## LOQ 1a: Lipid Oxidation Evaluation by Sensory Methods

*Chairs: Minwei Xu, North Dakota State University, USA; and Shalla Ramnarain, DSM Nutritional Products, USA*

**Sensory Foundations, What to Keep in Mind When Evaluating Products** Bob Baron*, *Sensory Spectrum, Inc., USA*

Sensory Evaluation is defined as a scientific discipline which deals with the methods to evoke, measure, analyze, and interpret human responses to properties of foods and materials as perceived through the five senses taste, smell, sight, hearing, and touch. As a discipline, it can be used effectively to support product stability, raw material quality, product development, and consumer insight. Used improperly however it can lead to misguided and incorrect assumptions. This presentation will focus on basic Sensory Evaluation principles, practices, and proper execution. The human body is a highly complex and sensitive instrument for measuring stimuli, often time more sensitive than laboratory analytical equipment. Sensory Scientist use affective and discriminative methods for measuring stimuli. Affective methods focus on subjective measures such as liking or preference. These methods are commonly referred to as consumer test. Strict control over the screening of participants, questionnaires, sample preparation, and test conditions are required for this method. Discriminative methods are objective and designed to detect difference. They are further differentiated as Difference Testing and Descriptive Testing. Difference Testing focuses on identifying difference between or among samples. Descriptive Testing uses trained assessors to describe and intensify attributes of products. These methods must also be executed with very specific and controlled procedures. A summary of the requirements for these types of Sensory methods will be addressed. Additionally, the errors that can occur when proper sensory testing procedures are not followed will be addressed by focusing on the five S's (Subjects, Site, Samples, Statistics, Sensory Methods).

**Characterization of the "Fusty, Muddy Sediment" and "Musty" Off-flavor of Olive Oils Using the Molecular Sensory Science Concept** Michael Granvogl*, Anja Neugebauer, and Peter Schieberle, *Technical University of Munich, Germany*

Due to its valuable nutrients and its unique flavor profile, olive oil is a favored edible oil around the globe for hundreds of years. The aroma of a certain food is one of the most important criteria for consumers' buying behavior. Thus, it is of high importance to gain detailed knowledge about the aroma profile of olive oil eliciting perfect sensory attributes and, in contrast, of olive oils showing sensory defects leading to a so-called off-flavor development leading to consumers rejection. Thereby, the off-flavor may be formed either during production or storage. Using the molecular sensory science concept, including comparative aroma extract dilution analysis (cAEDA) based on gas chromatography-olfactometry (GC-O) and identification experiments via gas chromatography-mass spectrometry (GC-MS) as well as quantitation via stable isotope dilution analysis (SIDA), extra virgin olive oil eliciting the desired aroma properties and olive oils with "fusty, muddy sediment" and "musty" off-flavors were compared. For the most potent aroma compounds odor activity values (OAVs; ratio of concentration to respective odor threshold) were calculated. Finally, aroma simulation experiments were carried out to validate the analytical data. Fourteen odorants with an OAV ≥ 1 were detected as key aroma compounds in extra virgin olive oil including 1-

penten-3-one, (*Z*)-3-hexenal, *trans*-4,5-epoxy-(*E*)-2-decenal, acetic acid, 2-isobutyl-3-methoxypyrazine, (*E*)-2-hexenal, and (*Z*)-3-hexenylacetate. Twenty-three odorants with an OAV ≥ 1 were found in the "fusty, muddy sediment" off-flavor oil revealing butanoic acid, 4-methylphenol, 2-methoxyphenol, ethyl butanoate, hexanal, acetic acid, ethyl 2-methylbutanoate, and pentanoic acid with the highest OAVs. The "musty" off-flavor olive oil showed 21 aroma compounds with OAVs ≥ 1 with 2-methoxyphenol, butanoic acid, hexanal, acetic acid, and ethyl 2-methylbutanoate as key odorants. The lecture will summarize the results for the "fusty, muddy sediment" and the "musty" off-flavor obtained for the first time by the systematic characterization on a molecular level based on the combined approach of instrumental and sensorial analysis. Differences will be demonstrated and possible formation pathways for the compounds responsible for the off-flavor development will be discussed.

**Best Practices for In-house Sensory Testing of Oils and Fats** Clare A. Dus* and Lee Stapleton,*Sensory Spectrum, Inc., USA*

Smelling, tasting, feeling and/or visual assessments of Oils or Fats are conducted by Producers every day and for many purposes. Whether as a QC step during production, emergency investigation of a real or suspected product failure, ongoing shelf life test, competitive screening or check during development, product evaluations by human testers provide meaningful information for decision making…..if they are performed with reasonable scientific rigor. Historically, that has not always been the case. This paper provides best practices for internal product evaluations that apply to routinely scheduled tests using trained personnel as well as to occasional testing using small groups of employees. Minimizing subjectivity in testing, tips for minimizing carryover and fatigue with edible oils and procedures for assessor preparation, data collection, and data interpretation are among the topics addressed.

**Understanding Aroma Off-notes with Analytical Techniques** Tanya F. MacGillivray*, *DSM Nutritional Products, Canada*

The right analytical techniques can provide great insight into sensory data. Methods for analyzing headspace volatiles can help identify the source of aroma off-notes. This is particularly important for products containing Omega-3 oils, where aroma has a strong influence on overall acceptability. Various techniques are available to provide complementary information to give greater depth of understanding of such off-notes. Good understanding of an off-note is the first step to reducing/preventing it in the future.

## ANA 1.1 / LOQ 1b: Evaluation and Prediction of Oxidative Stability and Shelf-life

*Chairs: Hong-Sik Hwang USDA, ARS, NCAUR USA; and Min Hu DuPont Nutrition & Health USA*

**Analyzing Multiple Lipid Oxidation Products—Required, or Not?** Karen M. Schaich*, *Dept. of Food Science, Rutgers University, USA*

With increasing use of polyunsaturated lipids in foods, there is increasing challenge to prevent oxidation and loss of beneficial health effects as well as to maintain food quality. In addition, as BHA and BHT are replaced by natural antioxidants, resulting oxidation in foods must be tracked accurately to assess effectiveness of new formulations. Traditional approaches to analyzing lipid oxidation focus mostly on hydroperoxides and perhaps also volatile products such as hexanal. However, routine analyses of multiple lipid oxidation products from alternate competing pathways, generating epoxides, dimers, and hydroxylipids are almost never performed and detailed information about volatile products other than hexanal is seldom collected. These practices underestimate extent of lipid oxidation and misses important products, particularly epoxides and hydroxylipids, that have toxic potential and provide evidence for shifted mechanisms of lipid oxidation. This paper discusses these issues in the context of mechanism and antioxidant studies, with consideration of new assays for epoxides and hydroxylipids.

**Explaining the Polar Paradox and Cut-off Effect for AO Distributions and Reactivity in Emulsions** Laurence S. Romsted*[1], and Carlos Bravo Diaz[2], *[1]Rutgers University, USA; [2]University of Vigo, Spain*

The polar paradox and cut-off effect are two important general observations about antioxidant (AO) efficiencies and distributions in surfactant-based emulsions that have, until recently, evaded clear explanation. Both problems were solved using the chemical kinetic method. The results provides values for the distributions of AOs of different alkyl chain lengths between the aqueous and interfacial regions and the oil and interfacial regions of emulsions and also provide a method for comparing AO efficiencies in different emulsions. In addition, a maximum appears naturally in the distributions of AOs with increasing alkyl chain lengths that depends on AO hydrophobicity, matches AO efficiencies, and explains the cut-off effect. The chemical kinetic method is based on the use of a chemical probe and the results are interpreted using pseudophase kinetic models that were originally developed in homogeneous association colloids, but also work in kinetically stable emulsions for two reasons: (a) in kinetically stable emulsions, the totalities of emulsified surfactant, oil and aqueous regions act as separate reaction regions, i.e., the observed reaction rates depend on the total volume of each region and not on droplet size; and (b) reactant distributions are in dynamic equilibrium between the oil, interfacial and aqueous regions. The talk will include: (a) the logic and basic assumptions of the pseudophase kinetic model as applied to emulsions; (b) the important properties of the chemical probe, a hydrophobic arenediazonium ion; (c) a brief description of the dye derivatization method for monitoring reactions in emulsions; and (d) the explanation for the cut-off effect.

**Oxidative Stability and Shelf-life of Bulk Oils and Fats** Min Hu*, *DuPont Nutrition & Health, USA*

Bulk oils and fats can be used as ingredients to develop a variety of foods to increase nutritional value, impact structures and enhance the flavor of the foods. The quality and oxidative stability of bulk

oils and fats have big impacts on the oxidative stability and shelf-life of foods containing the bulk oils and fats. Examples of bulk oils and fats available in food industry are: vegetable oils such as soybean, canola, corn, sunflower, safflower coconut, palm and flaxseed oils, high oleic soybean, canola and sunflower oils, fish, algal and krill oils, and plant-based EPA and/or DHA oils; animal fats such as chicken and pork fats, and tallow. There are a number of methods that may be employed to assess the oxidative stability of bulk oils and fats. There are many antioxidants that may be selected to enhance oxidative stability of the bulk oils and fats. In the presentation, we will address fatty acid profiles of the bulk oils and fats, and will highlight how to select appropriate methods to evaluate the oxidative stability and shelf-life of bulk oils and fats, and to increase the oxidative stability of the bulk oils and fats.

