1,2], [1]Wilmar (Shanghai) Biotechnology Research & Development Center Co., Ltd., China, [2]Henan University of Technology, China.

The effects of different sugars on the isothermal crystallization rate, thermal properties, hardness, and viscosity of hydrogenated palm kernel oil were studied. The results showed that the crystallization rate of hydrogenated palm kernel oil was promoted by sugars. The crystallization rate of sample additioned with lactitol was the fastest, followed by isomaltulose, sucrose, and maltitol. After adding sugars, the products density was increased, which led to the increase of hardness, especially for adding lactitol, which had the highest value. For the product viscosity, the highest viscosity was observed by adding isomaltulose, followed by the maltitol, isomaltulose, and sucrose gave the lowest viscosity. Overall, maltitol may act as one of the sugar substitutes for chocolate.

Arrested Coalescence of Droplets Containing Crystalline Fat Networks. A. Thiel, University of Wisconsin-Madison, USA.

In oil-and-water emulsions, a phenomenon known as arrested coalescence, can occur when two fat globules begin to coalesce but are stopped from fully merging. Micromanipulation techniques were used to bring two oil droplets into contact to gain a more microscopic view of coalescence. Here, four different combinations of fats were investigated microscopically to map full coalescence to total stability. Increasing amounts of a high melting fat (higher solid fat content), with larger elastic modulus of the fat crystalline networks, decreased extent of coalescence. To achieve the same degree of coalescence, emulsions prepared using anhydrous milk fat (AMF) or palm oil (PO) required a larger elastic modulus than emulsions made using coconut stearin (CS) or palm kernel oil (PKO). Extent of coalescence of AMF or PO droplets changed linearly with log G' while samples containing PKO or CS had an exponential relationship with log G'. Droplet diameter also was shown to affect coalescence. The degree of coalescence decreased for all fats investigated as diameter increased from 10 to 90mm. Understanding when oil droplets undergo full coalescence, arrested coalescence, or total stability microscopically allows for predictions of their behavior in foods like ice cream and whipped topping.

Surfactant-mediated Interfacial Crystallization of Solid Fat-encapsulated Water-in-Oil Emulsions. N.L. Green, T. Tran, and D. Rousseau, Ryerson University, Canada.

We have previously shown that shear-crystallized fat/oil mixtures will produce spheroidal fat crystals. The addition of 10% (w/w) water to these systems can form encapsulated droplets surrounded by Pickering crystal shells. Encapsulation efficiency was dependent on emulsifier type, with the molecular complementary of the nonpolar moiety and the polar head group size together seeming to dictate which emulsifiers were most capable. The emulsifiers studied were glycerol monostearate (GMS), glycerol monopalmitate (GMP), glycerol monooleate (GMO), sorbitan monostearate (SMS), sorbitan tristearate (STS), and polyglycerol polyricinoleate (PGPR). Emulsions were prepared using a rotor-stator and then further shear-crystallized using a rheometer with parallel plate geometry. Analysis of viscosity profiles, thermal behaviour, and resulting polymorphic forms revealed that the addition of emulsified water did not significantly affect crystal polymorphism compared to bulk systems. However, the glycerol-based emulsifiers (GMS, GMP, and GMO) produced smooth-surfaced crystal spheroid shells while the sorbitan-based emulsifiers produced irregularly-shaped shells and droplet cores (SMS) or incomplete crystal shell formation (STS). The propensity for interfacial crystallization was explored using a temperature-controlled tensiometer to observe the onset and progress of crystallization over time.

Effect of Hydrocolloids and Crystal Promoter on the SFC of a Palm Oil Based Fat Reduced W/O Solid Emulsion.
M. Cordova-Barragan and E. Dibildox-Alvarado, Universidad of Autónoma de San Luis Potosí, Mexico.

The effect of the addition of hydrocolloids and crystal promoter on the solid fat content (SFC) of a palm oil based fat reduced water-in-oil (w/o) emulsion was studied. In this line, a palm oil based w/o emulsion (w/o 35/65) was prepared dispersing hydrocolloids [xanthan gum, K-carrageenan, carboxymethyl cellulose (CMC), sodium alginate and citric pectin] in the aqueous or the oil phase adding or not a crystal promoter. After the preparation, the emulsion was crystallized in two water baths at 8 and -4°C, looking for an emulsion temperature of 22 and 12°C, respectively; the emulsion was storaged at 18°C. These emulsions were analyzed after 72 hours by SFC and the results were statistically analyzed. The outcomes obtained reflected that the addition of a crystal promoter and the dispersion of hydrocolloids in the water phase significantly increased ($p<0.05$) the SFC. In this way, citric pectin, K-carrageenan, xanthan gum, CMC and sodium alginate dispersed in water phase increased the SFC of the emulsions added with a crystal promoter in 1.12, 1.09, 1.04, 1.04 and 1.04%, respectively. These results suggest an increase in stability in fat reduced w/o solid emulsions that can be used in the food industry (i.e., margarines).

Algae Butter, a Novel Structuring Lipid, Its Similarities and Differences in Composition, and Observed Functionality When Compared to a Conventional Stearin Butter.
A.G. Marangoni[1], W.G. Rakitsky[2], E. Blum[2], and L. Zou[3], [1]Dept. of Food Science, University of Guelph, Canada, [2]Solazyme Inc., USA, [3]Bunge North America, Inc., USA.

The focus of this research is to evaluate algae butter properties and composition, their differences and similarities when compared with Shea stearin. In particular, their fatty acid profiles, triglyceride structures, melting properties, crystallization kinetics, and crystal structures are evaluated. Also, areas of compositional differences that leads to functionality differences in food products will be highlighted.

In addition, application results of this study revealed that this unique technology can be used in many different food product applications such as confectionery, bakery, etc. However, these functional products confer superior performance without affecting taste, texture, and mouthfeel of the finish products.

Conclusion: The study demonstrates unique novelty of Algae butter as a structuring agent when compared to conventional Shea stearin.

A Materials Science Approach to Understanding the Functionality of Low Saturated Emulsion Systems. P. Smith[1], F. Davoli[2], S. Metin[2], and D. Karleskind[3], [1]Cargill R&D Centre Europe, Belgium, [2]Cargill Inc, USA, [3]Cargill Malt, Belgium.

To provide function in bakery fat systems provide more than triglycerides. Solids are needed to develop structures for products. This is provided by hard saturated fat. Therefore it is not simple to reduce saturated fat. However there is a drive to reduce saturated and total fat in foods. Consequently its structural behavior must be replicated by other components.

Emulsion systems allow the opportunity to create structure at lower amounts of fat and so can be used to replace higher saturated systems. In this work we demonstrate how by understanding the performance of the original and low-saturated systems novel solutions are created that can allow for improved behavior of reduced fat systems.

The work focuses on relating the physical and rheological properties of structured systems and the structure and performance of pastries. This is used to create models that allow prediction of performance with composition. Careful manufacture creating shells around droplets allows control of the behavior and good performance with low saturate systems.

The work demonstrates how shells are built and structured. Effects of composition/ emulsifiers and processing on shells are shown.

The combination of shell building and materials science approach gives capability to design low saturated emulsions for varied applications.

Oil Structuring Using Particle Stabilized Systems. S. Metin[1], P. Smith[2], F. Davoli[1], D. Karleskind[3], and B. Wainwright[4], [1]Cargill Minneapolis R&D Center, Cargill Inc., USA., [2]Cargill R&D Centre Europe, Belgium, [3]Cargill Malt, Belgium, [4]Cargill Dressing, Sauces, & Oils, USA.

There is a drive to reduce the saturated fat in foods to improve nutritional content. Many foods are structured and stabilized by solid fat. Traditionally, solid fat was contributed by trans-fat and by saturated fat. Today, the vast majority of trans-fat has been eliminated from the food supply thereby relegating structuring capacity more or less exclusively to saturates. Consequently, there is widespread interest in developing alternative structuring elements. Many researchers are active in this area, but it is a challenge to commercialize low-saturated fats with desired functionality and cost with label friendly ingredients that is acceptable to consumers.

Our approach starts with understanding the functionality of a particular fat in a high fat food product. This work focused on reduction of saturated fat using label friendly porous edible particles. Processing, characterization and application of particle stabilized fat systems in bakery and confectionery products will be demonstrated.

Low-saturated particle stabilized fat systems were developed by coupling particle stabilization technology with fat crystal optimization. Interactions between the lipid and the particles must be controlled so that the lipid forms a continuous, phase wherein the particles are distributed. The particles act as network builders structuring the lipid phase, providing a structured system.

EAT 2: Dairy Lipids

This session sponsored in part by Land O'Lakes.

Chairs: F. Maleky, Ohio State University, USA; F. Peyronel, University of Guelph, Canada; and R. Gnanasambandam, Land O'Lakes, USA

The Effect of Emulsification and Emulsion Droplet Distance on the Nanoscale Structure of Milk Fat. P.R.R. Ramel, Jr., F. Peyronel, and A.G. Marangoni, University of Guelph, Canada.

Subjecting different forms of milk fat available in the market today, such as anhydrous milk fat/AMF (~99% fat), concentrated milk fat/CMF (~80% fat), and cream or whipping cream (~35 to 42% fat), to cold isobutanol extraction and imaging allowed us to characterize crystalline nanoplatelet size as influenced by state of dispersion. Furthermore, using ultra-small-angle x-ray scattering (USAXS) and interpreting the patterns using the Guinier-Porod and Unified Fit models allowed us to characterize the properties of the CNPs and CNP aggregations present in the different forms of milk fat stated above.

Initial studies showed that anhydrous milk fat is composed of CNPs which have smooth surfaces, with average sizes of 600nm x 100nm to 900nm x 300nm (LxW). Furthermore, results indicated that the CNPs aggregate via reaction limited cluster-cluster aggregation (RCLA) with average sizes of 1.14µm, and at even larger scales, the aggregates showed a diffuse surface.

As milk fat is used as a key ingredient in many products, studying its nanostructure for different macrostructural forms may provide valuable information on its functionality.

The Miracle of Milk Fat. T. Landon, Land O'Lakes, USA.

We often cannot appreciate the forest for studying the intricate complexity of a pine needle or a leaf. I love fresh butter because I love fresh butter—and because I have tasted "good" fresh butter, I will always know "good" fresh butter. The circular nature of this reasoning works in this case. Milk fat, at various concentrations, has been harvested as calories for thousands of years—deservedly earning our respect in the food world and as scientists. Milk fat can be harvested using a variety of methods. The cream that is used as the starting material is derived from milk. The overall quality and source of the milk and cream is vitally important to the finished milk fat/butter final product. What makes milk fat so unique? Its uniqueness. Imagine a fatty system that is designed from its origin to be delivered in an aqueous environment. The mixture of short, medium, and long-chain fatty acids of milk fat provide an array of opportunities in product development, as do the interfacial components that aid in stabilizing the internal fat phase. My hope is for you to hear the story and/or listen now and hear it later.

Role of Dairy Ingredients in Nutritional Products. K. Vasist and N. Rangavajla, Abbott Nutrition, USA.

Dairy Ingredients are the principal source of high-quality proteins and nutrients in nutritional products. Dairy ingredients portfolio, developed over the years, through technical advancements in processing, functionality improvements has resulted in a number of new commercial nutrition products. These nutritional products appeal to consumers and address immediate needs such as convenience, specific disease conditions, maintain and enhance quality of life. Dairy ingredients play an important role in infant foods, both for healthy and premature infants. Elderly and patients in critical care, or recovering from a major surgery, immensely benefit from nutritional products. Nutritional products can be in liquid, powder or other formats, such as bars and are often the sole source of nutrition for patients on tube feed, in renal conditions, fluid restrictions requiring concentrated source of nutrients. Dairy proteins are preferred as they provide the essential amino acids needed for recovery and maintenance of skeletal muscle, such as in sports. Technological advances in dairy processing has brought forth many functional milk fractions including Caseinates, Milk Protein Isolates, Whey Protein Isolates, Milk Protein Hydrolysates, that help in making products with consumer appeal and also provide the life-saving nutrients. The advantages and challenges of using dairy ingredients in some of the applications will be presented.

Microstructure Engineering of Milk Fat by Recombination of Its Fractions. A.G. Marangoni and P.R.R. Ramel, Jr., Dept. of Food Science, University of Guelph, Canada.

The microstructure and crystallization kinetics of binary and ternary mixtures of milk fat fractions during isothermal crystallization at 15 and 20°C were characterized using polarized light microscopy and the Avrami model. Results showed that large differences in crystal structure can be observed by increasing the crystallization temperature (*i.e.*, decreasing supersaturation) from 15 to 20°C. Furthermore, increasing the proportion of high melting fraction (HMF) results in the formation of rod or needle-like crystals (*i.e.*, low values of Avrami index, n). Decreasing HMF and increasing the low melting fraction results in the formation of disc-like or spherical crystals. Creation of this concentration-temperature map for the formation of different milk fat crystal structures allows for targeting desirable microstructures for the optimization of product functionality.