**Correlation of Sensory Evaluation with Chemical Assays in Oils/Fats and Oil/Fat-based Foods** Robin Boyle, and Nora Yang*, *Kalsec, Inc., USA*

The most important method to assess the flavor quality of foods is sensory evaluation, which plays a key role to determine the shelf life of foods; however, sensory evaluation is subjective and can be very time and cost-consuming. Therefore, chemical analyses that generate more reliable and accurate information from the molecular level are attracting more and more interest in both the food industry and academia. The most crucial question is whether there is correlation between sensory evaluation and chemical assays. This talk will focus on examining whether the most commonly used chemical markers, such as free fatty acids, peroxides, anisidine values and volatiles as secondary oxidation compounds, can be utilized to predict the stability and quality of oils/fats and oil/fat-based foods. Both opportunities and challenges in this area will be discussed

## LOQ 2a: Metabolic Products and Toxicity of Lipid Oxidation and Antioxidants in Foods and Biological Systems

*Chairs: Weerasinghe Indrasena, DSM Nutritional Products, Canada; and Jian Kong, Abbott Nutrition, USA*

**Successful Prevention of Fish Oil Flavor Deterioration by Sphingoid Base** Mariko Uemura[1], Ako Shibata[1], Saiko Sudo[1], Masashi Hosokawa[1], Kazuo Miyashita*[1], Ai Iwashima-Suzuki[2], Ai Iwasawa[2], and Makoto Shiota[2], *[1]Hokkaido University, Japan; [2]Megmilk Snow Brand Co. Ltd., Japan*

Eicosapentaenoic acid (20:5n-3, EPA) and docosahexaenoic acid (22:6n-3, DHA) are active forms of n-3 polyunsaturated fatty acids (PUFA) and are found in fish oils. Considering the strong link between the consumption of both PUFA and the reduction of cardiovascular disease risk, a high intake of fish oil-containing foods and supplements has been widely recommended. On the other hand, fish oil is highly susceptible to oxidation, which causes undesirable flavors and loss of the functionality of n-3 PUFA. Natural antioxidants are often added to protect EPA and DHA from oxidative deterioration; however, it has been still very difficult for them to avoid the flavor deterioration, even though they can inhibit hydroperoxide formation. In the present study, we demonstrate the complete inhibition of volatile formation in fish oil oxidation by the combination of dihydrosphingosine (d18:0) and tocopherol. The analysis of oxygen consumption and total volatile formation showed a small effect of d18:0 on TAG oxidation in the absence of tocopherol, while the volatile formation was completely inhibited by the combination of d18:0 with tocopherol. Several experimental results strongly suggest the formation of antioxidants by reaction of d18:0 with aldehydes formed during the very early stage of oxidation in the presence of tocopherol. The antioxidants produced could effectively inhibit the lipid oxidation by the regeneration of α-tocopherol and/or direct scavenging free radicals. At the same time, above reaction could scavenge volatiles responsible for the flavor deterioration.

**Lipid Oxidation Products and Aging** Mary E. Camire*, *University of Maine, USA*

As the human lifespan increases around the world, more research addresses physiological and anatomical changes associated with aging. A lifetime of exposure to radiation and free radicals impacts tissue damage. Diet and lifestyle can influence such damage, but interventions such as supplementation with antioxidant nutrients are not always successful in controlling aging-related damage. Free radicals can alter many compounds in the body, including lipids, proteins, carbohydrates and DNA. At the cellular level, methylation of DNA is being studied as an epigenetic marker of aging. Free radicals are associated with atherosclerosis, cancer, and inflammatory diseases as well as conditions typically accepted as a “normal” consequence of aging- reduced vision and coordination, and increased skin wrinkling. Can dietary interventions later in life control or reverse tissue damage accumulated over decades? Do the Dietary Guidelines for Americans provide adequate guidance for minimizing aging-related damage? Interactions among nutrients, phytochemicals, macronutrients and lifestyle will be addressed.

**Lipid Oxidation in Complex Nutritional Matrices** Gary Katz*, *Abbott Nutrition, USA*

Dietary lipids are of major importance for the growth and development of infants and to meet

the nutritional needs of children and adults. They provide a significant source of energy in infant formulas, and help to facilitate the absorption of lipid soluble vitamins and other key nutrients such as carotenoids. The body of knowledge around the nutritional and developmental value of polyunsaturated fatty acids is continually growing, and incorporation of these key fatty acids into complex nutritional matrices presents a challenge. A number of approaches can be taken to assure the nutritional, oxidative and organoleptic quality of polyunsaturated fatty acids in nutritional products. This begins with identifying and sourcing high quality oils, and developing product formulations that meet the specific nutritional needs of the target population. Approaches also include the incorporation of effective antioxidant systems to minimize lipid oxidation, designing a manufacturing process that is compatible with the sensitive ingredients, and establishing a quality assurance program that utilizes the appropriate analytical approaches to monitor key quality attributes throughout the manufacturing process and during shelf life. Understanding, anticipating and controlling potential interactions that occur when ingredients are combined and exposed to various processing parameters is the key to managing potential lipid oxidation in these matrices, and delivering a high quality nutritional product that meets the needs of the consumer.

**Re-evaluation of Toxicity of Lipid Oxidation Products and Natural Antioxidants** Karen M. Schaich*, *Dept. of Food Science, Rutgers University, USA*

Toxicity of food components is now under intense scrutiny as the general public has become increasingly concerned about the safety of our food supply. Synthetic antioxidants BHT and BHT, presumed to be carcinogenic, are being replaced by natural antioxidants, assumed to be safe. However, the literature is replete with misinformation on both sides. Products of lipid oxidation and thermal degradation, have long been recognized as potential toxins, but have received little attention since the low/no fat era. Thus, the time seems ripe to re-evaluate whether and how oxidizing lipids and natural antioxidants as consumed in foods may (or may not) be toxic, considering levels ingested, modification during digestion, absorption vs action only in the gut, contradictory evidence for biological effects published in the literature, and consequences to all this for food stabilization approaches. Particular attention is given to toxicity of epoxides and potential physiological effects of hydroxylipids that have been largely ignored.

**Acrolein as Important Flavor Contributor in Fish Oil Oxidative Deterioration** Ako Shibata, Mariko Uemura, Masashi Hosokawa, and Kazuo Miyashita*, *Hokkaido University, Japan*

Eicosapentaenoic acid (EPA; 20:5n-3) and docosahexaenoic acid (DHA; 22:6n-3) are typical n-3 polyunsaturated fatty acids (PUFA) found in fish oils. These two long-chain PUFA have been demonstrated to cause significant biochemical and physiological changes in the body that primarily exhibit a positive influence on human nutrition and health. However, the development of fishy and metallic off-flavors that are often found in fish oils rich in EPA and DHA dissuades people from consuming them. Usually, flavor deterioration is analyzed GC method. The typical GC analysis of the volatile compounds from oxidized lipids is a dynamic headspace method with a SPME fiber. However, there is a limitation in the analysis of several characteristic compounds when using the SPME method. Lower-boiling compounds, especially carbon 3 compounds may be lost during the purging cycle in the SPME method, whereas relatively higher molecular weight compounds,

such as heptadienal and decadienal, can be concentrated in the trap. On the other hand, a static headspace GC analysis can measure the actual level of volatile compounds, including low molecular weight or low boiling volatile compounds. By using sensitive static headspace method, we have found higher quantities of acrolein (2-propenal) as a major volatile at the early stages of fish oil oxidation. The preferential formation of acrolein during the fish oil TAG oxidation would be strongly related to the flavor deterioration of fish oil, especially during the very early stage of the oxidation.

## LOQ 2b: Stabilization of Omega-3, Bioactive Lipids, and Antioxidants Strategies

*Chairs: Constantijn Sanders, Nestlé, Switzerland; and Kazuo Miyashita, Hokkaido University, Japan*

**High Throughput Electro-hydrodynamic Processing for the Encapsulation of DHA Enriched Oils** Jose M. Lagaron*, *CSIC, Spain*

Electro-hydrodynamic processing, comprising electrospraying and electrospinning, is a physical technology used for the formulation of capsules and fibers with controlled size, size distribution and phase morphology by subjecting a solution or a melt to a high electric field. At a critical high voltage (2-100 kV), the solution droplet at the tip of the injector distorts and forms the so-called Taylor cone to be ejected as a charged jet. This jets containing the solids fraction stretches and is accelerated by the electrical field towards the collector. As the jet travels through the electric field, the solvent is completely evaporated while the control over the encapsulant entanglements facilitates or prevents it from breaking up, resulting in capsules or in fibers, respectively, which most efficiently encapsulate the lower molecular weight bioactive molecules. Up until recently this technology, especially the electrospraying process, remained to a laboratory scale, however, recent developments in engineering and industrial investment have allowed this unique non-thermal processing technology to be scaled up by the company Bioinicia S.L. to achieve the production volumes required by pharma/cosmetic and even food related industrial commodity applications. The current paper will introduce the technology and highlight the recent advances over other existing technologies, to offer an innovative approach for the stabilization, shelf-life extension and controlled release of DHA enriched oils.
Acknowledgements: Contract sponsors: Bioinicia S.L and the Spanish MINECO (Grant AGL2015-63855-C2-1-R)

**Influence of Legume Proteins on the Lipid Oxidation of Omega-3 Emulsions** Cansu E. Gumus*, Eric A. Decker, and David J. McClements, *University of Massachusetts Amherst, USA*

There are issues with incorporating omega-3 fatty acids into foods due to their low water-solubility and oxidative instability, which may be overcome using emulsion-based delivery systems. There is growing interest among consumers in “clean label” and all plant-based food products. Therefore, the food industry is looking for ways to address consumer concerns about synthetic ingredients. The aim of this study is to provide a better understanding of the effects of several surface-active plant-proteins on lipid oxidation of omega-3 emulsions. Lipid oxidation in oil-in-water emulsions depends on the thickness of the interfacial layer formed by the proteins, as well as the amino acid composition of the proteins utilized. Primary and secondary lipid oxidation products of omega-3 emulsions stabilized by surface-active plant proteins (lentil, pea, faba bean) and whey protein were monitored by peroxide value and thiobarbituric acid reactive substances (TBARS) assays. The particle size, charge and microstructure of the emulsions were monitored using static and dynamic light scattering and confocal microscopy to detect changes in physical stability also. Even though it is reported elsewhere that the free sulfhydryl group of cysteine can inhibit lipid oxidation by scavenging free radicals, blocking the free sulfhydryl groups of protein isolates did not affect their ability to inhibit lipid oxidation in emulsion systems.