EAT 2.1/IMG 2: Nano-, Micro-, Macrostructure

This session developed in conjunction with the Agricultural Microscopy Division (Imaging Techniques Interest Area).
Chairs: A.G. Marangoni, University of Guelph, Canada; and P.R.R. Ramel, Jr., University of Guelph, Canada

Formation and Microstructures of Whipped Oils Composed of Vegetable Oils and High-melting Fat Crystals. K. Sato, S. Mishima, and S. Ueno, Hiroshima University, Japan.

In food materials, foams have significant advantages of shape retention, soft texture, thermal barrier, and low calorie content. We reports the experimental results of formation processes of whipped oils composed of vegetable oils (salad oil) and high-melting fat crystals of fully hydrogenated rapeseed oil rich in behenic acid (FHR-B). No emulsifier was added to form this whipped oil. Micro-probe FT-IR spectroscopy, synchrotron radiation microbeam X-ray diffraction, polarized optical microscopy, and DSC were employed to observe fine fat crystal particles of the most stable beta polymorph of FHR-B, and their adsorption at the air-oil surfaces before, during, and after the formation of the whipped oil. The following results were obtained. (1) Preparation of organogel composed of salad oil and small fibrous beta fat crystals by special tempering procedure is a prerequisite for forming whipped oil. (2) The beta fat crystals are adsorbed at air-oil surfaces to encapsulate the air bubbles during the formation process of whipped oil. (3) The values of overrun of the whipped oil reached >200% after an aeration time of 30min at 20°C. (4) The microbeam X-ray diffraction experiments demonstrated that the lamellar planes of the beta fat crystals near the air-oil surfaces were arranged almost parallel to the air-oil surface plane.

Acoustic Cavitation and Bubble Dynamics in Edible Oils. P.R. Birkin[1], T. Foley[1], S. Martini*[2], and T. Truscott[2], [1]University of Southampton, UK, [2]Utah State University, USA.

The objective of this research is to detect, quantify, and exploit cavitation events generated by high intensity ultrasound (HIU, 20kHz) in edible lipids. In this study lipids, such as soybean and sunflower oil, were sonicated and the dynamics of the bubbles produced in these systems characterized using high-speed imaging, laser scattering, and a hydrophone. The results from this research show, for the first time, that the cavitation field in these lipids is significantly different from that observed in water. Cavitation clusters were generated in these oils even though the viscosity of this media was significantly higher than water. In addition, a ‘streamer’ of bubbles was also formed during sonication. This dense and opaque streamer appears more axially defined compared to water and was composed of many small bubbles that traveled rapidly vertically away from the tip at velocities greater than 5m s^{-1}. In addition long-lasting bubbles can be observed in the bulk of the oils. Lastly, a significant increase in temperature was observed in the stream of bubbles. The knowledge gained from this research could be exploited to optimize sonication conditions in the processing of lipids in order to change their final physical properties.

Effect of Interfacial Crystallization on the Rheological Behavior and Droplet Localization of a Fat Crystal Network-stabilized Emulsion. R.R. Rafanan and D. Rousseau, Ryerson University, Canada.

Physical properties of water-in-oil (w/o) emulsions, such as tablespreads, depend on the presence of interfacial fat (Pickering, PK) crystals and/or a 3D fat crystal network in the continuous oil phase. How the droplet interface interacts with the surrounding fat crystal network is not widely understood. Investigating these interactions is of great importance to control textural properties of the final product, such as fracture, spreadability, or flow. It is hypothesized that interfacially adsorbed solids will alter viscoelastic properties, namely by reinforcing the network than droplets without adsorbed solids. To test this, fat crystal network emulsions comprised of hydrogenated soy oil were constructed containing 0-20wt% water. Water droplets were stabilized by either monoglycerides (PK) or polyglycerol polyricinoleate (liquid surfactant, LS) at a constant water:surfactant ratio. PK emulsions displayed better storage stability, exhibited higher storage moduli (G') than their LS counterparts and resisted time-dependent shear deformation above $10\%_{w/w}$ water content. Light microscopy showed that LS droplets adsorbed to the fat crystal surface, while PK droplets localized within individual crystals. This indicates that surfactant structure mediates droplets localization within the network which is a contributing factor in viscoelastic behaviour.

Freeze-thaw Stability of O/W Emulsions: Influence of Crystallization Behavior of Fats. C. Ishibashi, H. Hondoh, and S. Ueno, Graduate School of Biosphere Science, Hiroshima University, Japan.

O/W emulsions are highly unstable in freezing, and it is demulsified rapidly after thawing. It is well known that partial coalescence of fat crystals is one of the factors affecting freeze-thaw stability. The morphology, the size, and the amount of fat crystals influence the freeze-thaw stability of O/W emulsion. However, there are few reports which focus on the crystal growth of fats in O/W emulsion during freezing. In this study, we observe the crystallization behavior of O/W emulsion which used rapeseed oil or soybean oil as an oil phase. In addition, the influence of fat crystals on the freeze-thaw stability of O/W emulsion are reported.

The main results are as follows: (i) the soybean oil emulsion showed higher freeze-thaw stability than the rapeseed oil emulsion. (ii) In the rapeseed oil emulsion, partial coalescence was clearly observed during a storage. Finally, the microscopic images were almost covered with fat crystals. (iii) In contrast, in the soybean oil emulsion, fat crystals only appeared at the interface of the oil droplet. From these results, we conclude that the rapeseed oil emulsion were destabilized by partial coalescence, whereas, the soybean oil emulsion were not.

Effect of Oil Type on Fat Crystallization Thermodynamics. N.L. Green[1], G. Marinoni[2], and D. Rousseau[1], [1]Ryerson University, Canada, [2]University of Udine, Italy.

Much of the exploration into fat crystallization has neglected to focus on the effect of oil type in a fat-oil mixture. The fats studied are an industrially-relevant blend with high tristearin content and also pure tristearin. We used a variety of oils, including triacylglycerols with varying degrees of unsaturation, their corresponding unsaturated fatty acids, as well as both pure and blends of aliphatic compounds. Crystallization at both slow and fat cooling rates revealed the relative stability of the mixtures. Concurrent x-ray diffraction revealed the polymorphic form(s) upon crystallization onset as well as their evolution. Further, microscopic observation of the resulting crystals revealed large differences in crystal morphology. Reduced molecular complementarity of fat and oil led to earlier crystallization onset, as well as the formation of lesser polymorphs. We explain our results using solubility parameters that are typically utilized for a polymer-solvent system. These account for a range of interactions (dispersion, polar, and hydrogen bonding) to quantify the complementarity of solute (fat) and solvent (oil).

Structural and Physical Characteristics of Fats Crystallized Under High Pressure. M. Zulkurnain[1], V.M. Balasubramaniam[1,2], and F. Maleky[1], [1]Dept. of Food Science & Technology, Ohio State University, USA, [2]Dept. of Food, Agricultural, & Biological Engineering, Ohio State University, USA.

Different processing conditions have been introduced to modify lipid crystalline network. Limited effort is placed in evaluating effects of high pressure processing. However, application of pressure during crystallization have shown texture improvement. We plan to understand how pressure influences lipid crystallization mechanism and its ultimate functional properties. Binary mixtures of hard fats (5-30%) and oil were crystallized under pressure (0.1-600MPa) using a laboratory scale high pressure kinetic tester with thermally controlled condition. Crystallized sample’s structural and physical properties were characterized using X-ray diffraction, polarized light microscopy, rheometer, differential scanning calorimetry, and analyzed for binding capacity. Samples crystallized at 0.1MPa were used as control. The presence of smaller nano- and microscale of spherical ß crystals were documented in samples crystallized under high pressure. However, when initial temperature fell below sample's crystallization temperature, nucleation prior to compression produced mixture of ß’ and ß spherulitic crystals. These observations propose different mechanisms of lipid crystallization under the influence of quasi-instantaneous volume reduction during compression. Substantial increment in storage modulus and oil binding capacity suggested improvement in plasticity and network stability towards oil migration.

Microviscoity of Liquid Oils in Confined Colloidal Fat Crystal Networks. M.A. Rogers[1] and M.G. Corradini[2], [1]University of Guelph, Canada, [2]Rutgers University, USA.

Molecular rotors may be utilized as non-invasive, non-disruptive, and highly sensitive alternatives to conventional measures of bulk viscosity when the oil is entrained in a colloidal fat crystal network. Oil viscosity changes based on the molecular confinement of the oil, which is dependent on its molecular volume. Changes in micro-viscosity were not dependent on the solids content, but instead were strongly dependent on the box-counting fractal dimension in high-space filling colloidal fat crystal networks (i.e., D>1.89). A bulk oil viscosity is often an overestimation of the actual viscosity of the entrained oil and may not be appropriate when predicting diffusion in multi-phase materials.

Mechanisms of Retardation Effects of Polyglycerine Fatty Acid Esters on Crystallization of Diacylglycerols Examined with Small-angle X-ray Diffraction. K. Saitou[1], R. Homma[1], M. Shimizu[1], K. Yasunaga[1], K. Taguchi[2], S. Ueno[2], and K. Sato[2], [1]Kao Corp., Japan, [2]Hiroshima University, Japan.

Edible oil containing high concentration (>80%) of diacylglycerols (DAG-rich oil) have beneficial healthy effects on obesity and obesity-related diseases. The DAG-rich oil, however, causes precipitation of 1,3-positional isomers of DAGs containing high-melting saturated fatty acid chains at chilled temperatures, which is undesired for real applications. We examined the effects of polyglycerine fatty acid esters (PGFEs) on retardation of the crystallization of the DAG-rich oil by using SFC, DSC, polarized optical microscopy, and X-ray diffraction (XRD) techniques. We found that, prior to the crystallization of high-melting DAG fractions, the cooled DAG-rich oil containing the PGFE additives showed birefringence under polarized light. In addition, small angle XRD patterns revealed the formation of supramolecular assembly composed of DAG and PGFEs, which are quite different from those of DAG crystals. From these results, it can be considered that the retardation of crystallization of DAG-rich oil is caused by the formation of liquid crystal-like supramolecular complex structures in which high-melting fractions of DAGs is incorporated.

Structure-function Relationship of Puff-pastry Shortenings. B.A. Macias-Rodriguez, F. Peyronel, and A.G. Marangoni, University of Guelph, Canada.

Puff-pastry shortenings are specialty fats rich in saturated (SFA) and trans fatty acids (TFA). We studied different commercial systems to identify key parameters underlying their functionality. We characterized the solid structure by ultra small angle (USAXS), small angle (SAXS), and wide angle (WAXS) X-ray scattering. We used small and large deformation rheology to obtain rheological parameters in the linear and non-linear regime. All shortenings have similar polymorphism (ß, b', or both) while the domain sizes are in the range of 300-400Å. USAXS indicate that the aggregation of crystalline nanoparticles (CNPs) for laminating shortenings is either via diffusion limited-cluster aggregation or reaction limited cluster aggregation, while for the multipurpose shortening, CNPs remain un-aggregated. Creep-recovery parameters show lower retarded compliance values and higher zero-shear viscosity for laminating shortenings compared to multipurpose shortening. The viscoelastic moduli (G' and G": 1-3MPa) and yield stresses (350-750Pa) remain unremarkable. Lissajous curves (stress versus strain) and Fourier-transform rheology in the nonlinear region suggest less strain-stiffening behavior in laminating shortenings compared to the multipurpose one. This study provides novel insight on the structural and rheological signatures of laminating fats, and opens up the possibility for the design of healthier shortenings.

EAT 2.2/IOP 2b: Waxes and Phase Change Materials

This session developed in conjunction with the Industrial Oil Products Division.
Chairs: J.F. Toro-Vazquez, Universidad of Autónoma de San Luis Potosí, Mexico; and S.S. Narine, Trent University, Canada

Designing Superior Phase Change Materials from Lipids. M.C. Floros, Trent University, Canada.

Recently, renewable energy such as wind and solar power has gained economic viability for heating and power generation. However, renewable energy is intermittent in nature; the wind does not always blow nor the sun always shine. This necessitates the need for storage solutions for the excess energy during peak times for off peak use. We have prepared a series of renewable diesters from saturated vegetable oils using cheap facile chemistry and have shown that they melt and crystallize sharply (between 39 and 77°C) and possess high enthalpies of melt, making them ideal green phase change materials for thermal energy storage. All of these diesters have enthalpies of melt and crystallization values in excess of 230 J/g – significantly higher than commercial paraffin wax phase change materials currently on the market. The phase change temperatures of the materials can be predictively varied, while maintaining similar latent heat values, as a function of the length of the fatty acid and/or short chain dialcohols. In addition to applications including thermal energy storage for building heating and power generation, this temperature range is also congruent with hot food and beverage consumption temperatures, allowing foods and beverages to be precisely heated or cooled to a desired temperature.

Controlling the Crystallization and Melting Behavior of Self-metathesized Vegetable Oil Waxes. L. Bouzidi and S.S. Narine, Trent Centre for Biomaterials Research, Dept. of Physics & Astronomy/Dept. of Chemistry, Trent University, Canada.

Oligomers of triacylglycerols (TAGs) derived from the self-metathesis of vegetable oils were used to produce functional non-toxic waxes. A significant understanding, not present before, of the structure and waxy functionality of TAG oligomers was achieved. The TAG-oligomers were shown to present an architecture that deliver performance waxes that are tunable with respect to crystallization and melting ranges. The effectiveness of this architecture relates to the balance between amorphous and crystalline phases, which are functions of chain length, van der Waals additive attractive forces and steric hindrances due to symmetry and unsaturation. Although the effect of saturation on the phase behavior was the most dramatic, with differences in crystallization temperature up to 62°C, isomerism and molecular mass were shown to affect crystallization significantly, leading to differences of up to 30°C. The unveiled predictive trends indicate that the thermal parameters can be adjusted in a very broad range by saturation, isomerism, and size, making the development of a large variety of lipid sourced high-value waxes possible.

Biorefinery Technology: New, High-performance Renewable Approaches in Performance Waxes. J.A. Brekan, G. Zopp, and R. Littich, Elevance Renewable Sciences, USA.

The demand for increased sustainability has expanded the use and research activity of synthetic and bio-derived performance waxes. Waxes such as montan and carnuba are highly desired in many applications including plastics additives, personal care, polishes and adhesives. They contain significant amounts of long chain (>C22) fatty esters, and in the case of montan, the natural wax is commonly oxidized to fatty mono and di-acids and esterified with ethylene glycol (EG) to generate a montan ester derivative (Montan E). These long chain structures impart desirable properties such as hardness, high melt point (>70°C) and high thermal stability.

The ERS C18+ fatty acid methyl ester (FAME) stream also provides a mixture of mono- and di-acids (C18-24) and may provide a feedstock to generate derivatives for use in similar applications. This presentation will focus on the ester wax work targeting this biorefinery stream. This presentation will review the development and design of a number of new, synthetic waxes obtained through either direct functionalization or through esterification reactions of these biorefinery outputs.

Ethylcellulose Oleogels in Cream Cheese. R. Nicholson, A.G. Marangoni, and S. Barbut, University of Guelph, Canada.

A direct reduction of milk fat in cheese is detrimental to various qualities desired by consumers. Recently, new strategies have evolved to gel vegetable oils with ethylcellulose (EC) in combination with fatty acid/fatty alcohol mixtures. Substituting these oleogels for milk fat reduces levels of saturated fat while maintaining the textural properties of the full-fat variety. Mechanical tests have been used to evaluate the effects of incorporating EC oleogels into low-fat cream cheese. Certain formulations of canola oil with an EC and fatty acid/fatty alcohol combination produced samples comparable to those containing identical proportions of added milk fat. Back extrusion results showed hardness values of approximately 18N for samples made with these oleogels. Only slightly less than the 20N value obtained from samples containing milk fat, but significantly greater than the 7N value produced with canola oil. Additionally, cream cheese products containing EC and fatty acid/fatty alcohol oleogels were similar in appearance to those with only milk fat, as demonstrated by lightness (L*) values of 93.89 and 95.35 respectively. This work has the potential to produce low-saturated fat cream cheese alternatives without compromising the appearance or textural attributes and should help expand the use of EC in dairy systems.

Low Saturated Functional Fat Systems Structured by MAGs and Waxes. F. Davoli[1], S. Metin[1], D. Karleskind[2], and P. Smith[3], [1]Cargill Inc., USA, [2]Cargill Malt, Belgium, [3]Cargill R&D Centre Europe, Belgium.

The reduction of saturated fat while keeping the functional performance of traditional shortenings has been a challenge faced by industry. In general, low saturated fat systems lack performance mainly because of the weak structure. Vegetable waxes and monoglycerides have been studied as structuring agents for oleogels. They are also known for their properties as fat crystallization nucleators. This study also showed their properties as crystal habit modifiers. This study aimed to develop robust fat systems targeting low saturated fat content. Structuring, nucleation and habit modifier properties of monoglycerides and waxes were studied in a fat system containing canola oil and palm stearin. The structures of the experimental compositions were compared to commercial low saturated shortening by analysing rheology, thermal profile and microstructure. The experimental fat systems studied were submitted to different processing conditions using a pilot-scale scraped surface heat exchanger (SSHE).A combination of controlled crystallization process and composition resulted in promising results so that the obtained fat structures were comparable or even (in some cases) showed improvements to the performance of commercial shortenings.

EAT 3: Effect of Structure on Lipid Functionality

Chairs: P. Smith, Cargill R&D Centre Europe, Belgium; and G. Yang, Kellogg, USA

Application of Enzymatically Structured Symmetric Triacylglycerol Molecules as Cocoa Butter Equivalent and Human Milk Fat Analog. K.T. Lee ***(Timothy H. Mounts Award Winner)***, Chungnam National University, Republic of Korea.

Ranges of palmitic, stearic, oleic, and linoleic acids in total fatty acid compositions of cocoa butter (CB) are 21.7–27.0%, 30.6–39.2%, and 29.4–35.4%, respectively. According to positional distribution, oleic acid dominated at the sn-2 position (85–89%), whereas the palmitic and stearic acid are 1.3–2.9% and 1.5–3.0% at sn-2 position, respectively. These total and sn-2 fatty acid compositions led to simple triacylglycerol species, which are sn-1,3-dipalmitoyl-2-oleoylglycerol (POP), sn-1,(3)-palmitoyl-stereoyl-2-oleoylglycerol (POSt), and sn-1,3-distereoyl-2-oleoylglycerol (StOSt). These TAG molecules provide unique characteristics to chocolate. Also, sn-1,3-dioleoyl-2-palmitoylglycerol (OPO) and sn-1,(3)-oleoyl-linoleoyl-2-palmitoylglycerol (OPL) are unique TAG molecule in human milk fat. All of them are symmetric, of which TAG molecules of saturated-unsaturated-saturated or unsaturated-saturated-unsaturated fatty acids are composed. Such structures afford the special properties of nutritional and physicochemical aspects. In this session, commercialized process patented by Korean company (CJ Jeiljedang Co.) will be discussed, and some results of new possible process scheme for production of OPO, for which tripalmition (PPP)-rich fat is excluded, will be briefly presented. Also, using pH-stat digestion and a simulated *in vitro* digestion model, the digestion degree evaluation of symmetric TAG molecules will be presented.

Raman Analysis of the Liquid-solid Phase Transition of Stearic Acid-based Lipids. D. Rousseau, E. Serre, O. Dubova, E. Da Silva, and N.L. Green, Ryerson University, Canada.

The conformation of four stearic acid-based lipids (tristearin, distearin, monostearin, and stearic acid) during their liquid-solid phase transition was explored with Raman spectroscopy and x-ray diffraction. Raman bands were broader in the liquid vs. solid state which we ascribed to less distinct interactions between the key functional groups explored (C-H, O-C=O, -OH, C-C, etc.). In the 3100 -2700cm^{-1} region, two strong bands at 2881cm^{-1} and 2845cm^{-1} characteristic of antisymmetric and symmetric methylene C-H stretching vibrations showed large differences between the four lipids. The 1700-1800cm^{-1} region, which contains information on the geometry of the ester region (O-C=O) also showed distinct trends between the four lipids. In the 1500 -1350cm^{-1} region, corresponding to C-H deformation vibration, presence of C-H_3 asymmetric bending vibrations in the solid state suggested that methyl group interactions were absent in the liquid state. Intensity of the 1420cm^{-1} band was greatest for monostearin and lowest for tristearin, where the alcohol functional group is absent. The 1000-1200cm^{-1} region characteristic of C-C skeletal stretching vibrations also produced broader bands in the liquid-state, with fully extended (all trans) chains (and thus similar conformations) in the crystal structure of the lipids. These lipids showed distinct Raman spectra related to their different molecular structures.

Effect of Cooling and Shear Rates on Physicochemical Properties of Binary Fat Blends Based on Shea Stearin. S. Danthine[1], S. Delatte[1], K.W. Smith[2], K. Bhaggan[3], and C. Blecker[1], [1]University of Liège (GxABT), Belgium, [2]Fat Science Consulting Ltd., UK, [3]Loders Croklaan B.V., The Netherlands.

Filling fats are used in bakery and confectionery applications. Fillings functionalities are influenced by their fat composition but also by the processing conditions applied for crystallization. Indeed, consistency and stability of fat samples are not only influenced by the amount of solid fat they contain, but also by the microstructure of the fat crystal network, which is highly dependent on the process. Processing parameters such as shear rate and crystallization temperature can have a marked impact on the physical properties of filling fats.

In this study, the effects of processing conditions (cooling rate, shear rate and crystallization temperature) on physical properties of binary blends, all based on shea stearin as hard fat (SOS-source) were investigated by means of p-NMR, powder XRD and texture analysis.

It was found that the firmness was higher for samples crystallized at slow cooling rate. However it decreased with the increase of the agitation rate and the final agitation temperature. All the selected binary blends exhibited different crystallization mechanisms according to the triacylglycerol composition of the liquid phase and their complementarity with triacylglycerol of the solid phase, and this both under static and dynamic conditions.

Preparation of Cocoa Butter Equivalent via Lipase-catalyzed Esterification of 2-monoolein and a Mixture of Fatty Acid Ethyl Esters. H.R. Park[1,2], N.K. Choi[1,2], and I.H. Kim[1,2], [1]Dept. of Food & Nutrition, Korea University, Republic of Korea, [2]Dept. of Public Health Science, Graduate School, Korea University, Republic of Korea.

Cocoa butter equivalent (CBE) high in POS was prepared by lipase-catalyzed esterification of 2-monoolein and a mixture of fatty acid ethyl esters (of palmitic and stearic). Two different lipases namely, Novozym 435 from *Candida antartctica* and Lipozyme RM IM from *Rhizomucor miehei* were employed in this study. Firstly, 2-monoolein was prepared from high oleic sunflower oil (HOSO) in a recirculating packed bed reactor via Novozyme 435-catalyzed ethanolysis. The optimal conditions for 2-monoolein were 1:77 of molar ratio (HOSO to ethanol), 25°C of temperature, and 3min of residence time, respectively. Subsequently, CBE high in POS was synthesized via Lipozyme RM IM-catalyzed esterification with 2-monoolein and a mixture of fatty acid ethyl esters under vacuum. Optimal molar ratio (2-monoolein to a mixture of fatty acid ethyl esters), temperature, enzyme load, and vacuum were 1:4, 50°C, 10%, and 5mmHg, respectively. Under these conditions, up to 91.3% of TAG was formed, composed of POS (39.0%), POP (20.0%), and SOS (17.7%).

Structured Lipids and Specialty Fats: A Novel Class of Products. V. Gibon, Desmet Ballestra Group, Belgium.

Most of edible oils have limited applications in their original form. Various modification processes can be applied to extend their use and tailor novel classes of lipids for specific food or nutraceutical applications. Structured lipids are triacylglycerols modified either by changing the position of existing fatty acids or by incorporating other fatty acids in order to yield new fats with desired chemical, physical or nutritional properties; specialty fats are formulated ingredients required by the food industry for specific applications. Physical, chemical or enzymatic processes like fractionation, interesterification, acidolysis, alcoholysis, or glycerolysis are capable to adjust the required specifications. The most relevant modified lipids are Oleic/Palmitic/Oleic (OPO) fats for infant formula, cocoa butter equivalents (CBE), and cocoa butter substitutes (CBS) for confectionery formulations, anti-blooming (BOB) for chocolate applications, hard stearins as substitutes of fully hydrogenated fats, low/zero trans fats for margarine and shortening applications, Medium/Long/Medium (MLM) oils for malabsorption problems, Long/Short-Long/Medium fats as low calorie source, partial acylglycerols as emulsifiers or functional cooking food, high omega-3 concentrates for the treatment of cardiovascular and inflammatory diseases, and MediumChainTriacylglycerol (MCT) oils with dietary and medical relevance.

Physical and Chemical Characterization of Interesterified Brazilian Pequi Oil. A.M.M. Guedes[1], R. Antoniassi[1], M.C. Galdeano[1], R. Grimaldi[2], M.G. de Carvalho[3], and A.E. Wilhelm[1], [1]Embrapa Food Technology, Brazil, [2]School of Food Engineering, University of Campinas (UNICAMP), Brazil, [3]Federal Rural University of Rio de Janeiro (UFRRJ), Brazil.