**Label Friendly Ingredient Toolbox for the Stabilization of Omega-3 at Different Stages** Lan Ban, Joan Randall, Yvonne Gildemaster, Marie Shen, and Will Schroeder*, *Kemin Food Technologies, USA*

Omega-3 fatty acids are highly susceptible to oxidative stress. Oxidation will greatly reduce the nutritional values of the fatty acids as well as the food that is fortified with it. Omega-3 degrades through oxidation not only through storage of fish oil or in the fortified foods, but harsh processing conditions to manufacture food grade fish oil or fish meal also makes it challenging to maintain the derisible shelf life of omega-3. In this study, two new clean-label ingredients, water-soluble spearmint extract and oil-soluble green tea extract, are introduced and tested either alone or in combination with various common ingredients including tocopherols, ascorbyl palmitate and rosemary extract. Fish meal, fish oil and a model food system with fortified salad dressing were tested separately as the matrices which represent omega-3 in different stages. Accelerated oxidative stability, oxidative byproducts and organoleptic properties are monitored to evaluate the effectiveness of the ingredient combinations. The results have shown that different blends are required for each specific application. Oil-soluble green tea extract and water-soluble spearmint extract have shown to be good free radical scavengers with tolerance of heat treatment, and represent ingredients with different polarities that would work in different micro-domains in a complex food system. The ingredient combinations that have been developed in this study have benefits as clean label options for omega-3 oxidative stability improvement and they are application specific.

**Oxidative and Physical Stability of Fish Oil-in-Water Emulsions Stabilized with Sodium Caseinate and Modified Alginate** Betül Yesiltas*[1], Ann-Dorit M. Sørensen[2], Pedro J. Garcia-Moreno[2], Sampson Anankanbil[3], Zheng Guo[4], and Charlotte Jacobsen[2], *[1]National Food Institute, Technical University of Denmark, Denmark; [2]Technical University of Denmark, Denmark; [3]Dept. of Engineering, Aarhus University, Denmark; [4]Aarhus University, Denmark*

The objective of this study was to create oxidatively and physically stable high fat (50-70%) fish oil-in-water emulsions by combined use of sodium caseinate (CAS) and commercial/modified alginates. It is hypothesized that modified alginate works as an emulsifier together with CAS and partition between the water-oil interface and water phase leading to increase in physical stability by increasing viscosity and creaming stability. Oxidation stability was also expected to be increased by antioxidative properties of modified alginates and providing a physical barrier at the water-oil interface. Three types of alginates namely commercially available sodium alginate (Commercial alginate – CA), alginate modified with succinic anhydride (SAC0) (Short chain modified alginate – SCMA) and alginate modified with dodecenyl succinic anhydride (SAC12) (Long chain modified alginate – LCMA) were used in combination with CAS. The fish oil ratios used was 50, 60 or 70%. Total emulsifier content was 1.4% and the ratio of CAS to alginate was 1.2. Physical stability was evaluated by droplet size, viscosity, creaming and zeta potential; whereas oxidative stability was determined by measuring primary oxidation products (peroxide value) and volatile secondary oxidation products (GC-MS). Results showed that viscosity and creaming stability increased with increasing fish oil content. Emulsions prepared with CAS+LCMA or CAS+CA were physically stable but oxidized more compared

to emulsions prepared with CAS+SCMA or only with CAS. Emulsions with only CAS were not physically stable in terms of creaming. SCMA improved physical stability of emulsions and kept the oxidation stability at the same high level as when only CAS was used.

**Ferulates in Fish Oil Enriched Milk: Partitioning, Protein, and Lipid Oxidation** Xujian Qui[1], Charlotte Jacobsen[2], Pierre Villeneuve[3], and Ann-Dorit M. Sørensen*[2], *[1]National Food Institute, Technical University of Denmark, Denmark; [2]Technical University of Denmark, Denmark; [3]CIRAD/INRA, UMR 1208 IATE, France*

Omega-3 PUFA containing foods are highly susceptible to lipid oxidation. One strategy to limit lipid oxidation is addition of antioxidants. The efficacy of antioxidants can vary with the complexity of the food matrix. Lately, extensive work has been performed on phenolipids (caffeates and ferulates) and their antioxidant efficacy in food emulsions. Results indicated a cut-off effect in relation to the alkyl chain length grafted to the phenolic compounds. Different phenolipids resulted in different critical chain lengths (cut-off) for various food emulsion systems. For ferulates a clear cut-off effect at a chain length of 1 carbon (methyl ferulate) was observed, whereafter the antioxidative efficacy dropped. Milk is a complex food matrix with several components. In the earlier study only lipid oxidation was followed during storage. The aim of this study was to evaluate the antioxidative effect of ferulic acid and its esters, ferulates, in fish-oil-enriched milk. Ferulic acid, methyl ferulate and dodecyl ferulate were applied as antioxidants in this study. Additionally, synergistic effects of combinations of ferulic acid – dodecyl ferulate and methyl- and dodecyl ferulates were evaluated. Lipid oxidation (peroxide value, volatiles and tocopherol concentrations) and protein oxidation (carbonyls) were measured during storage. Moreover, partitioning of the ferulates in the milk was determined to enable interpretation of lipophilicity, antioxidative effect and partitioning observed in milk. The presentation will discuss the observed antioxidative effects of the ferulates in relation to their partitioning in the milk system.

## LOQ 3a: Lipid Oxidation and Antioxidants—Fundamentals and Applications

*Chairs: David Johnson, University of Massachusetts Amherst, USA; and Fereidoon Shahidi, Memorial University of Newfoundland, Canada*

**Lipid Oxidation and Antioxidants: Fundamentals and Applications** Fereidoon Shahidi*, *Memorial University of Newfoundland, Canada*

Oxidation is a deteriorative process that affects the wholesomeness and properties of fats and oils and products containing them. The oxidation process generally proceeds via autoxidation, photooxidation, thermal oxidation and enzymatic oxidation. While the processes involved dictate the outcome, antioxidants are generally employed to arrest oxidation and extend the shelf life of products. In general, the fatty acid composition, positional distribution, minor components, storage conditions and presence of antioxidants deliberately added to foods affect the oxidation of foods. As clean label is now most desirable, use of natural inhibitors of oxidation is preferred, but this may also face regulatory challenges. When consumed, the dietary antioxidants may exert a positive influence of oxidative stress condition in the body and contribute to health promotion. Examples will be provided to briefly illustrate different factors involved in lipid oxidation and its control by employing antioxidants.

**Protein-polysaccharide Mixtures as Wall Material in Fish Oil-loaded Nano-microcapsules Obtained by Electrospraying** Pedro J. Garcia-Moreno*, Andres Pelayo, Ramona V. Mateiu, Ioannis S. Chronakis, and Charlotte Jacobsen, *Technical University of Denmark, Denmark*

Omega-3 polyunsaturated fatty acids (PUFA) are highly susceptible to oxidation, which limits their use as nutritionally beneficial lipids in foods. Electrospraying is a promising technique for encapsulation of omega-3 PUFA since the process does not require the use of heat, which avoids deterioration of these thermosensitive compounds. Furthermore, nano-microcapsules with a reduced size are obtained by this technique, which makes their dispersion into food matrices easier when compared to traditional encapsulates (e.g. microcapsules produced by spray-drying). Biopolymers such as proteins (e.g. zein, whey protein) and polysaccharides (e.g. pullulan, dextran) are required for the production of food-grade nano-microcapsules. Wall materials consisting of both proteins, which exhibit emulsifying properties, and polysaccharides, which influence the glass transition temperature of the glassy matrix and thus the transport within it, have been suggested to improve the oxidative stability of traditional fish oil encapsulates. The aim of this work was to develop oxidatively stable fish oil-loaded nano-microcapsules by electrospraying with protein (e.g. zein, whey protein)-polysaccharide (e.g. pullulan, dextran) mixtures as wall materials. First, the influence of total concentration of biopolymers and polysaccharide to protein ratio on the morphology of the capsules and on the yield and productivity of the process was assayed. Secondly, the effect of the oil-incorporation approach (e.g. mechanical stirring or high pressure homogenization) on the ability of the capsules to entrap the oil and on the oil distribution was investigated. Finally, the oxidative stability of selected nano-microcapsules was monitored by determining the formation of lipid hydroperoxides and volatiles oxidation products.