From a nutritional perspective, pequi fruit (*Caryocar brasiliense*, Camb) is considered important since its pulp is rich in oil and carotenoids encouraging industrial production for small community use. Pequi pulp oil triacylglycerols (TAGs) contain mainly oleic (~57%) and palmitic (~37%) fatty acids, distributed mainly among OOO, POP/PPO and POO TAGs. The oil has a tendency to fractionate upon storage and has a relatively low melting temperature (SFC of 5% at 25°C). Here we modified pequi oil through chemical interesterification, which increased the PPP content to ~6%. This caused a flattening in the SFC-temperature profile, effectively raising the melting temperature significantly (SFC of 5% at 42°C). The interesterified oil does not fractionate and is thermally stable up to 40°C, with an SFC-temperature profile which resembles that of puff pastry shortening despite containing high amounts of oleic acid. TAG composition, fatty acid composition and distribution, melting profiles by SFC and DSC, polymorphs by powder X-ray diffraction, microstructure by polarized light microscopy, and carotenoid content were determined for both non-interesterifed and interesterified oils. Moreover, the dramatic increase in stability and functionality of the interesterifeid oil was not accompanied by a significant decrease in total carotenoid content.

Reduction of Oil Migration Using High Intensity Ultrasound. Z. Cooper, M. Kimball, and S. Martini, Utah State University, USA.

Interesterified soybean oil was crystallized at 32, 30, 28, and 26°C with and without high intensity ultrasound (20kHz). Samples were placed in 1cm diameter tubes and stored at 5°C for 48h. After storage, a 1.3cm height cylinder was cut from the cylinder and placed in a filter paper (diameter 12.5cm) at 5 and 25°C for 24h. Oil migration was measured as the diameter of the oil diffusing out of the crystalline matrix into the filter paper. Oil migration in samples stored at 25°C occurred very fast and the diameter of diffused oil exceeded the diameter of the filter paper after 24h. Oil migration occurred more slowly in samples stored at 5°C. Results show that samples crystallized in the presence of sonication and stored at 5°C for oil migration measurements had a significantly lower oil migration compared to the non-sonicated ones. For example, samples crystallized at 32 and 30°C had diameters of 6.6±0.2 and 8.9±0.6 for samples crystallized without sonication compared to 1.9±0.1 and 1.8±0.1 for samples crystallized in the presence of sonication. This delay in oil migration can be attributed to differences in the characteristics of the crystalline network formed. Harder and more elastic crystalline networks are obtained in the presence of sonication with significantly smaller crystals.

Saturated Specialty Diglycerides as Oil Structuring Agents for the Replacement of Partially Hydrogenated Oils with Reduced Total Saturated Fat Content. J.B. Botts, J. Robertson, and M.E. Walsh, Corbion, USA.

When added to a liquid oil, saturated specialty diglycerides promote the formation of a fine crystalline solid network capable of entraining the liquid oil within a gel-like matrix. The resulting system possesses similar mechanical, physicochemical and organoleptic properties as traditional partially hydrogenated oils allowing for the successful replacement of the *trans*-fat products. Traditional solid fat content data is not a successful predictor of the mechanical and organoleptic properties of these new oleogel systems. Oscillatory rheology proves to be a valuable tool for understanding the viscoelastic properties of these systems. Shortenings prepared using the specialty diglyceride technology have superior handling and sensory attributes with improved nutritional claims as compared to palm-based alternatives. Trans-fat free shortening systems prepared using this technology have proven application success in a variety of bakery applications such as donuts, pie crusts, cookies, and croissants.

EAT 3.1: Confectionary Fats

Chairs: D.A. Kim, Mondelēz, USA; and J. Komaiko, University of Massachusetts Amherst, USA

Chocolate Microstructure: From Bean to Bar. K. Dewettinck, Lab. of Food Technology & Engineering, Ghent University, Belgium.

Chocolate can be considered as a dispersion of several kind of particles such as sugar, cocoa, and dried milk in a continuous matrix of cocoa butter. Such a microstructure determines several quality attributes related to handling, storage, shelf life and sensory, and a better understanding of it is the key towards redesigning production processes and reformulating the product itself with respect to its caloric value, sugar and fat content. The creation of the chocolate microstructure starting from the most important raw material i.e. cocoa beans will be discussed from bean to bar and beyond such as the production of the world-famous Belgian pralines.

Study the Mechanism of Diffusion in Different Cocoa Butter System Using Magnetic Resonance Imaging. H. Wang and F. Maleky, Ohio State University, USA.

In a multi-component chocolate product, oil migration, from high oil content filling into chocolate, is one of the major contributors to quality loss. To model and study the effect of chemical composition and processing conditions on oil migration, five different cocoa butter samples were used. Samples were crystalized in the presence and absence of shearing (static and sheared), and were placed in contact with a cream as a source of liquid oil. Using Magnetic Resonance Imaging, the movement of liquid oil into samples was investigated. Sample oil diffusivity was analyzed based on Fickian diffusion model. A short-time approximation to Fick's second law was used to investigate the contribution of diffusion-controlled and relaxation-controlled mechanisms to the migration process. Among all the samples, the highest effective diffusion coefficient was observed in the one of higher unsaturated fatty acids and phospholipids. This trend was identical in static and sheared samples, while shearing can delay oil migration. We have also observed a more homogeneous network made of smaller particles under shearing. This study successfully highlights that even minor differences in cocoa butter composition can affect oil migration rate. Although shearing decreased cocoa butter oil diffusivity, the effects of chemical composition is still dominant.

Physical Properties of Dark Chocolate Made of Cocoa Butter/SSO-fat/OSO-fat Mixtures Forming Molecular Compound Crystals. S. Watanabe[1], F. Yokomizo[1], and K. Sato[2], [1]Oil & Fat Development Dept., Fuji Oil Co. Ltd., Japan, [2]Hiroshima University, Japan.

We formulated dark chocolate using ternary fat mixtures of cocoa butter (CB)/SSO-fat/OSO-fat, cacao mass, sugar particles, and other ingredients (lecithin and vanillin). SSO-fat (*rac*-SSO:64%, *rac*-PSO:11%, OSO:6% and others) and OSO-fat (OSO:72%, *rac*-OSL:10 % and others) were high-melting and low-melting fractions prepared by hydrogenation-esterification-fractionation of canola oil. Although the crystal structures and crystallization behavior of CB and CB/SSO-fat/OSO-fat are largely different, miscible mixtures of CB)/SSO-fat/OSO-fat of the stable b(V) polymorph having the melting points around 33°C were formed. We confirmed that physical properties of melting, SFC, XRD, and crystal morphology of the fat mixtures at selected concentration ratios of (CB)/SSO-fat/OSO-fat are suitable for confectionery fats. Hardness and occurrence of fat bloom of dark chocolate were examined by changing relative ratios of SSO-fat and OSO-fat at constant CB concentration (50%). Hardness increased with increasing SSO-fat concentration, whereas fat bloom was retarded with increasing OSO-fat concentration.

Overall, the present study has shown that the fat mixtures of CB/SSO-fat/OSO-fat can be employed for a non-tempering CBE fat, which is rich in oleic acid and does not contain *trans*-fats.

Phase Behavior of Cocoa Butter-alternative Fats Mixtures. N.I. Murillo-Hernández[1], S. Martini[2], and E. Dibildox-Alvarado[1], [1]Universidad of Autónoma de San Luis Potosí, Mexico, [2]Dept. of Nutrition, Dietetics, & Food Science, Utah State University, USA.

In the chocolate manufacturing according to the current legislations concerning its main components a maximum amount of 5% of the other vegetable fats could be used. In Mexico, the chocolate industry produces chocolate type products, utilizing the cocoa butter's alternative fats (CBA), which share properties and characteristics with the cocoa butter. This work aimed at studying phase behavior of mixtures of cocoa butter with vegetable fats. These mixtures were formed by cocoa butter and 5, 10, 20, 30, 40, 50, 60, 70, 80 and 90% (w/w) of cocoa butter equivalent extender (CBEE) or improved (CBEI), and by cocoa butter with cocoa butter substitutes (CBS). The phase behavior of mixtures was analyzed using differential scanning calorimetry and nuclear magnetic resonance by determining the solid fat content (SFC). On the basis of the results, the solid fat content of the mixtures with CBS was found higher respected CBEE and CBEI. The solid fat content was explained by the composition of triacilglieridos present in mixtures. The melting temperature profile exhibited eutectic points in mixtures of cocoa butter with 50% CBS, 30% of CBEM and 60% of CBEE. In this line, the presence of vegetable fats could influence the crystallization properties and the resistance to surface fat separation in chocolate type products. This procedure constitutes a way of qualitatively judging the compatibility of fats.

Enzymatic Interesterification and Dractionation of Palm Mid Fraction and Algal Shea Sterain to Synthesize Cocoa Butter Equivalent. S. Mirzaee Ghazani[1], W.G. Rakitsky[2], and A.G. Marangoni*[1], [1]University of Guelph, Canada, [2]Solazyme Inc., USA.

Fat blends were formulated based on the fatty acid composition and triacylglycerol (TAG) profile of cocoa butter. Different ratios (w/w) of palm mid fraction (PMF) and algal shea stearin were subjected to enzymatic interesterification to synthesize cocoa butter equivalent by selecting Lipozyme TL IM as catalyst for enzymatic esterification reaction.

The optimum reaction time, temperature, water content and molar ratio of algal shea stearin and PMF were determined to optimise the highest yield of POS, SOS and POP triacylglycerols. Solvent fractionation was a necessary step to remove mono- and diacylglycerols and high melting TAGs. The following parameters, before and after enzymatic interesterification reactions were determined to show engineered TAGs mimic similar physical properties of cocoa butter: fatty acid and triacylglycerol compositions, melting point, crystallization temperature, solid fat content, and XRD analysis.

Processing and Compositional Effects on the Physical Chemistry of Palm-based Confectionery Models.
R. West and D. Rousseau, Ryerson University, Canada.

Palm oil is considered to be a suitable alternative to partially hydrogenated vegetables oils for fat-based confections because of its natural semi-solid consistency and zero-trans fat content. However, this oil has slow crystallization behaviour and is prone to hardening during storage which can develop undesirable rheological and textural properties. While studies exist on palm oil characterization in bulk systems, the impact of sugar during storage has yet to be reported. Crystallization and rheology of two palm oils of commercial origin in both bulk and novel confectionery models are explored in this study over four weeks of storage. The higher saturated and diacylglyceride contents of one palm oil yielded significantly higher solid fat content and viscoelastic properties as the oils were stored. Interestingly, while sugar reduced solid fat content in the confectionery systems, their viscoelastic properties were substantially higher than the bulk systems, suggesting crystallization to be heavily driven by network formation rather than crystal formation. The findings generated from this study establish a foundation for palm-based confections, allowing for optimization and improvement in the product development and innovation of such applications.

EAT 4/S&D 4.2: Delivery and Dispersed Systems

This session developed in conjunction with the Surfactants and Detergents Division.
Chairs: S. Ghosh, University of Saskatchewan, Canada; and E. Szekeres, Method Products, Inc., USA

Engineering Interface of Encapsulation Systems to Limit Oxidation and Controlling Release of Bioactives. N. Nitin[1], Y. Pan[1], and R.V. Tikekar[2], [1]University of California, Davis, USA, [2]Dept. of Nutrition & Food Science, University of Maryland, USA.

Limiting oxidation and controlling the release of encapsulated compounds are the key goals for the rational design of encapsulation systems. This presentation will focus on our efforts to characterize the role of interfacial composition and its engineering in influencing oxidation processes during accelerated testing and the release of bioactives during simulated digestion. Specifically, the role of interfacial thickness and composition, layer by layer coatings and localization of antioxidants at the interface in influencing the oxidative barrier properties of the encapsulation systems will be discussed. The results will demonstrate the role of interfacial design in reducing permeation of radicals and oxidation of a model encapsulated bioactive in encapsulation systems. To advance understanding of the role of engineered structures in influencing the delivery and bioavailability of bioactives, development of a real time measurement approach to characterize interactions of encapsulation interface with digestive environment as a function of interfacial composition and structure will be presented. The results will illustrate a novel approach to analyze the dynamics of nanoscale structures during digestion processes and their relationship with release and delivery of bioactives.

Forming Essential Oil-loaded Hollow Solid Lipid Micro- and Nanospheres with Antimicrobial Properties Using Supercritical Fluid Technology. J. Yang, C.R. Kok, R. Hutkins, and O.N. Ciftci*, Dept. of Food Science & Technology, University of Nebraska-Lincoln, USA.

The main objective was to form essential oil-loaded novel hollow solid lipid micro- and nanospheres with antimicrobial properties using a simple and green method based on supercritical fluid technology. Hollow solid lipid micro- and nanospheres were formed from fully hydrogenated soybean oil using atomization of the carbon dioxide (CO_2)-expanded lipid. Particle formation conditions of 50µm nozzle diameter, 60°C, and 200bar CO_2 pressure yielded hollow solid lipid spheres ($d_{50\%}$ = 278nm). Shell thickness of the hollow spheres decreased with increasing pressure. Decreasing the nozzle diameter yielded the polymorphism of the particles from β to α. Clear dispersions of essential oil-loaded nanoparticles in water (d_{mean}= 173nm, polydispersity index (PDI)= 0.3) was formed by separating the microparticles in water by filtration. Hollow spheres were loaded with peppermint essential oil spontaneously during the particle formation with loading efficiencies up to 74%. Essential oil-loaded particles showed antimicrobial activity against selected microorganisms. Release properties and the morphology of the particles studied during storage showed that supercritical CO_2-assisted atomization process is a promising method to form hollow solid lipid micro- and nanospheres to develop “natural” antimicrobials for food systems.

The Effect of Contact Force on Coalescence of Water Droplets Suspended in Bitumen. S. Goel[1], S. Ng[2], and A. Ramachandran[1], [1]University of Toronto, Canada, [2]Syncrude Canada Ltd., Canada.

In the oil-sands industry, the presence of water in bitumen is highly undesirable in downstream unit operations. Therefore, processes such as centrifugation and inclined settling are currently used to achieve the separation of water from bitumen froth. They are, however, ineffective in removing extremely fine water droplets, which can form a significant fraction of the residual water content in bitumen after separation. The aim of this project is to understand the practically-relevant parameter regimes which will facilitate the coalescence of these extremely fine droplets with themselves or with larger drops. Accordingly, this will lead to the generation of larger droplets which can be further removed by conventional separation methods. Currently, the project is focused on conducting coalescence experiments in microfluidic device to determine the contact forces, bitumen solution properties and water chemistry that lead to coalescence of water droplets suspended in bitumen. In our experiments, coalescence time decreases with an increase in contact force and pH. Additionally, coalescence time increases with increasing bitumen concentration. Moreover, these experiments have also been quantified to develop coalescence time vs. contact force curves for different bitumen to solvent ratios and pH. Our results show that coalescence process is relatively faster beyond a critical force.

An In-depth Look at the Stabilization Factors Behind Low to Very Low Fat Spreads. K. Bhattacharya and P.G. Kirkeby, DuPont Nutrition Biosciences Aps, Denmark.

Retail or table margarines with about 82% fat content have become less popular with consumers globally. They are replaced with lower fat spreads with fat content ranging from 60% to as low as 15% and are being sold under the banners of ‘low fat’ or ‘low calorie’ products. However stability of such low fat water-in-oil emulsions is often a problem for producers affecting shelf life and consumer acceptance due to imperfection in taste and texture. There is also a growing preference of ambient stable low fat spreads from sustainability view point by the producers and retailers which require more careful selection of emulsifiers, fat blends and overall composition. Stability of different low fat systems depends on the synergy between the choice of fat blends, choice of emulsifiers and hydrocolloids and their interaction with other food additives. Processing conditions also play a very important role. The present work discusses in details the role of different distilled monoglycerides, structuring emulsifiers, alginates and pectins for emulsion stabilization of fat spreads having 60% to 15% fat in conjunction with varying processing parameters. Analytical data includes Solid Fat Content and water droplet size distribution by p-NMR, confocal laser imaging, rheology, texture analyses, and sensory evaluations.

Studies on Preparation of Margarine from Di–acyl Glycerol Rich Oleogel. M. Ghosh[1], D.K Bhattacharyya[1], N.R. Bandyopadhya[1], and M. Ghosh*[2], [1]Indian Inst. of Engineering Science & Technology, IIEST, India, [2]University of Calcutta, India.

Di-acyl glycerol (DAG) rich oils are gaining importance in the field of nutrition due to their suppressive effect on postprandial serum triglyceride elevation and body fat accumulation. Recently, novel kinds of products are being developed in the name of oleogel using liquid oils and appropriate food grade oleogelators. Objective of the present study was to prepare oleogels obtained from DAG rich oil and to prepare margarine from these oleogels. DAG rich rice bran, soybean, sunflower and palmolein oil and combination of palm stearin and cetyl oleate were used as gelators to prepare oleogels. For preparation of margarine, lecithin and monoglycerides were used as emulsifier along with 14% water and 1% NaCl. The products thus formed, had the desired textural and thermal properties along with suitable shelflife. All the margarine samples were smooth spread, homogeneous in texture. The samples did not lose their structural integrity after the application of load on to the gels. The peak melting temperatures of the margarine samples varied from 47°C to 54°C. The dilatation value of all the margarine samples decreased with increase in temperature. These oleogel products were very stable against oxidation during storage and thus proved their suitability as shortening or margarine-base stock.

Fully Water-dilutable Microemulsions for Delivery of Riboflavin. N. Garti, N. Lidich, and A. Aserin, Hebrew University, Israel.

Riboflavin (vitamin B2) is a naturally occurring micronutrient found in relatively high levels in, or added to, various foods and beverages. It plays an important role in biochemical redox reactions in humans and animals. It also acts as an antioxidant and is essential for the health of skin, hair, eyes, and liver.

Microemulsions (MEs) are isotropic and thermodynamically stable nanosized mixtures of water, oil, and amphiphiles.

In the present study, a ribophlavin phosphate (RFP)-loaded MEs were prepared and structurally characterized. The selected ME components self-assemble, forming transparent, thermodynamically stable, non-viscous and fully water-dilutable structured systems. RFP incorporation does not disorder the ME structure. We found that in formulations with up to 40wt% water, the hydrophilic surfactant headgroups and cosolvent strongly bind water molecules (DSC and SD-NMR). Above 60wt% water, globular, O/W nanodroplets, ~14nm in diameter, are formed (SAXS, cryo-TEM, and SD-NMR). The structure of MEs loaded with 0.14 to 4.25wt% RFP (0.29–8.89mmol per 100g formulation) is not significantly influenced by the presence of the RFP.

Microfluidics to Study Emulsifier Adsorption and Emulsion Stability. K. Muijlwijk, C.C. Berton-Carabin, and C.G.P.H. Schroën, Wageningen University, The Netherlands.

To understand droplet formation and stabilization, technologies are needed that combine micrometer length and millisecond time scales. Microfluidics are used to bridge this gap, and the present work shows that droplet formation and stability in food emulsions can be investigated in close detail through high speed recording and image analysis. The dynamic interfacial tension during droplet formation was measured in the millisecond range with a microfluidic Y-junction that was purpose built and validated extensively. This method allows exploration of surfactant behaviour at the oil-water interface, which is not possible through any other technique at this very short time scale. Emulsion stability under flow was measured with a microfluidic coalescence channel. Results include coalescence measurements of protein stabilised emulsion droplets and we show that emulsions are stable at low protein concentrations.

We discuss underlying principles of these microfluidic methods and their potential for the food industry, which we believe to be very versatile, since the flow conditions during production and storage can both be covered adequately. Through this, we give evidence that microfluidic investigations can add greatly to the knowledge needed for the rational design of large scale emulsion production.

EAT 4.1/IMG 3: Length Scales and Lipids

This session developed in conjunction with the Agricultural Microscopy Division (Imaging Techniques Interest Area).
Chairs: K.B. Koch, North Dakota State University, USA; and C. Rogers-Kelly, Mississippi State Chemical Lab, USA

Influence of Maillard Conjugation on the Stability of Emulsion-based Delivery Systems: Lutein-enriched Corn Oil Emulsions at Different pH and Temperature Conditions.
C.E. Gumus[1] ***(Hans Kaunitz Award Winner)***, G. Davidov-Pardo[1,2], and D.J. McClements[1,3], [1]Dept. of Food Science, University of Massachusetts Amherst, USA, [2]Dept. of Human Nutrition & Food Science, California State Polytechnic University, USA, [3]Dept. of Biochemistry, King Abdulaziz University, Saudi Arabia.

Lutein may be utilized in foods as a natural pigment to replace synthetic colorants, or as a nutraceutical ingredient to improve eye health. However, due to its poor water-solubility and chemical instability the incorporation into foods needs further research. In this study, we evaluated the effect of storage temperature and pH on the physical and chemical stability of lutein-enriched emulsion-based delivery systems prepared using caseinate and Maillard conjugates as the emulsifier. Also, we compared the fate of the emulsions stabilized by casein alone versus casein-dextran conjugates in the gastrointestinal tract as well as the lutein bioaccessibility. The emulsions stabilized with both emulsifiers remained physically stable at all temperatures (5-70°C); however the rate of chemical degradation of lutein increased with increasing temperature (E_a = 38kJ/mol). The samples stabilized with protein alone irreversibly aggregated at pH 4 and 5; however, the samples at the same pH values stayed stable when the protein was replaced by protein-polysaccharide conjugates. The emulsion stabilized by casein alone was not stable in the gastric phase of the digestion, whereas the samples made with Maillard conjugates were still stable after the same step. The emulsifier type did not influence the lutein bioaccessibility.

Surfactant-free Solid Lipid Nanoparticles Prepared with Novel Synthetic Ultra-long Chain Fatty Acyl Based Amphiphilic Lipids.
W. Wei[1,2] ***(Honored Student Award Winner)***, F. Feng[2], B.C. Pérez[1], M. Dong[1], H. Mu[3], X. Xu[1], and Z. Guo[1], [1]Aarhus University, Denmark, [2]Zhejiang University, China, [3]University of Copenhagen, Denmark.

Recently, scientific and technological interest in nanocarrier system arises from the possibility of making surfactant-free system. However, most of the studies reported are focusing on surfactant-free emulsions which are liquid. The surfactant-free “solid” lipid nanoparticles (SF-SLN) are of great interest yet rarely reported. A major challenge is developing structurally simple amphiphilic lipids that are solid state at physiological temperature. In our recent study, we have design and synthesized an array of amphiphilic lipids based of behenic acid (22:0) as ultra-long acyl hydrophobic tail with varied hydrophilic heads *via* enzymatic approach. The synthetic compounds were examined as novel excipients for developing SF-SLN. Results revealed that the headgroup has a dramatic influence on the forming of the SF-SLN. Small size and narrow distribution SF-SLNs were made with some of the synthetic compounds. The SF-SLNs show high loading efficiency of lipophilic model drug, Fenofibrate and long term *in vitro* drug release. Moreover, the structures of the particles were investigated using Atomic force microscopy and Transmission electron microscopy which revealed that SF-SLNs were vesicles with drug incorporate into the lipid bilayer. The SF-SLNs is a safe and versatile system for drug and active delivery, which are suitable for different administration routes.

Shear-induced Aggregate Creation or Destruction in Edible Oils: Models and Computer Simulation.
B. Townsend[1], N. Callaghan-Patrachar[2], F. Peyronel[1], K. Ramadurai[3], A.G. Marangoni[1], and D.A. Pink*[2,1], [1]Dept. of Food Science, University of Guelph, Canada, [2]Physics Dept., St. Francis Xavier University, Canada, [3]Dept. of Mathematics, College of the North Atlantic, Canada.

Edible Oils are Complex Fluids possessing many components some of which are in a non-liquid state. Flowing Edible Oils generally experience shear: In processing and consumption a non-shear flow is generally not realized due to the presence of surfaces with which the fluid interacts. In order to understand experimental data it is advantageous to possess models which predict observable effects. These models must correctly represent both the system of interest and the flow. Modeling the flow involves the Navier-Stokes equation(s) while the boundary conditions involve defining fluid flow at surfaces which themselves might undergo change as the flow proceeds. We shall present simple models of aggregates in order to identify aspects relevant to Edible Oil flow. We used Dissipative Particle Dynamics to model a sheared system and computed structure functions, S(q), to interpret the results, approaches not hitherto used. We found that a critical shear gradient exists which distinguishes between shear which destroys aggregates and shear which accelerates aggregation compared to aggregation under static conditions. This critical value is system-dependent. We shall exhibit examples for isotropic (spheres) and anisotropic (cylinders) objects, and compare them to USAXS measurements on sheared and non-sheared samples.

Organogels of Comprised of a Cyclic Peptide from Flaxseed Oil.
M.A. Rogers[1] and M.J.T. Reaney[2], [1]University of Guelph, Canada, [2]University of Saskatchewan, Canada.

To the best of our knowledge, this is the first report on an orbitide, capable of self-assembling from 0D objects to 1D nano-fibers and resulting in 3D molecular gel networks. LOB3 (a.k.a. cyclolinopeptide A), extracted from *Linum* *usitatissimum* L. (flaxseed), forms molecular gels in acetonitrile. Molecular gels, comprised of cyclic peptides, allow for much more complex amino acid sequences than have been currently reported. It appears that cyclization to form orbitides imparts conformational aspects to the molecule that drives self-organization into fibrillar objects with very large aspect ratios. These nanoscale fibers, ~300nm in diameter and >100mm in length, seem to aggregate into bundles of fibers that reach micron dimensions. Within the nano-fibers, the orbitides adapt an antiparallel b-sheet-like conformation with very high periodicity, as illustrated by NMR and XRD giving rise to the very high aspect ratio fibers.