**Impact and Parameters of Active Oxygen Scavenging Packaging on the Oxidative Stability of Oil-in-Water Emulsions** David Johnson*, and Eric A. Decker, *University of Massachusetts Amherst, USA*

The oxygen content of food systems is one of the most important factors influencing the rate and extent of lipid oxidation. Previous research in our lab has shown that greater than 90% systemic oxygen reduction is needed in order to meaningfully extend the oxidative stability of oil-in-water emulsions. In the present work, conditions were defined where oxygen scavenging packaging could inhibit lipid oxidation in a model fish oil-in-water emulsion system. The oxygen scavenging technology is based on an iron-based oxidation that consumes oxygen, but is still separated from the food contact by multiple layers of plastic. The active packaging was able to scavenge oxygen from the emulsion within the first 24 hours of storage at 32°C and oxygen reduction values were quantified simultaneously and non-destructively, by O2-sensitive fluorescent sensors. Lipid oxidation markers, as measured by PV and TBARS, suggested that the >90% reduction in systemic oxygen by the commercial active packaging lead to enhanced oxidative stability. The efficacy of this packaging was determined across ionic strength, pH, and fat concentration. Further, the activity of antioxidants under reduced oxygen atmospheres are discussed. Parameters for when oxygen reduction can be a successful antioxidant strategy for emulsions are outlined. The results of this study provide a reference and parameters where active oxygen scavenging packaging can be an effective and 'label friendly' option.

## LOQ 3b: New Antioxidants from Agricultural By-products, Food Processing Waste, and New Sources

*Chairs: Xin Tian, Kalsec, Inc., USA; and Min Hu, DuPont Nutrition & Health, USA*

**Food Processing and Agriculture By-products as Natural Sources of Antioxidants: Reviews and Challenges** Xin Tian*, *Kalsec, Inc., USA*

The growing interest in the replacement of synthetic food antioxidants has led to increasing investigations in the field of naturally-sourced antioxidants. With the high cost implicated with these materials, the exploration of antioxidants from agriculture residual origin emerged in large numbers in recent years that looked into cost-effective alternatives of natural antioxidants. These exploration tallies with initiatives to solve substantial food waste problems around the global. The present work provided a comprehensive review of the most recent research on the recovery and utilization of food processing wastes. In most cases, high levels of active compounds were found in waste materials, which encompass a wide range of sources from fruits and vegetables, roots and tubers, to grains and seeds. Gaps in the area of by-products research, extraction and recovery technologies, as well as constraints on commercialization were also discussed.

**Application of Antioxidants from New Sources in Foods Rich in Omega-3 Fatty Acids** Charlotte Jacobsen*[1], Ditte B. Hermund[1], Sabeena Farvin Koduvayur Habeebullah[2], Ann-Dorit M. Sørensen[1], and Pedro J. Garcia-Moreno[1], *[1]Technical University of Denmark, Denmark; [2]Environmental and Life Science Research Center, Kuwait Institute for Scientific Research, Kuwait*

Addition of synthetic antioxidants such as butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT) and propyl gallate are often used to control lipid oxidation in foods. However, the use of synthetic antioxidants has been restricted because of their health risks and toxicity. Moreover, consumers increasingly prefer food products with natural ingredients rather than synthetic ones. Phenolic compounds from plants and algae as well as peptides from dairy products, fish, meat and plants have been demonstrated to have excellent antioxidant properties and there are several examples on commercial applications of these antioxidants. During the last decade there has been an increasing focus on sustainable food production in which raw materials including side streams from food production are fully exploited and used to generate “value from waste”. Side streams from processing of agriculture products are in many cases rich in phenolic compounds with antioxidant activity. One example is potato peel. Extracts hereof have been demonstrated to have high antioxidative activity in various food products. This presentation will demonstrate how side streams from the fish and potato industry can be turned into antioxidant extracts with high activity in food products. Examples of studies on food applications of antioxidants from new sources such as seaweed will also be given. Results presented are obtained in the authors’ lab.

**Impact of Processing on n-3 Long Chain Poly-unsaturated Fatty Acids Derived from Microalgae** Lore Gheysen[1], Tom Bernaerts[2], Charlotte Bruneel[1], Koen Goiris[3], Jim Van Durme[3], Ann Van Loey[2], Luc De Cooman[3], and Imogen Foubert*[1], *[1]Katholieke Universiteit Leuven Kulak, Belgium; [2]Katholieke Universiteit Leuven, Belgium; [3]Katholieke Universiteit Leuven, Technology Campus Ghent, Belgium*

There is a need for an alternative source of n-3-LC-PUFA due to the reducing fish stock. Microalgae

could provide such an alternative. In addition, the daily intake of n-3-LC-PUFA is not reached in Western countries. Therefore it is useful to enrich food products with n-3-LC-PUFA from microalgae. Mechanical and thermal processing are crucial steps in the production of food products. However this could result in a reduction of the amount of n-3-LC-PUFA and the formation of oxidation and polymerization products. This study investigates the impact of processing on different microalgae (Isochrysis, Nannochloropsis, Phaeodactylum, Porphyridium and Schizochytrium) delivered as biomass. For each microalgae an artificial aqueous matrix was made containing 80 mg n-3-LC-PUFA. The matrix was a model system for fruit and vegetable based products. Therefore the pH was reduced to 4. In a next step a mechanical processing was done by high pressure homogenization and this was followed by thermal processing. As is done for real food products with a pH of 4, the matrix was pasteurized. The impact of (mechanical and thermal) processing on the n-3-LC-PUFA enriched matrices was followed in a dual way. First, the matrices were characterized before and after processing for the amount of n-3-LC-PUFA, free fatty acids, carotenoids and the amount of lipid polymers. In addition, the fully processed samples were stored for 12 weeks at 37°C to follow up their oxidative stability at different time intervals. The results showed that different microalgae were not affected to the same degree due to differences in fatty acid profile, type and amount of carotenoids, ...

**Antioxidant Effect of Water and Acetone Extracts of Fucus Vesiculosus on Oxidative Stability of Skin Care-emulsions** Candelaria Poyato[1], Birgitte R. Thomsen*[2], Ditte B. Hermund[2], Diana Ansorena[1], Iciar Astiasarán[1], Rosa Jónsdóttir[3], Hordur G. Kristinsson[3], and Charlotte Jacobsen[2], *[1]University of Navarra, Spain; [2]Technical University of Denmark, Denmark; [3]Matis, Iceland*

Naturally derived antioxidants from various plants and Marine algae have shown great potential in improving oxidative stability in these kinds of products. A high variety of bioactive compounds, such as pigments, sulphated polysaccharides, proteins and polyphenols, have been described for different types of Danish brown and red algae Naturally derived antioxidants from brown algae have shown great potential in improving oxidative stability. Brown algae contain a high variety of bioactive compounds with anti-oxidative effects, such as pigments, sulphated polysaccharides, proteins and polyphenols. In this study, water and acetone extracts of the Icelandic brown algae Fucus vesiculosus were evaluated as potential natural sources of antioxidant compounds in skin care emulsions. Their efficacy in inhibiting lipid oxidation caused by photo- or thermo-oxidation was evaluated by exposure to light and room temperature, or darkness and 40°C during 56 days. Changes in lipid oxidation and product quality were measured by color, droplet size, fatty acid composition, tocopherol content, peroxide value and dynamic headspace GC-MS. The result showed that presence of extracts in the skin care emulsions induced remarkable color changes when the emulsions were exposed to light, and more extensively under high temperature. High temperature also caused greater increments in the droplet size of the emulsions. The analysis of the tocopherol content, peroxide value and volatile compounds during the storage revealed that, whereas both water and acetone extracts showed protective effect against thermo-oxidation, only the water extract showed antioxidant activity against photo-oxidation.

## LOQ 4a: Novel Strategies to Stabilize Foods with and without Antioxidants

*Chairs: Vishal Jain, Mondelēz International Inc., USA; and Will Schroeder, Kemin Food Technologies, USA*

**Synthesis and Characterization of a Novel Array of Phenolic-containing Emulsifiers: A Physicochemical Study** Sampson Anankanbil*[1], Zheng Guo[2], and Bianca Perez[1], *[1]Dept. of Engineering, Aarhus University, Denmark; [2]Aarhus University, Denmark*

Lipid autoxidation in emulsions is postulated to occur as a result of interactions between trace transition metals in the aqueous phase and preformed lipid hydroperoxides located at the oil-water interface. As the main barrier to prevent the diffusion of oxygen and free radicals, the thickness, molecule packing and mechanical stability of interfacial layer are governing the physical and oxidative stability of the emulsion. Hence, a surface-active molecule containing a phenolic moiety might be an ideal surfactant to function as both emulsifier and antioxidant to trap diffusing free radicals at the interface. To this end, a new homologous series of amphiphilic lipids were synthesized through the acylation of monoacylglycerols with phenolic acid derivatives. The resulting products were structurally identified and characterized by means of mass spectroscopy, Fourier transform infrared spectroscopy (FTIR), and 1H and 13C nuclear magnetic resonance. A pronounced structure-property relationship was established through Differential Scanning Calorimetry (DSC) and Langmuir monolayer analysis in correspondence to their molecular packing and assembly at the interface. The newly synthetic amphiphilic lipids displayed excellent dual functionality as oil-in-water emulsifiers and as antioxidants against lipid peroxidation.