Which Length Scales are Affected in Sheared Edible Fat Systems? F. Peyronel[1], D.A. Pink[2,1], and A.G. Marangoni[1], [1]University of Guelph, Canada, [2]St. Francis Xavier University, Canada.

The food industry must eliminate trans fats and reduce saturated fats in food products. By understanding how solid fat structures respond to shear, one can seek alternatives with similar characteristics.

We combined ultra small angle X-ray scattering (USAXS) with wide and small angle scattering (WAXS, SAXS) to investigate structures on length scales from angstroms to ~6 micrometres. Static and sheared systems containing up to 20% SSS in OOO, 20% FHSO in HOSO and 20% SSS in OOO + cotton seed oil, were studied employing cooling rates of 30 and 0.5deg/min. Theoretical prediction indicate that structures arising from crystalline nanoplatelet (CNP) aggregation can formed: (1) cylinders (TAGwoods) with fractal dimension D_m ~1, (2) DLCA structures (D_m ~1.7-1.8); (3) RLCA structures (D_m ~2.0-2.1); (4) aggregates with D_m ~2.2-3, all of which were confirmed by USAXS. Sheared fats exhibited CNP morphologies that were similar to those of static systems but with different average sizes: (1) 70% larger for the sheared systems and (2) 40% larger for samples using a 0.5°C/min ramp. Sheared samples did not form fractal structures on length scales from ~0.8µm to ~3µm as indicated by an absent of a constant slope in the double logarithmic plot of scattering intensity vs scattering vector.

CLA-rich Chocolate Bar and Chocolate Paste Production and Characterization. S.E. Mayfield[1], D. Van de Walle[2], C. Delbaere[2], S.E. Shinn[1], A. Proctor[1], K. Dewettinck[2], and A.R. Patel[2], [1]University of Arkansas, USA, [2]University of Ghent, Belgium.

Conjugated linoleic acid (CLA) is an 18-carbon fatty acid with multiple health benefits, including anti-obesity and anti-carcinogenic properties. CLA-rich soy oil (CLARSO) can be produced through a heterogeneous catalysis process, and this oil was previously used to produce CLA-rich margarines and shortenings. The objectives of this study were to produce CLA-rich chocolate bars and pastes by replacing a portion of the fat with CLARSO and compare the rheological, textural, and thermal properties of these pastes/bars to controls made with either soy oil or traditional fats. CLARSO was used to prepare bars/pastes. Rheology, firmness, and thermal behavior of the pastes and fracturability, hardness, and thermal behavior of the bars were determined. The CLARSO chocolate pastes/bars contained no additional saturated fat relative to soy oil controls but the pastes had more solid-like rheology and were firmer and the bars had a higher fracture force relative to soy oil controls. Relative to non-soy controls, CLARSO pastes had similar rheology and CLARSO bars had similar fracturability, despite containing less saturated fat. The fat crystals of all samples were in the same polymorphic form. Therefore, it was successfully demonstrated that CLARSO has the ability to produce chocolate pastes/bars with similar physical properties as traditional products containing more saturated fat.

Physical Properties of Shea Butter and Its Blends with Cocoa Butter. M.L. Herrera[1] and R.J. Candal[2], [1]Inst. de Tecnologia en Polimeros y Nanotecnologia, University of Buenos Aires, National Research Council of Argentina, Argentina, [2]Inst. de Investigacion e Ingenieria Ambiental, University of San Martín, Argentina.

Shea butter is an off-white or ivory-colored fat extracted from the nut of Shea tree. Shea butter is known for its use in cosmetic industry and as it is an edible fat is also used in food formulation. Shea butter is solid and a good alternative to replace *trans*-fat in food or use as cocoa butter alternative. The objective of this study was to investigate the physical properties of Shea butter and its blends with cocoa butter. Blends with different amounts of cocoa butter, from 10 to 90% were prepared. Thermal behavior and melting points were studied by DSC. Fatty acid and TAG compositions were analyzed by capillary GC. Polymorphism was described by X-ray diffraction. Compatibility with cocoa butter was analyzed from iso-solid diagrams built from NMR experiments. Thermal behavior and polymorphism of Shea butter were similar to the ones of cocoa butter. Up to 20% Shea butter may be added to cocoa butter without changing SFC values. However, iso-solid diagrams showed eutectic formation above this percentage. Shea butter may be used as cocoa butter extender or in applications were solid fats are required**.**

Holistic Control of Fish Oils Based on NMR Spectroscopy. B.W.K. Diehl, E. Zailer*, and Y.B. Monakhova, Spectral Service AG, Germany.

The assessment of quality of fish oils is traditionally performed based on several separate analytical methods, which were developed and standardized several decades ago being very time-consuming and sometimes even erroneous.

A proton nuclear magnetic resonance (^{1}H NMR) spectroscopic method was developed and validated for targeted control of oil quality parameters (e.g., peroxide value, anisidine value, acidic value, iodine value, hydroxyl value, and fatty acid composition) and tocopherols (a, b, g, d) within short time and in only one analytical run.

Additional information about glyceride composition and free fatty acids can be obtained using ^{13}C NMR spectroscopy. Furthermore, DHA, EPA, and total Omega-3 content are quantitative determined.

Furthermore, multivariate models (principal component analysis and classification methods) based on the NMR distribution of major and minor components were developed for type screening of fish, especially of salmon oils. An important point is the distinction between natural and synthesized fish oil in general.

Thus, NMR spectroscopy combining with targeted and non-targeted approaches is a versatile technique, which can be applied for the quality and authenticity control of fish oils. The developed NMR method can replace multiple tedious conventional techniques for routine fish oil analysis.

Extraction and Characterization of Montmorency Sour Cherry (*Prunus cerasus* L.) Pit Oil. N. Korlesky[1], L.J. Stolp*[1], D.R. Kodali[1], W.C. Byrdwell[2], and R.J. Goldschmidt[2], [1]University of Minnesota, USA, [2]USDA, ARS, USA.

Montmorency sour cherry (*Prunus cerasus* L.) pit oil (CPO) was extracted and characterized by various methods including: GC, LC-MS, NMR, TGA, DSC, and XRD. The cherry kernels were 22.4% of the weight of the pits and contained 30.9% oil. The oil had an acid value of 1.45mg KOH/g, saponification value of 193mg KOH/g and unsaponifiable matter content of 0.72%. The total tocopherols and sterols were 525 and 3766ppm respectively. The major fatty acids of CPO were oleic (47.6%) and linoleic acids (34.5%) along with smaller concentrations of saturates (11.6%) and a nutritionally important fatty acid, a-eleostearic acid (El, 5.6%). The CPO contained six major triacyglycerols (TAG), OOO (16.91%), OLO (16.72%), LLO (13.27%), PLO (7.29%), OOP (6.52%), and LElL (6.19%) plus a number of other minor TAGs. The TAGs containing at least one saturated fatty acid constitute 32% of the total. The polymorphic behavior of CPO as studied by DSC and XRD confirmed the presence of a, ß' and ß crystal forms. The CPO has moderate oxidative stability and high thermal stability with oxidative induction time of 30.3 minutes at 130°C and a thermal decomposition temperature of 352°C.

EAT 5/H&N 5.1: Satiety and Sensory

This session developed in conjunction with the Health and Nutrition Division.
This session sponsored in part by Nestlé and Young Living Essential Oils.
Chairs: S. Martini, Utah State University, USA; and F. Dionisi, Nestlé, Switzerland

The Taste of Fat and Its Role in Dietary Fat Intake.
T.A. Gilbertson, Utah State University, USA.

It has been well over a decade since our laboratory first identified the ability of free fatty acids to activate mammalian taste receptors cells, consistent with there being a "taste of fat". Since that time, the ability of fatty acids to act as the proximate stimuli for fat taste has been validated in a number of species spanning the molecular, cellular and behavioral levels. We have recently identified several novel fatty acid-activated G protein-coupled receptors that, in conjunction with the fatty acid binding protein CD36, allow the recognition of chemically distinct classes of free fatty acids. Interestingly, this pathway also plays an important role in the control of dietary fat intake. Mice with genetic deletions in signaling proteins in this pathway show a reduced preference for dietary fat and concomitantly gain less weight and put on less body fat than wild type mice. Further, there are pronounced sex differences in this pathway and its role in fat intake suggesting that the 'fat taste worlds' of males and females are markedly different. This presentation will summarize what is known about the receptors and transduction pathway for free fatty acids and its contribution to dietary fat preference.

Gut-brain Endocannabinoid Signaling: Fatty Acid Sensing and Beyond. N.V. DiPatrizio, Div. of Biomedical Sciences, School of Medicine, University of California, Riverside, USA.

The endocannabinoid (eCB) system is an important regulator of feeding, energy balance, and reward. We reported that tasting dietary fats – but not carbohydrate or protein – initiated production of the eCB, 2-AG, in the rat small intestine, and this signaling event at local CB_1Rs drives the intake of fatty foods. Importantly, surgical disruption of the vagus nerve – which communicates neurotransmission between the brain and gut – blocked increases in intestinal eCB levels after fat exposure, suggesting that cholinergics are required for orexigenic eCB activity in the gut. We recently extended these findings and reported that similarly to tasting fats, fasting for 24 h drives production of intestinal 2-AG. This effect occurred in a time-dependent manner that paralleled increases in the 2-AG precursor, SAG. Importantly, vagotomy blocked fasting-induced rises in jejunal 2-AG, an effect that was mimicked by inhibiting muscarinic acetylcholine receptors (mAchRs) in the small intestine, and reduced refeeding after a fast. Similarly, administration of a peripherally-restricted CB_1R antagonist inhibited refeeding after a fast. This result suggests that vagal activity at intestinal mAchRs drives production of 2-AG, which in turn, functions as a general hunger signal. Thus, these investigations advance our understanding of gut-brain eCB signaling and suggest potential new treatment options for appetite control.

Small Intestinal Sensing of Lipid in Humans—Relationship with Appetite and Energy Intake. C. Feinle-Bisse, Discipline of Medicine & NHMRC Centre of Clinical Research Excellence in Nutritional Physiology, Interventions, & Outcomes, University of Adelaide, Australia.

Small intestinal receptors play a key role in sensing the arrival of nutrients in the intestinal lumen, initiating feedback loops that lead to adjustments in the rate of gastric emptying and the release of gut hormones, both of which are involved in the regulation of energy intake. Lipid has potent effects on these functions, requiring fat digestion and fatty acids with a chain length of =12 carbon atoms. There is evidence from studies in animals and humans that these GI functions can adapt to both dietary restriction and excess nutrient exposure, modifying the sensitivity to nutrients, particularly, with potential implications for the regulation of energy intake. For example, consumption of a high-fat, high-energy diet accelerates gastric emptying and small intestinal transit of a fat-containing meal. Since obese individuals have an increased energy/nutrient intake, it is conceivable that they may have a reduced ability to sense nutrients, both in the oral cavity and the lumen of the GI tract, associated with reduced modulation of gut functions, thus, compromising the capacity to limit their energy intake. In support, our recent studies demonstrate that habitual fat and energy intake and BMI are inversely related to the ability to taste fat in the oral cavity in healthy humans. Furthermore, obese individuals have reduced pyloric contractile and plasma CCK responses to intraduodenal oleic acid infusion, compared with lean individuals, and the oral and small intestinal responses to fat are correlated. Finally, recent evidence suggests that both oral and small intestinal fat sensitivity can be modulated by dietary interventions, ie enhanced in response to a low-energy diet and reduced in response to a high-fat diet. For example, both acute (for 4 days) and longer-term (for 12 weeks) dietary restriction markedly enhances the sensitivity to the gastrointestinal (GI) and appetite-suppressant effects of fat. Taken together, while fat has potent effects on those GI functions that contribute to energy intake regulation, these effects are diminished by a high-fat, high-energy diet, but can be reinstated, at least in part, by dietary restriction. Much further research is required to investigate the mechanisms underlying the effects of fat on energy intake regulation to determine whether these findings can be translated into efficient, novel approaches to the prevention and management of obesity.

The Taste of Non-esterified Fatty Acids in Humans.
R. Mattes, Purdue University, USA.

Accumulating evidence indicates humans can detect non-esterified fatty acids (NEFA) through the sense of taste as well as other sensory systems. Support for a taste component stems from finding that: A) there is an adequate stimulus concentration in the oral cavity when fatty foods are masticated; B) fatty acid receptors have been identified on taste cells and taste thresholds reflect their functionality; C) oral signals of fat detection are conveyed centrally by gustatory nerves; D) central decoding of gustatory signals from oral fat exposure are not based on somatosensory cues; E) there is a unique quality percept for NEFA; and F) oral fat exposure alters lipid metabolism. The taste quality of NEFA is generally rated as unpleasant when sampled in simple systems, but the possibility that it enhances overall flavor acceptability at low concentrations remains to be examined. Improved understanding of the sensory detection of NEFA holds implications for the food industry, clinical practice, public health, and sensory science.

Sensory Determinants of Fat (and Oil) Intake—The Consumer Perspective. J.X. Guinard, University of California, Davis, USA.

Our consumer research model considers product, consumer, and context variables as determinants of consumer behavior. Humans may not be as sensitive to fats and oils as they are to most gustatory, olfactory or trigeminal stimuli, as measured by difference thresholds, and in simple emulsions and complex matrices. Sensory-specific satiety, the short term food intake regulation mechanism by which humans satiate to specific sensory attributes, is not as relevant to the regulation of fat intake as it is to that of nutrients with taste and smell properties. The main means of sensory perception of fats and oils are by touch and kinesthesia as well as olfaction of aromas associated with fats and oils more than direct chemoreception and transduction. Consistently, sensory preferences for fats and oils are primarily learned, based on exposure (as for olfactory stimuli) rather than innate (as for taste stimuli and chemical irritants). It follows that a segment of the population actually prefers olive oils with some degrees of rancidity and fustiness, likely based on sustained exposure to oils with such qualities. Our Healthy Flavors Research Initiative shows how beef can be substituted with healthier and flavor-boosting (umami) mushrooms in taco blends without loss of consumer acceptance, and how olive oil can be used in place of butter in equally successful culinary strategies for healthier eating.

Complexity of Structure-sensory Relations: Science and Application. G.A. van Aken[1,2], [1]NIZO Food Research, The Netherlands, [2]Insight FOOD Inside, The Netherlands.

Being the portal of the gastrointestinal tract, the mouth is functional for testing the safety and expected nutritional value, pleasure, and possible toxicity of the food, and to prepare the food to form a slippery, smooth bolus that can be swallowed safely. During this oral processing, the food is broken up into pieces and mixed with saliva, during which changes occur in food particle size, rheological properties, and adhesion to the oral surfaces, and tastants and volatiles are released. The perception of texture and flavor is intricately connected to the way the food behaves during oral processing and the way this elicits responses by tactile, taste, and aroma receptors. These responses function to adjust oral processing optimizing the release and composition of saliva and to adjust mastication time and intensity. The time of oral exposure to sensory stimuli and the masticational effort before the food can be swallowed has furthermore been shown to strongly affect the desire to eat more or to stop eating. Moreover, already starting during consumption, the food enters the gastrointestinal tract, where its volume, consistency, and restructuring in the stomach and the release of absorbable nutrients by digestion and absorption signals the brain about the nutritional properties of the food, reflected in sensations of fullness and hunger reduction.

As a consequence, perception, liking, satiety, and the desire for a follow up meal generally depends on the dynamic interaction between the food and the body, and as a consequence can usually not be related directly to food composition, structure, and rheological properties before consumption.

This presentation will focus on the perception of the dynamic food structures in the mouth and specifically the role fat in this. It will be outlined how this can be approached experimentally and how a correct understanding of the way a food behaves during oral processing can be used in practice to adjust the sensory quality.

EAT-P: Edible Applications Technology Poster Session

Chair: M.A. Rogers, University of Guelph, Canada

1. **Addition of Pure Monoacylglycerols to Pure Triacylglycerols in Different Proportions: Effects on Crystallization Properties.** R.C. Silva, J.M. Maruyama, F.A.S.M. Soares, N.R. Dagostinho, Y.A. Silva, J.N.R. Ract, and L.A. Gioielli, São Paulo University, Brazil.

In most foods, the isolated crystallization of triacylglycerols (TAGs) is considered the most important event, although crystallization of these minor lipids (MLs) has a strong influence on the quality of a wide variety of products. The objective of this study was to investigate the effects of adding pure monoacylglycerols (MAGs) to pure TAGs on the crystallization properties in a fat system. Differential scanning calorimetry and polarized light microscopy methods were used for the analysis. Different MAGs (monoolein - O, monopalmitin – P, and monolaurin - L) were added at 1, 3 and 5% to each TAG (triolein - OOO, tripalmitin – PPP, and tristearin - SSS). DSC results showed that the addition of MAGs changed the crystallization of the TAGs (PPP, SSS and OOO). The same MAG may have different behaviors (induction or retardation of crystallization) depending on the proportion added. The incorporation of MAGs to OOO favored the crystallization process at all proportions for all MAGs, although crystallization was most effectively promoted with the addition of 3% of each MAG. Based on the results, the addition of 5% MAG to saturated TAGs delayed the crystallization process, and that the optimum proportion of added MAGs for promoting crystallization was 3%.

2. **Synthesis and Characterization of Non-hydrogenated, Trans Free Cocoa Butter Substitutes by Chemical Interesterification of Palm Oil Fractions.** V.R.R. Yettella and B. Eapen, AAK USA, Inc., USA.

The synthesis of non-hydrogenated and *trans* free cocoa butter substitutes that provide similar mouth feel, flavor release and crystallization behavior as their hydrogenated counterparts is not easy. Removal of hydrogenated fat components from a fat system without compromising on product and process functionalities pose significant technological challenges due to the contribution of partial hydrogenation to the formation of a solid crystal network. In this study, a non-hydrogenated and no-*trans* cocoa butter substitute was synthesized by chemical interesterification of palm oil fractions and the impact of emulsifiers to promote product functionality and sensory characteristics was also investigated. The developed fat system is non-hydrogenated, *trans* fat free, and complies with the new US Food and Drug Administration (US FDA) regulation on partially hydrogenated oils. Results show that the developed fat system exhibits excellent crystallization properties and good bloom stability.

3. **Conjugated Linoleic Acid (CLA) Rich Eggs: Dried for Convenience and Evaluated for Use in Breakfast Sandwiches.** S.E. Shinn, A. Proctor, N.B. Anthony, J.I. Baum, and J.O. Lay, University of Arkansas, USA.

The objective of this study was to develop a commercially viable CLA-rich dried egg product that would be used to prepare scrambled egg sandwiches that would deliver the daily amount of CLA per serving needed to produce clinical effects. Recently, successful production of conjugated linoleic acid (CLA)-rich eggs has been accomplished by adding CLA-rich soy oil to chicken feed, Subsequently, CLA-rich mayonnaise was prepared from these eggs in combination with CLA-rich soy oil. Drying CLA-rich eggs has the potential to increase the products shelf-life and transportability as well as improve ease of use and nutrition. Developing a production and processing protocol, determining shelf stability and functional food properties are necessary for the commercialization of these egg products to be readily available. Furthermore, scrambled eggs and egg sandwiches are commonly consumed in Arkansas and the US, and using these foods as a vehicle to deliver CLA should be readily received by consumers. Daily consumption of these products has the potential to reduce serum cholesterol, markers of diabetes and potentially reduce obesity; e.g. reduce body fat and have a significant impact on human health.

4. **Functional Elaidic Containing Trigylcerides: Synthesis, Physical Properties, and Solution Behavior.** G.R. List[1], R.O. Adlof[1], and A.G. Marangoni[2], [1]USDA/Retired, USA, [2]University of Guelph, Canada.

A previous report from our laboratory showed that heavily hydrogenated soybean margarine and shortening basestocks (IV 65-80) are composed mainly of elaidic and stearic acid trigylcerides (EEE, ESE ,ESS). The object of this study was to synthesize a number of elaidic and stearic acid trigylcerides, determine their physical properties , and solution behavior in soybean oil. The mixtures behave as ideal solutions since plots of 1/Tdeg K vs log concentration are linear. Enthalpies of fusion were calculated from the slopes of the melting point lones and the Hildebrand solubility equation and compared to DSC data. In general the data were in good agreement. In addition the data were in agreement with published data from Timms (1978) who derived enhalpies of fusion from the equivalent carbon numbers of the trigylcerides. Many attempts have been made to classify the functional properties of fats and oils including grouping by melting points into four groups contributing to functionality ranging from -13 to 73°C. Group 4 consist of those obtained by completehydrogenation of vegetable oils, and do not occur to any great extent in unhydrogenated oils. However, the elaidic/stearic trigylcerides are important members of the Group 4 and their physical/functional properties have received little attention.

5. **Crystallization Behavior of Neutralized and Bleached Shea Butter Under Dynamic Conditions.** V. Gibon[1], J. Maes[1], P. Dijckmans[1], C. Blecker[2], and S. Danthine[2], [1]Desmet Ballestra Group, Belgium, [2]Gembloux Agro-Bio Tech, Belgium.

Shea butter and fractions receive today increased interest for food and cosmetic applications due to their unique composition; one example is the use of shea butter stearin as ingredient for CBE.

In this context, neutralized shea butter was first bleached at pilot scale and its crystallization behavior in a stirred pilot crystallizer was monitored by rheology, microscopy, p-NMR, DSC, GC, and HPLC.

Optimal isothermal conditions were selected to overcome unpredictable crystal initiation and excessive viscosity of the slurry. Surprisingly, many small but well-shaped crystals were observed in the early stages with minor impact on SFC and dynamic viscosity of the slurry. Later, a second population of larger crystals appeared inducing slurry SFC and viscosity increases. The slurry was vacuum filtered at regular intervals. Thermal properties and triacylglycerol composition of the oleins were similar to the feedstock until crystallization of the second population. Moreover, the karitene was depleted in all the oleins in favor of the stearins, which is not desired to maintain the melting profile required for food applications. Attempts to remove karitene by crystallization have been tried but only half of the total content could be removed. Moreover, this depletion led to a new system instability during crystallization.

6. **Microstructure and Polymorphism of Enzyme-catalyzed Interesterified Blends of Rice Bran Oil and Fully Hydrogenated Rice Bran Oil.** N. Callejas[1], N. Estefan[2], L. Suescun[2], and I. Jachmanián[1], [1]Grupo de Derivados de la Industria Alimentaria (CYTAL), Uruguay, [2]Cryssmat/Lab (DETEMA), Uruguay.

The Interesterification of oils blended with fully hydrogenated oils has been gaining interest for the design of cero-trans fats to be used as food ingredients. Obviously, microstructure and polymorphism of these products determines their applicability to different type of edible products.

In this work, fully hydrogenated rice bran oil (FHRBO) was blended with rice bran oil (RBO) and interesterified using Lipozyme TL-IM as catalyst. Crystallization properties of both, blends and products, were studied by X-ray diffraction (XRD) and polarized light microscopy (PLM).

While XRD patterns of blends showed peaks corresponding to both ß and ß' polymorphs, products showed only peaks corresponding to the ß' form.

PLM analysis showed too significant changes in crystalline microstructure after interesterification. While blends showed highly compact needle-shaped crystals forming “sea urchin” like structures, products showed regular spherulites containing tiny needles, less compacted and with larger empty space.

Results showed that interesterification produced drastic changes in the crystal habit of these blends, enhancing the prevalence of ß' polymorph in the products, which is the most suitable for shortening or margarine products.

7. **Non Hydrogenated Structuring Fat for Confectionery Fillings and Chocolate Spreads.** K. Bhaggan and H. Manson, IOI Loders Croklaan BV, The Netherlands.

While chocolates are hard and crisp when we bite into it, products like confectionery fillings and chocolate spreads are softer, creamier and more luxurious. When we use and consume these at temperatures around 20°C, the fillings and spreads will be soft and creamy. When the temperature begins to rise, these attributes start changing due to fat phase separation resulting in an oily layer on the top of the product and in case of chocolate spreads recrystallization of cocoa butter resulting in fat bloom.

Hydrogenated, high melting fats, are used to overcome these problems, but still it is very difficult to produce chocolate spreads and/or fillings that will withstand the kinds of temperatures found in warmer climates, especially those where the ambient temperatures are often above 30°C.

In this study, we have investigated different fats on their structuring/oil binding properties and developed a non hydrogenated fat that:

(a) impart temperature tolerance to chocolate spreads by preventing oil exudation at higher temperatures, and,

(b) prevent the recrystallization of cocoa butter in both soft chocolate fillings and chocolate spreads held at lower ambient temperatures (e.g. 20°C).The fat phase used in these application was further characterized by means of Differential Scanning Calorimeter (DSC), powder XRD, and polarized light microscopy.

8. **Binary Phase Behavior of Tripalmitin and 1,3-dipalmitoyl-2-stearoyl-sn-glycerol.** K. Bhaggan[1], K.W. Smith[3], C. Blecker[2], and S. Danthine[2], [1]IOI LOders Croklaan BV, The Netherlands, [2]ULg, GxABT, Belgium, [3]Fat Science Consulting Ltd., UK.

Formulation of most lipid-based products requires the use of solid fats to provide the necessary functionality, desirable texture as well as good stability. The conventional approach for structuring oils relies on the use of high melting triacylglycerols as the building blocks.

The crystallization and subsequent crystal network formation of these high melting crystalline triacylglycerols creates a structural framework that can physically trap liquid oil resulting in the desired texture and stability. The most functional of these high-melting triacylglycerols have been produced by fully hydrogenating oils such as palm oil or other soft oils, resulting in a complex mixture of saturated triacylglycerols.