**Strategy to Develop Natural Antioxidant Solutions in Complex Food Systems** Denis Xie*, *Kalsec, Inc., USA*

Application of antioxidants in the modern food industry is a major method to help improve product's shelf-life and retain nutritional value. As the "cleaner label" trend becomes more and more popular in recent years, synthetic antioxidants like BHA, BHT and TBHQ are no longer preferred. Instead, the industry has started utilizing of "natural" or "clean label" options of antioxidants (rosemary extracts, green tea extract, etc.) Finding a balance between cost impact, flavor impact and efficacy with these natural products can be a challenge. One good method is to use a combination of antioxidants to achieve improvements. In this case, a number of "clean label" antioxidant combinations were investigated in multiple food products including different types of bulk oil, roasted seed/nuts and oil-in-water emulsions. The oxidation markers in these products were measured and tracked to reveal the effectiveness. It was found that comparing to using rosemary extracts alone, combination of rosemary extract with selections of tocopherols, ascorbic acid, green tea extract and citric acid can greatly improve the antioxidant capacity in these food products. Detailed data analysis will be discussed on how they extend the shelf-life based on target food composition, storage conditions and antioxidant's water/oil solubility.

**Impact of Storage Conditions on Lipid Oxidation and Effect of Ingredients with Antioxidant Properties** Lan Ban*, Marie Shen, Joan Randall, and Will Schroeder, *Kemin Food Technologies, USA*

The kinetic behavior of lipid oxidation, in or

without the presence of ingredients that have antioxidant capabilities, is impacted by factors including storage temperature and oxidizing reagents. Accelerated tests are commonly used to simulate lipid oxidation under ambient conditions. However, the difference in kinetics of lipid oxidation between those conditions, and its impact on common ingredients has not been fully evaluated. This study focuses on how different storage conditions affect the performance of antioxidative ingredients on lipid oxidation. In this study, canola oil was stored under three conditions and ingredients including tert-butyl hydroquinone (TBHQ), tocopherols and rosemary extract were compared to a new ingredient, oil soluble green tea extract. Oxidation by-products, ingredient degradation, organoleptic properties and the apparent oxidation kinetics were monitored. The results showed that relative ratios of resulting aldehydes were different for different storage conditions, indicating deviated oxidation pathways for accelerated storage. With the exception of tocopherols, the use of other ingredients improved the stability of the oils under all conditions. However, the level of improvement at different storage conditions was different. In addition, darkening of treated oils was observed during heated storage, but the color of the oils has no correlation with either the performance of an ingredient or the amount of oxidation byproducts. In conclusion, evaluation of ingredients with antioxidant capabilities need to be carried out with caution for accelerated conditions. Oil soluble green tea extract has comparable performance to TBHQ without affecting organoleptic properties of the oils at ambient temperature regardless of oxygen levels.

**Oxidative Stability Impact of Various Interesterification Methods on Edible Oils** Alex M. Milligan*, Joshua Tuinstra, Roger Daniels, and Matthew Ulmer, *Stratas Foods, USA*

Interesterified vegetable oils are growing in consumer goods applications, especially as partially hydrogenated replacements in both bakery and frying shortening applications. In this study various blends of soybean oil and/or high oleic soybean oil were analyzed for oxidative stability after processing of enzymatic interesterification using Lipozyme IM - a sn-1,3-specific lipase or chemical interesterification using sodium methoxide. The starting mixture and the products of interesterification were analyzed by mass spectrometer to identify triglycerol positional difference and then tested by Oil Stability Index (OSI, AOCS Cd 12b-92). The investigation showed that the vegetable product of enzymatic interesterification have a comparable oxidative stability to that of the product prepared from the starting mixture. In contract, the vegetable oils obtained from the products of chemical interesterification had inferior oxidative stability due to their fatty acid positioning on the triglycerol backbone.

**A Continuous Oil Treatment Device to Improve Fryer Oil Quality and Fry Life** Monoj K. Gupta*, *MG Edible Oil Consulting International, Inc., USA*

Frying oil degrades from the time the frying operation begins and it continues. In many operations the oil is filtered through filter media and/or treated with additives. A new continuous oil treatment device can slow down the oil degradation in the continuous frying process. The device reduces the oil decomposition products and improve the fry-life of the oil significantly.

## LOQ 4b / PHO 4: Phospholipids as Antioxidants and the Analysis of Their Oxidation in Industrial Applications and Complex Matrices

*Chairs: Nora Yang, Kalsec, Inc., USA; and Matthias Rebmann, Perimondo, USA*

**Impact of Phospholipids on Lipid Oxidation** Eric A. Decker*[1], Anuj G. Shanbhag[1], Gautam Samdani[1], and Leqi Cui[2], *[1]University of Massachusetts Amherst, USA; [2]Fuli School of Food Equipment Engineering and Science, Xi'an Jiaotong University, China*

Phospholipids are important minor components in edible oil that play a role in lipid oxidation however whether they are antioxidative or prooxidative is often controversial. Surface active phospholipids have intermediate hydrophilic-lipophilic balance values and can form association colloids in bulk oil. These association colloids can influence lipid oxidation since they create lipid-water interfaces where prooxidants and antioxidants can interact with fatty acids. In general, phosphatidylcholine produces reverse micelles and the water-oil interfaces created by these reserves micelles promotes lipid oxidation. Phospholipids have also been postulated to inhibit lipid oxidation by scavenging free radicals, chelating metals and forming antioxidative Maillard products. The role of these pathways on the ability of phospholipids to inhibit lipid oxidation is still not well understood. Phosphatidylethanolamine has been shown to inhibit lipid oxidation in the presence of tocopherols by regenerating oxidized tocopherols back into effective free radial scavenging antioxidants. The ability of phosphatidylethanolamine to regenerate tocopherols is dependent on the tocopherol homolog type and the physical properties of the food system. Unfortunately, use of phosphatidylethanolamine to inhibit lipid oxidation is limited by the high cost of purified compounds and by its low concentrations in most commercial lecithins.

**Marine Phospholipids: Oxidation Mechanisms and Analysis of Quality Deterioration** Charlotte Jacobsen*, *Technical University of Denmark, Denmark*

Recent research has shown that there are interesting differences in the nutritional benefits of marine phospholipids (PL) and fish oils. Some studies have documented a higher uptake of omega-3 fatty acids from marine PL such as krill oil into various tissues, when compared with the uptake from fish oil. For that reason there is an increasing interest in using marine PL for dietary supplements. Marine PL are rich in highly unsaturated omega-3 fatty acids and are therefore susceptible to lipid oxidation. However, the degradation reactions in marine PL are more complex than those in fish oils. For example, non-enzymatic browning reactions may occur between lipid oxidation products and primary amine group from phosphatidylethanolamine or amino acid residues present in marine PL. Therefore, marine PL contain Strecker aldehydes, pyrroles, oxypolymers etc. that may positively or negatively affect the oxidative stability and quality of marine PL. For that reason, classical techniques such as Peroxide Value and Anisidine Value are not useful for the evaluation of the quality of marine PL. These techniques may grossly underestimate lipid oxidation and other degradation reactions in marine PL. This presentation will give an overview of the oxidation mechanisms in marine PL and will discuss analytical methods recommended for the quality evaluation of marine PL. The presentation will give examples from research conducted in the author's lab on krill oil as well as emulsions with different types of marine PL.

**Incorporation of High-oleic and High-linoleic Lecithin Can Improve the Oxidative Stability of Vegetable Oils** Solmaz Alborzi[1], Matthias Rebmann[2], and Rohan V. Tikekar*[1], *[1]University of Maryland-College Park, USA; [2]Perimondo, USA*

Lecithin, a commonly used emulsifier contains diverse saturated and unsaturated fatty acids depending upon its source with little control over this composition. However, if its fatty acid composition can be tailored, it is possible to improve its sacrificial antioxidant activity and thereby improve the oxidative stability of vegetable oil it is typically incorporated in. With this hypothesis, we are investigating various lecithin samples with diverse fatty acid profiles for their ability to lower the oxidation of vegetable oils. Specifically, we are comparing novel high-oleic lecithin, and high-linoleic lecithin with conventional soy lecithin and egg lecithin. Vegetable oil is incorporated with 5% (w/v) lecithin and incubated at temperature of 120 °C for up to 3 h to simulate accelerated oxidation. Periodically samples are taken and absorbance is measured at 270 nm to detect the formation of conjugated trienes, a typical oxidation product. At 120 °C, vegetable oil without lecithin showed an increase in the absorbance from 0.41 to 1.43 during 3 h of incubation indicating significant oxidation. Addition of 5% high-oleic lecithin lowered the extent of oxidation where the absorbance increased from 0.37 to 1.1. Similarly, addition of high-linoleic lecithin also lowered the extent of oxidation where the absorbance increased from 0.4 to 0.98. Further investigation is underway to compare the conventional soy and egg lecithin samples, to perform more comprehensive analysis of oxidation products and develop mechanistic understanding. Success of this approach can result in rational design of lecithin to improve oxidative stability of vegetable oil.

**Controlled Singlet Oxygen Oxidation of Soybean Phospholipids** Jean-Francois Fabre*[1], Audrey Cassen[1], Romain Valentin[2], and Zéphirin Mouloungui[3], *[1]LCA UMR1010 INRA-INP/ENSIACET, France; [2]INRA, France; [3]Laboratoire de Chimie Agro-Industrielle, France*

Oxidation of phospholipids has a huge impact on their hydropathicity and the functional properties of vesicles formed in water. Using a controllable Xenon lamp (SUNTEST benchtop Xenon Instrument), soybean phosphatidylcholine in ethanol with different concentrations of a photosensitizer (methylene blue) was submitted to different irradiation powers and durations. At moderate conditions, this singlet oxygen oxidation mode induces the formation of hydroperoxidized phospholipids with conjugated insaturations. Spectrophotometric study and 1H, 13C and 31P NMR analysis allow the control of the chemical modification of the phospholipids according to the experimental design performed. The ratio between conjugated dienes and conjugated trienes depends mainly on the duration of irradiation and the concentration of the pigment. A granulometric and zeta potential study also indicates the high importance of this parameter on the stability and size of the vesicles formed when oxidized pigments are redispersed in distilled water. As revealed by Differential Scanning Calorimetry, water trapping abilities of oxidized phospholipids are also altered. Coarse-grained molecular dynamics can help to understand the different arrangements of phospholipids in water according to a gradual modification of their fatty acid chains. Combining this experimental and theoretical approach, a better tailoring of oxidized phospholipids could be reached.

## LOQ 5a: Frying Oils—Applications, Quality, and Chemistry

*Chairs: Rick Della Porta, Pepsico / Frito-Lay, USA; and Jill Moser, USDA, ARS, NCAUR, USA*

**The Advantage of Frying Foods in High Oleic Oils: Bridging the Intersection of Health and Functionality** Susan Knowlton*, *DuPont Company, Pioneer, USA*

Many conventional oils used in the US have a high polyunsaturated fatty acid content which makes them unsuitable for frying. Traditional methods used to stabilize these oils with additives and/or process changes are losing their attractiveness due to a consumer desire for pure and simple ingredients. As a result, high oleic versions of most oilseed crops have been created enabling use of these high stability oils for food service and industrial frying. These oils are expected to increase in use since they have the required stability, address many of today's consumer interests and are healthy alternatives to other oils. Results from frying studies and clinical trial data demonstrating effects on CVD risk markers based on diets which include fried foods will be presented.

**The NMR Analysis of Frying Oil: A Very Reliable Method for Assessment of Lipid Oxidation** Hong-Sik Hwang*[1], Jill Moser[1], and Sean Liu[2], *[1]USDA, ARS, NCAUR, USA; [2]USDA, ARS, USA*

There are many analytical methods developed for the assessment of lipid oxidation. However, one of the most challenging issues in analyzing oil oxidation is that there is lack of consistency in results obtained from different analytical methods. The major reason for the inconsistency is that most methods detect one kind of oxidation products while there are numerous oxidation products produced during the course of oxidation. Therefore, depending on which method is used, in other words, which oxidation product is detected, the result can be very different. For this reason, continuous efforts are being made to develop more reliable methods that can concomitantly detect many oxidation products. The 1H NMR method monitoring the changes in oil molecules had been developed in the late 1980's focusing on the lipid oxidation at relatively low temperatures and very few studies have been conducted for frying oil. We conducted a systematic study validating the reliability of the NMR method for frying using soybean oil and two different high-oleic oils. We evaluated correlations of the 1H NMR method with conventional methods including total polar compounds (TPC), polymerized triacylglycerols (PTAG) and the loss of polyunsaturated fatty acids (PUFAs) and observed very strong correlations. We found that the NMR method was very reliable, non-destructive, fast, and convenient analytical method to analyze the oxidation level of frying oil. In addition, some oxidation products found in the 1H NMR spectrum during frying with these oils will be discussed.

**Formation of 4-Hydroxy-2-trans-nonenal (HNE), a Toxic Aldehyde, in Thermally Treated Olive and Sunflower Oils** Jieyao Yuan*, and A. S. Csallany, *University of Minnesota, USA*

4-Hydroxy-2-trans-nonenal (HNE) is a toxic aldehyde produced in oils containing polyunsaturated fatty acid (PUFA) due to heat-induced lipid degradation. This study examined the effects of heating time, degree of unsaturation, as well as the antioxidant concentration on the formation of HNE in olive and sunflower oils at frying temperature. HNE concentrations in three different olive oil samples and two different sunflower oil samples heated for 0, 1, 3, and 5 hours at 185˚C were measured by high performance liquid chromatography. Fatty acid

distribution and antioxidant capacity of these five oils were also analyzed. Results showed that all oils, except extra virgin olive oil, had very low HNE concentrations (< 0.5 µg/g oil) before heating. After 5 hours of heating at 185˚C, HNE concentrations were increased to 17.98, 25.00, 12.76, 12.51, and 40.00 µg/g in the two light olive oils, extra virgin olive oil, high oleic sunflower oil, and regular sunflower oil, respectively. Extending heating time increased HNE formation in all tested oils. It related to their fatty acid distributions and antioxidant capacities. Regular sunflower oil contained high levels of linoleic acid (59.6%), a precursor of HNE, was more susceptible to degradation than high oleic sunflower oil and olive oils which contained only 6% to 8% linoleic acid. Surprisingly, the HNE formation of extra virgin olive oil increased slowly and remained the same after 3 to 5 hours of heating. It was possibly due to its high antioxidant capacity (3.88 µmol of Trolox/g oils) which retarded the HNE formation.

**Quantitative Determination of Epoxy-and Hydroxy Fatty Acids in Edible Oils** Wei Xia*, and Suzanne M. Budge, *Dalhousie University, Canada*

Epoxy-and hydroxy fatty acids have been considered as important lipid oxidation products formed from triacylglycerols. The determinations of these two classes of compounds had been achieved with hydrogenation, which resulted in a loss of information regarding double bonds and their positions. In this study, we report a sensitive method to simultaneously determine the concentrations of epoxy- and hydroxy-fatty acids without hydrogenation. First, triacylglycerols were converted into fatty acid methyl esters (FAME) using transmethylation procedures and then subjected to a three-step solid phase extraction (SPE), where epoxy FAME and hydroxy FAME were separated into two fractions to avoid co-elutions in GC chromatograms. The epoxy fatty acids were identified using GC-MS in their FAME forms, while the hydroxy FAME were silylated into their TMS ethers for GC-MS identification. Based on their mass spectra and retention times, 6 epoxy fatty acids and 16 hydroxy fatty acids were identified in canola oil and sunflower oil, including their cis- and trans- isomers. GC-FID was then employed to quantitatively analyze their concentrations in the two fractions with methyl nonadecanoate (C19:0) as an internal standard. Response factors were determined for epoxy FAME and TMS ethers of hydroxy FAME using synthetic methyl epoxystearate and commercial hydroxy FAME standards. The limits of detection for epoxy FAME and hydroxy FAME were 1.5 µg/mL and 0.3 µg/mL, respectively.

## LOQ 5b: *Trans* Fats Replacements and Low Saturated Fats: Impact on Shelf-life, Oxidative Stability, and Application

*Chairs: Michelle Peitz, Archer Daniels Midland Co., USA; and Bingcan Chen, North Dakota State University, USA*

**Substitution of Vegetable Shortening with Mixtures of more Unsaturated Oils in Pie Crust and Its Effect on Sensory and Oxidative Stability** Denis Xie*, Robin Boyle, and Anna Cheely, *Kalsec, Inc., USA*

The removal of partially hydrogenated oils (PHO) from the GRAS list by the FDA has led to a search of replacement fats/oils that are low in trans fatty acids (TFA), higher in unsaturated fatty acids, and yet do not significantly change the sensory attributes and oxidative stability. One product that is affected by this change is pie crust. Pie crust is made by creating a layered effect of the fat and flour in order to create a flaky, yet tender texture. Vegetable shortening was compared to lard and a combination of high oleic safflower oil (HSO), avocado oil, and peanut oil in different ratios. The crust was evaluated by sensory, and by tracking oxidation markers via GC/MS headspace and peroxide value of extracted fat.

**Role of Solid Fat on Lipid Oxidation in a Model Cracker System** Thanh P. Vu*, Lili He, David J. McClements, and Eric A. Decker, *University of Massachusetts Amherst, USA*

Crackers and other low moisture foods represent a potential health concern due to their high content of saturated fats, which increase a risk of coronary heart disease. Traditionally crackers are made from semi solid fats that are high in saturated fatty acids to provide structure. The objective of this study was to investigate the role of solid fat content (SFC) on the oxidative stability of a model cracker system. Oxidative stability was evaluated by lipid hydroperoxide and headspace hexanal measurements. Our results showed that SFC had an influence on oxidation stability of crackers. Oxidative stabilities of commercially available oils were in order: palm shortening (SFC at 55 °C = 6.3 – 8.7%) > interesterified soybean oil (SFC at 55 °C = 13.8 - 14.6%) > liquid soybean oil = canola/cotton blend (SFC at 55 °C = 0%). In second condition, different fats were used to produce varied SFCs but fixed 18:2 at 20%. Tocopherol homologs were adjusted to produce similar tocopherol profiles. Similar to the previous experiment, oxidative stabilities decreased in an order SFC at 55 °C = 12.9 - 13.5% > SFC at 55 °C = 24.4 - 25.4% > SFC at 55 °C = 31.1 - 32.7%. However, in the third test, fully hydrogenated soybean oil was blended with liquid soybean oil to produce similar fatty acid profile with interesterified soybean oil. Tocopherol homologs were also adjusted to produce similar tocopherol profiles. The oxidative stability in the third test could not be predicted by SFC.