In this study, the binary phase behavior of tripalmitin

(PPP) and 1,3-dipalmitoyl-2-stearoyl-sn-glycerol (PStP), mainly present in hydrogenated palm oil, was studied by means of Differential Scanning Calorimeter and powder XRD.

It was found that the PPP-PStP mixtures displayed three general types of phase developments:

(a) An alpha to beta-prime to beta phase development via recrystallization from the melt for mixtures having PStP content of up to 20%,

(b) An alpha to beta-prime phase development via recrystallization from the melt for mixtures having PStP content from 30% to 60%,

(c) An alpha to beta-prime phase development via solid-solid phase transition for mixtures having PStP content from 70% to 90%.

9. **Effect of Cream Ripening and Churning Conditions on Physical Properties of Butter.** J. Lee and S. Martini, Utah State University, USA.

The effects of ripening and churning at 5, 10, and 15°C with low and high agitation (LA, HA) applied to ripening conditions on heavy cream (40% fat) were evaluated. Cream's solid fat content (SFC) over time and droplet size distribution after crystallization were measured. Butter obtained by churning the ripened creams for 14.5min at the same temperature as the ripening was characterized by its melting behavior, firmness, and water content. In cream, higher SFC was observed at 5°C and HA while smaller fat droplet sizes were observed with HA regardless of temperature. Creams crystallized at 15°C with LA and HA and crystallized at 5°C with LA did not make butter. All butters showed two melting peaks at approximately 17°C and 33°C. The enthalpy of the first peak of butter churned at 5°C with HA was significantly higher than the others, but this trend was lost after its 24 hr of storage at 5°C.The enthalpies of the second peaks were not significantly different. The cream ripening and churning conditions had no impact on water content of butter or the amount of buttermilk generated. Less spreadable butters were obtained when creams were ripened at lower temperatures regardless of agitation levels.

10. **Analysis of Solubility Behavior of High Melting Wax in Low Melting Oil Using Hildebrand Equation.** S. Jana, N. Chiew, and S. Martini, Dept. of Nutrition, Dietetics, & Food Sciences, Utah State University, USA.

The solubility behavior of six different waxes such as beeswax, candelilla, paraffin, rice bran, sunflower, and yellow carnauba wax was studied in six oils including canola, corn, olive, safflower, soybean, and sunflower oils at concentrations of 0.1, 0.25, 0.5, 1.0, 2.5, 5.0, and 10.0% (% wt. basis). The Hildebrand equation was used to establish if the wax/oil combinations behaved as ideal solutions over the range of concentrations tested. Results showed that all wax/oil combinations behaved as ideal solutions with the exception of sunflower wax in canola, sunflower, and safflower oils. However, sunflower wax in canola and sunflower oils behave as ideal solutions at concentrations above 0.25% and at concentrations above 0.5% when crystallized in safflower oils. Crystal morphology was mainly affected by wax type. Beeswax formed needle and spherulite shaped crystals while candelilla wax crystals were granular shaped. Yellow carnauba wax was characterized by spherulite-shaped crystals while needle or rod-shaped crystals were observed in sunflower wax and rice bran wax. Paraffin wax crystallized forming a cloud of crystals. Wax crystals morphology might be affected by the solubility of the waxes in the oil.

11. **A Prediction Tool for Accelerated and Non-accelerated Fat Bloom in Compound Chocolate Bars Using Image Analysis, Triglyceride Profile, and FAME Content.** N. Quezada, E. Marcillo, L. Macias, and J. Cajape, La Fabril, Ecuador.

Fat bloom is still an issue for some chocolate producers due to the lack of knowledge when replacing high-trans fats for fractionated and/or fully hydrogenated lauric fats. The objective of this study was to develop a prediction tool for accelerated and non-accelerated fat bloom in six compound chocolate bar formulations.

Palm kernel stearin and fully hydrogenated palm kernel stearin were used in six formulations of compound chocolate bars. Each fat was used in three formulations with cocoa powder, cocoa powder plus milk powder and cocoa powder plus maltodextrin for sugar reduction. A set of bars was placed in a chamber under two temperatures cycles (16 and 26°C). A second set of bars was kept in a chamber at 20°C. Fat bloom in the bars was monitored using a capture image system and the whiteness index was determined. All the experiments were conducted in duplicate. Using the compound chocolate bar formulations, triglyceride and FAME content, and the whiteness index, a simulation tool was developed to predict fat bloom under accelerated and non-accelerated conditions.

The compound chocolate bar formulation with more resistance to fat bloom was the one prepared with fully hydrogenated palm kernel stearin, cocoa powder and maltodextrin.

12. **Encapsulation of Lipid Soluble Food Components with Organogelation.** K. Sislioglu[1], I. Karabulut[1], and D.J. McClements[2], [1]Inonu University, Turkey, [2]Dept. of Food Science, University of Massachusetts Amherst, USA.

Most of bioactive components; such as carotenoids, fenolic compounds, oil soluble vitamins, flavours, and antimicrobials are lipophilic components. Lipid carrier systems are encapsulation methods preferred for their ability to improve bioavailability of lipophilic components. Nano-structured lipid carriers are the recently developed delivery systems offering enhanced loading capacity, long term stability during storage.

Solid and liquid lipid blends are main ingredients of the system. However solid lipids cause unhealthy affect, it is suggesting to use gelators in order to obtain organogel particles forming three dimensional network.

Production technique of both organogelation and encaptulation with nano-structured lipid carrier includes same procedures. Dissolving the gelator and bioactive component in an oil at high temperature and then cooling to room temperature are steps to be followed. Using high pressure homogenization or sonification provides submicron sized gelled lipid particles before cooling.

13. **Holistic Control of Edible Oils Based on NMR Spectroscopy.** E. Zailer, B.W.K. Diehl, and Y.B. Monakhova, Spectral Service AG, Germany.

The assessment of quality and authenticity of edible oils is traditionally performed based on several separate analytical methods. These methods were developed and standardized several decades ago undergoing only minor changes since then, which makes oil analysis time-consuming and sometimes even erroneous.

A proton nuclear magnetic resonance (^{1}H NMR) spectroscopic method was developed and validated for targeted control of necessary quality parameters (e.g., peroxide, anisidine, acidic and iodine value, fatty acid composition) within short time and in only one analytical run. Additional information about glyceride composition and free fatty acids can be obtained using ^{13}C NMR spectroscopy. The developed method was successfully applied for several edible oil types. Furthermore, multivariate models (principal component analysis and classification methods) based on NMR distribution of major and minor components were developed for screening of type, age, adulteration, and origin of edible oils. Partial least squares regression was applied to correlate NMR profilings with sensory characteristics detected by qualified panels for different types of edible oils.

Thus, NMR spectroscopy combining with targeted and non-targeted approaches is a versatile technique, which can be applied for quality and authenticity control of edible oils. NMR method can replace multiple tedious conventional techniques for routine oil analysis.

14. **Oil Content and Fatty Acid Composition of French Fries of Brazil.** A.M.M. Guedes[1], A.F. Faria-Machado[1], A.E. Wilhelm[1], B.C.M.T. Pinheiro[2], A.C. Oliveira[2], J.P. Menezes[3], and R. Antoniassi[1], [1]Embrapa Food Technology, Brazil, [2]Federal University of Rio de Janeiro, Brazil, [3]Federal Rural University of Rio de Janeiro, Brazil.

Although not limited by law, Brazilian industry and regulatory agencies came to a consensus on the progressive reduction on the content of trans fatty acids (TFA) based on the controversy on its negative health effects. French fries are popular among youngsters and the assessment of its total fat and TFA content is, therefore, relevant. Four brands of pre-fried frozen French fries sold in the Brazilian market and four brands of French fries from the main fast food chains in Brazil were evaluated on their fatty acid profile and total fat content according to AOAC Official Method 996.06. The frozen pre-fried potatoes showed total fat content of 3.5-7.4g/100g and total TFA content of 0.01-0.03g/100g. The fatty acid profile showed that palm oil, high linoleic sunflower oil, and high oleic sunflower oil were used for pre-frying the potatoes. For the 4 brands of fast-food chain French fries, the total fat content varied from 14 to 21g/100g, with a total TFA content of 0.01-0.14g/100 and palm oil, cottonseed and soybean oil were used for frying. In summary, the results showed that no hydrogenated fat was used for frying potatoes.

15. **Cocoa Butter Equivalent Prepared from Enzymatic Interesterification of Fractionated Palm Oils.** N. Biswas[1], Y.L. Cheow[1], C.P. Tan[2], and L.F. Siow[1], [1]School of Science, Monash University Malaysia, Malaysia, [2]Dept. of Food Technology, Universiti Putra Malaysia, Malaysia.

This study was to produce cocoa butter equivalents (CBE) from enzymatic interesterification of cocoa butter substitutes (CBSs) with commercial stearic and oleic fatty acids. Two CBSs (CBS-A, CBS-B) consisting of different proportions of palm mid fraction:palm kernel oil:palm stearin that show comparable physicochemical properties to that of cocoa butter (CB) were selected. Fatty acids, triacylglycerol constituents, melting profiles and solid fat contents (SFC) were determined using gas chromatography (GC), high performance liquid chromatography (HPLC), differential scanning colorimeter (DSC) and pulsed nuclear magnetic resonance (p-NMR) respectively. Eight samples were prepared with different ratios of CBSs, stearic and oleic acids. Sample CBS-B2 (CBS 80:stearic 15:oleic 5) was found to have fatty acids composition close to CB. Interesterification of the sample for 6hr at 60°C using 4% (w/w) enzyme resulted in melting endotherm at 33.5°C, similar to that of CB. The sample showed three primary triacylglycerols (palmitoyl 17.7%, oleoyl 28.4% and stearoyl 19.5%), and ~48% SFC at 20°C and 0% SFC at body temperature, which are close to those of CB. The current study shows that enzymatic interesterification is able to produce CBE with physicochemical properties that are close to CB. Sample CBS-B2 is potentially used as a CBE in confectionary applications.

16. **Composition and Physicochemical Properties of Single-stage Solvent-fractionated Palm Stearin.** P. Podchong[1], S. Sonwai[1], and D. Rousseau[2], [1]Silpakorn University, Thailand, [2]Ryerson University, Canada.

The physicochemical properties of palm stearin (PS) and its fractions obtained via solvent fractionation at 30, 35 and 40°C were investigated using gas chromatography, high-performance liquid chromatography, differential scanning calorimetry, polarized light microscopy, pulsed nuclear magnetic resonance and x-ray diffraction. PS with an iodine value (IV) of 37 was fractionated into various liquid (LF) and solid (SF) fractions (24>IV>51). The IV of the LF increased with a decrease in fractionation temperature whereas that of SF (IV~24) was not affected. The main fatty acids of all PS fractions were palmitic and oleic acid, with the LFs having a lower palmitic acid but higher oleic acid content than the SFs. Fractionation temperature affected the thermal behaviour of all LFs, these showing much sharper melting profiles than the SFs. LFs and SFs obtained at higher fractionation temperatures showed faster crystallization rates. The LFs fractionated at 30, 35 and 40°C consisted of needle-like spherulites, spherulitic crystalline assemblies and dendritic crystals respectively whereas all SFs consisted of densely-packed, overlapping dendritic crystals. All samples existed as mixtures of β' and β crystals. This study serves as the basis for future efforts focusing on the development of tablespreads using fractionated PS.

17. **Physical and Oxidative Stabilities of Diacylglycerol Rich Algae Oil-in-Water Emulsion.** Y.S. Kim, B.R. Na, K.S. Shin, and J.H. Lee*, Dept. of Food & Nutrition, Daegu University, Republic of Korea.

Diacylglycerol (DAG) rich oil containing 55.9% DHA was synthesized with algae oil by lipase-catalyzed glycerolysis. The DAG rich oil consisted of 37.8% TAG, 54.7% DAG, and 7.4% MAG. Tocopherol contents of DAG oil and algae oil were 1.58g/kg and 1.62g/kg, respectively. Oil-in-water emulsions with DAG oil and algae oil were prepared with three emulsifiers of 0.5% Tween 80, sodium caseinate and whey protein concentrate (WPC), and their emulsion and oxidative stabilities were compared by measuring particle size and formation of hydroperoxide and TBARS during storage. Particle size ($d_{4,3}$) of oil-in-water emulsion was lower with DAG rich oil (0.33~2.56μm) than algae oil (0.44~13.50μm) during 28 days. Hydroperoxide and TBARS values were significantly lower in emulsion with DAG oil than algae oil (p<0.05) during storage at 40°C. Among the emulsifiers used for DAG oil-in-water emulsion tween 80 resulted in a lower particle size and hydroperoxide formation than sodium caseinate and WPC. The present study suggests that DAG rich oil synthesis from algae oil could deliver physically and oxidatively stable oil-in-water emulsion as well as high level of DHA supplementation which has beneficial effect for brain development.