**Comparison of Thermal Stability and Nutritional Quality of Palm Oil and Other Frying Oil** Yin Mee Thang*, Nur Azwani Ab Karim, Kawsalyavathi Kuppan, Haniza Ahmad, Maslia Manja Badrul Zaman, Sue Hern Haw, Mohd Fadlly Jumadi, and Ahmadilfitri Md Noor, *Sime Darby Research Sdn Bhd, Malaysia*

Palm oil which has a relatively high content of saturated fat has been wrongly perceived that is bad for consumption. It is an undeniable fact that the stability of an edible oil product will be affected if the levels of the saturated fatty acids are reduced. Therefore, the main goal of this research work was to investigate the thermal stability and nutritional characteristics of palm oil as compared

to various commercially available soft oils such as soybean, sunflower, and canola oil under intermittent frying conditions. Our findings showed that premium quality palm oil with free fatty acid levels of less than 0.05%, peroxide value of less than 1 meq/kg, anisidine value of 2, polar content of less than 5%, and polymer content of 0.2% is the most resistant to hydrolysis. The smoke point of all frying oils gradually dropped, tocopherol levels of all frying oils declined and at the same time, the total polar compound (TPC) and polymer levels increased in all oils over the course of frying. Nevertheless, the lowest TPC level was observed in the case of premium quality palm oil (approximately 10.4% after 40 frying cycles) as compared to more than 15% recorded with other soft oils with same frying cycles. The incremental increase of polymers in premium quality palm oil per frying cycle averages around 7.8%, which is quite low as compared to 15% for soft oils. In conclusion, this work confirmed that frying performance of premium quality palm oil is better than the other soft oils.

**Oxidative Stability Impact of Nitrogen Addition Approach on Edible Oils** Jacob E. Maynard*, Joshua Tuinstra, and Roger Daniels, *Stratas Foods, USA*

Vegetable oils are utilized singly or as components of blends in both bakery and frying shortenings. A critical determinant of achieving optimal oil shelf life is to minimize autoxidation reaction sites (eg. Degree of unsaturation) and exposure to the principle driver of autoxidation, oxygen. In this study refined, bleached and deodorized edible oils with varying levels of mono- and poly-unsaturates were subjected to one of three nitrogen treatments; Nitrogen Free (NF), Nitrogen Overlay (NO), and Nitrogen Infused (NI), stored in a covered light absent container and assessed for oxidative stability as measured by peroxide value, p-anisidine value, and oxidative stability index for 30 days. The results indicate that the nitrogen addition approach positively impacted the measures of oxidative stability during the early phases of the study yet were less effective during the later stages of the timed study indicating the need to ensure control of bulk oil storage as it relates to composition, temperature and storage time.

## LOQ-P: Lipid Oxidation and Quality Poster Session

*Chairs: Shawn Pan, Bunge North America, USA; and Hans-Jürgen Wille, Consultant, Switzerland*

1. **Assessment of Extra Virgin Olive Oil Quality Using Portable Mid-infrared Spectroscopy and Multivariate Analysis** Didem P. Aykas*, and Luis E. Rodriguez-Saona, *The Ohio State University, USA*

Extra virgin olive oil (EVOO) has become a major component of the American diet, consuming 320,00 tons of EVOO in 2015. The EVOO has distinct sensory and nutritional characteristics. To assess EVOO quality German Fat and Oil Society (DGF) developed 1,2-diacylglycerol (DAGs) and pyropheophytins (PPP) tests. Fresh EVOO contains a high amount of DAGs and PPP and according to Australian Olive Association's (AOA) EVOO standards; fresh EVOO should include DAGs ≥40% and PPP ≤15%. Current analytical techniques to evaluate the quality of EVOO rely on chromatographic methods that are time-consuming, expensive, and labor-intensive. The aim of this study is to develop a rapid and robust technique for rapid (~1min) quantification of DAGs and PPP in EVOO by using field-deployable portable infrared sensor combined with a multivariate analysis. EVOO samples (n=114) were kindly provided by Borsa Laboratories, a Turkish contract laboratory that tests the quality of exported EVOO. The spectra of a drop of EVOO were directly collected using a portable FT-IR with a temperature controlled (65°C) crystal and partial least squares regression (PLSR) was used to analyze the spectral data. Gas chromatography (GC) and high-performance liquid chromatography (HPLC) were used as a reference analysis technique to determine DGAs and PPP content, respectively in EVOOs. PLSR models showed excellent correlation (Rpred>0.92) between the mid-infrared spectrometer predicted values and the reference values, and low standard errors of cross-validation (SECV). Portable IR spectrometer offers the olive oil industry a simultaneous, simple and high throughput method to predict chemical characteristics of EVOO.

2. **Effect of Crushing Speed on Extraction Efficiency and Quality of Olive Oil Obtained from Super-high-density Arbosana Cultivar** Juan J. Polari*[1], David Garci-Aguirre Garci-Aguirre[2], and Selina Wang[3], *[1]University of California, Davis, USA; [2]Corto Olive Co., USA; [3]UC Davis Olive Center, USA*

Crushing is a key step during olive oil extraction. This physical process not only breaks the fruit's tissues to release the oil from the vegetal cell vacuoles but also initiates the enzymatic reactions that affect the volatile profile and phenolic compounds in the final product. While there are different options for commercial crushers, hammer mills are the most widely used in modern continuous facilities due to its robustness and high throughput. In the present work, the effect of hammer mill speed on extraction yield and olive oil quality in an industrial facility were assessed. Our results showed that increasing the rotor speed from 2400 RPM to 3600 RPM led to an increase of 1.2% in yield, while conserving quality parameters (free fatty acids, peroxide value, UV absorbance, diacylglycerols (DAGs) and pyropheophytins (PPP)). In addition, total phenols content in the oil increased from 228 ppm to 270 ppm. Sensory analysis of the oils showed slightly more bitterness and pungency with increased rotor speed while fruitiness was unaffected. Based our findings, faster crushing speed of the hammer mill appears to improve extraction efficiency. Processors can also tailor the phenolic contents and sensory attributes of olive oil they desire by modulating the crushing speed.

3. **Protection of Beta-carotene in Emulsion-based Delivery Systems Using Antioxidant Interfacial Complexes: Catechin-egg White Protein Conjugates** Luping Gu*, and David J. McClements, *University of Massachusetts Amherst, USA*

The aim of the present study was to fabricate catechin-egg white protein (CT-EWP) conjugates as novel food-grade antioxidant emulsifiers designed to improve the physicochemical stability of beta-carotene (BC) emulsions. CT-EWP conjugates were synthesized using free radical grafting and conjugation formation was confirmed by electrophoresis and liquid chromatography-mass spectrometry. The physicochemical stability of BC emulsions was characterized by measuring changes in particle size, ζ-potential and BC retention. Changes in particle size and ζ-potential were quicker at 37 °C than at 4 and 25 °C, however no creaming or oiling-off were observed at any of the storage temperatures, which suggested that all the emulsions were physically stable during the 30-days storage period. Compared to emulsions stabilized by EWP or CT+EWP physical mixtures (no conjugation), CT-EWP conjugate-stabilized emulsions exhibited greater resistance to environmental stresses, such as thermal processing and high ionic strengths, which was attributed to a stronger steric repulsion between the oil droplets. CT-EWP conjugates also significantly reduced the degradation rate of BC in emulsions during storage, which was attributed to their strong antioxidant and interfacial activities. These results indicate that CT-EWP conjugates can be utilized to develop food-grade delivery systems to protect chemically labile lipophilic bioactive compounds.

4. **The Impact of Diacylglycerols on Lipid Oxidation in Oils Containing Association Colloids** Mizue Ouchi*[1], David J. McClements[2], and Eric A. Decker[2], *[1]Kao Corporation, Japan/University of Massachusetts Amherst, USA; [2]University of Massachusetts Amherst, USA*

Association colloids formed by the various kinds of surface active components have an important role on the oxidation stability of oils. Diacylglycerols (DAG), one of the minor components in refined oils, have different chemical and physical properties from triacylglycerols (TAG) due to its chemical structure. There are several studies on the influence of DAG on the oxidation stability of bulk oils but these studies are inconsistent reporting DAG as both prooxidants and antioxidants. The objective of the study is to find out the impact of DAG on lipid oxidation in the bulk oil containing association colloids made from phosphatidylcholine. This research focused on how the DAG affects the oxidative stability of stripped oil with and without reverse micelle. The critical micelle concentration (CMC) of phosphatidylcholine increased as the DAG concentration increased suggesting that DAGs where incorporated into the reverse micelles. The oxidation rate of DAG containing oil was almost the same as TAG without reverse micelles while it was slightly different in the presence of reverse micelles. To reveal the influence of minor components on lipid oxidation would provide us useful knowledge to improve oxidative stability.

5. **Characterization and Determination of Walnut Oils from China** Pan Gao*, Xingguo Wang, and Qingzhe Jin, *Jiangnan University, China*

A total of 25 walnut oil samples from different geographical areas (Shanxi, Shandong, Liaoning, Hebei, Sichuan, Shaanxi, Hubei, Yunnan, Xinjiang, Tibet and Heilongjiang) from china were selected. The higher percentages of fatty acids and triacylglycerides , as determined by Gas Chromatography (GC).The fatty acids corresponded to palmitic (2.96-8.25 %), oleic (15.07 - 24.71 %), linoleic (51.21-68.97 %) and linolenic (6.67-12.60 %), and the main triacylglycerides were LLL (17.069-39.739 %), followed by PLL (5.881-14.983

%), OLL (8.662-15.462 %), OLLn (11.531-24.179 %) and LLLn (7.166-26.443 %). In addition, high amounts of micronutrients, including sterol, squalene, tocopherol and total phenolic content were also found in walnut oils by high performance liquid chromatography (HPLC) and Gas Chromatography-Mass Spectrometer (GC-MS) ranged from 453 to 1492, 125 to 1806, 159 to 2579 and 26 to 203 mg/kg among different varieties, respectively. The measurement of the antioxidant activity of the walnut oils was achieved by their ability to scavenge the free stable 2,2-diphenyl-1-picrylhydrazyl radical (DPPH ). A positive correlation was found between DPPH radical with total phenolic content (r=0.740) and linolenic (r=0.494).

6. **Relationship Between Malondialdehyde Formation and Reduction of Polyunsaturated Fatty Acids in Vegetable Oil** Ma Lukai*, and Liu Guoqin, *School of Food Science and Engineering, South China University of Technology, China*

Malondialdehyde (MDA), considered to derive from the secondary oxidation of polyunsaturated fatty acids (PUFA), has been extensively suggested to have genotoxic and mutagenic properties, and has additionally been associated with cardiovascular disease. The more consumption of vegetable oils rich in PUFA, the higher risk of MDA intake. In this study, the kinetic formation of MDA in palm oil (PO), corn oil (CO), rapeseed oil (RO), camellia oil (CLO) and linseed oil (LO) was investigated, methyl oleate, methyl linoleate, and methyl linolenate were chosen to verify the origin of MDA. The MDA content was determined by high performance liquid chromatograph (HPLC) after derivatization with 2-thiobarbituric acid (TBA). The results showed that: during the 30 days' Schaal oven test at 60 °C, the acid value (AV), peroxide value (POV), p-anisidine value (p-AV) as well as the MDA content increased gradually, while iodine value (IV) and PUFA contents decreased with time, which indicated the reduction of unsaturation degree of vegetable oil. The content of MDA ranked as: LO >CO >RO >PO >CLO, PO and CLO had a much lower MDA content than other test oils. In conclusion, MDA content was lower in PO and CLO which consisted of small amounts of PUFA, and vegetable oils with a higher degree of unsaturation certainly exhibited great susceptibility to MDA formation. Additionally, the formation of MDA was mainly caused by the oxidation of esters of linoleate acid and linolenate acid, rather than by the oxidation of oleate acid esters. This work is an important foundation for further study on formation mechanisms and inhibition/elimination methods of MDA in vegetable oils.

7. **Effect of Oil Oxidation on Glycidyl Ester Formation Involved in Radical-mediated Mechanisms in Refined Oil** Weiwei Cheng*[1], and Liu Guoqin[2], *[1]South China University of Technology, China; [2]School of Food Science and Engineering, South China University of Technology, China*

Glycidyl esters (GEs), a new class of oil-processing contaminant, possess potential genotoxic carcinogenicity and are commonly detected in refined oils and oil-based food products, even in infant formulas. Therefore, the formation and inhibition of GEs in refined oil draw increasing attention from oil researchers and industry. The aim of this research is to investigate the effect of oil oxidation on glycidyl fatty acid ester (GE) formation and the free radical-mediated mechanisms involving the recognition of cyclic acyloxonium free radical intermediate (CAFRI) for GE formation in both model oil (palm oil, camellia oil, soybean oil, and linseed oil) system and chemical model (dipalmitin and methyl linoleate) system heated at 200 °C. The results showed that oil oxidation exhibited a positive correlation with GE formation. Based on the monitoring of cyclic acyloxonium and ester carbonyl group by Fourier

transform infrared spectroscopy (FTIR), the promotion of Fe3+ and the inhibition of antioxidants (tert-butylhydroquinone and α-tocopherol) for GE formation not only through oil oxidation but also through directly affecting the formation of cyclic acyloxonium intermediate. Additionally, a quadrupole-time of flight (Q-TOF) MS/MS measurement was conducted and measured the presence of radical adduct captured by 5,5-dimethylpyrroline-N-oxide, proving the formation of CAFRI. Thus, the possible influencing mechanisms are that the free radical generated in oil oxidation can transfer to dipalmitin and promote CAFRI formation. Fe3+ and antioxidants can catalyze free radical generation and scavenge free radical, respectively, and therefore also directly affect CAFRI formation.

8. **A Mathematical Method for Determining the Appropriate Amount of Stigmasterol Added in Soybean Oil** Li Xu*[1], Li Yongru[2], Ruijie Liu[3], Zhao Chenwei[2], Qingzhe Jin[4], and Xingguo Wang[4],*[1]School of Food Science and Technology, Jiangnan University, China; [2]State Key Laboratory of Food Science and Technology, School of Food Science and Technology, Jiangnan University, China; [3]Jiangnan University/Cornell University, China; [4]Jiangnan University, China*

Phytosterols and their oxidation products (POPs) have become increasingly investigated in recent years with respect to their beneficial and harmful influence. The appropriate amount of nutritional phytosterols should be added in edible oil since they can be oxidized under heating condition to give POPs, known as toxic effect. The paper studied the degradation of stigmasterol in soybean oil during 10 day heating (60 ºC), 9 hour heating (180 ºC) and 9 hour frying (180 ºC) treatment. The formation and further degradation of POPs was analyzed by GC-MS. The results showed that POPs increased their concentration with the heating time and the stigmasterol added in oil. A POPs formation probability was revealed according to the following decreasing order: 7-keto > 7β-hydroxy ≈ 5,6β-epoxy > 5,6α-epoxy > triol > 7α-hydroxy. A mathematical model was established based on chemical (acid value and peroxide value), nutritional (iodine value and phytosterols content) and hazardous (POPs concentration) indices with the method of weight analysis and nondimensionalization. The comprehensive evaluation index illustrated the rule of first descended then rose with the turning point of 6.81‰ stigmasterol in oil. In order to keep the balance of chemical, nutritional and hazardous factors in three different conditions, the stigmasterol concentration of 11.45‰ was recommended in soybean oil.

9. **Physicochemical Stability of Flaxseed Oil-in-Water Emulsions Fabricated from Sunflower Lecithins: Impact of Varying Phospholipid Type** Li Liang*[1], Fang Chen[2], Xingguo Wang[3], Qingzhe Jin[3], Eric A. Decker[4], and David J. McClements[4],*[1]State Key Laboratory of Food Science and Technology, School of Food Science and Technology, Jiangnan University, China; [2]School of Public Health, Nanchang University, China; [3]Jiangnan University, China; [4]University of Massachusetts Amherst, USA*

There is great interest in the formulation of plant-based foods enriched with bioactive components, such as polyunsaturated fatty acids. This study evaluated the impact of sunflower phospholipid type on the formation and stability of flaxseed oil-in-water emulsions. In particular, two sunflower lecithins (lecithins 50 and 90) with different phosphatidylcholine (PC) levels (59 and 90%, respectively) were used in varying ratios to form the emulsions. The droplet size, ζ-potential, physical appearance, microstructure, and oxidative stability of the emulsions was then measured. Both the physical and chemical stability of the emulsions

increased as the PC content of the lecithin blends decreased. The oxidative stability of emulsions formulated using lecithins 50 was better than emulsions formulated using synthetic surfactants (SDS or Tween 20). These results are interpreted in terms of the impact of emulsifier type on the colloidal interactions between the oil droplets, and the molecular interactions between pro-oxidants and oil droplet surfaces. Overall, this study indicates that the interfacial properties of oil droplets can be engineered using natural plant-based phospholipids to improve their physical and oxidative stability, which may be important for the development of emulsion-based delivery systems for bioactive lipids.

10. **Oxidative Stability of Edible Oils Containing Different Types of Omega-3 Fatty Acids** Marina S. Nogueira*[1], and Inar A. Castro[2], *[1]Dept. of Food and Experimental Nutrition, Faculty of Pharmaceutical Sciences, University of São Paulo, Brazil; [2]University of Sao Paulo, Brazil*

Omega 3 fatty acids (N3 FA) can form potentially toxic secondary products depending on the storage conditions. Thus, the objective of this study was to identify chemical markers formed from the oxidation of the major N3 FA present in edible oils while simulating conditions for transporting, storing and consuming. Fish, Algae, Echium and Flaxseed oils were selected as the sources of eicosapentaenoic, hexadocosaenoic, steraridonic and α-linoleic acids, respectively. Fresh samples were obtained just after the oils extraction (F), kept in their original bottle at 50o C for 2 days to mimic transportation (T) and maintained an additional 30 days at 25oC to simulate storage (S). Finally, the bottles were opened and kept at 25oC for 30 days to simulate the conditions during domestic consumption (C). Results showed that oils were stable until the end of storage, except for Fish oil that presented an increase of hydroperoxides and TBARS values. Oxidation also increased according to the peroxidability index, showing a linear correlation with hydroperoxide ($r = + 0.86$; $p<0.001$) and TBARS ($r = +0.77$; $p= 0.006$). The presence of tocopherols clearly contributed to the stability of algae oil. Among the volatile compounds, nonanal, (E)-2-undecenal and undecanal were present in a higher amount and could be used as chemical markers of n3 FA high commercial oils oxidation. Fish oil was the most susceptible sample and must be protected from oxidative damage since its extraction, while the other oils must be kept under freezing only after the bottles are opened.

11. **Use of Oxitest for Testing Food Quality of Tuna Fish Fillet in Olive Oil During Storage** Paola Ornaghi*[1], Monia Scarsi[2], Stefano Casiraghi[2], Antonella Cavazza[3], Chiari Bignardi[3], Carmen Lagana[4], Paola Salvadeo[3], Claudio Corradini[3], and Stefani Corti[4], *[1]VELP Scientifica S.r.l., Italy; [2]VELP Scientific, Inc., USA; [3]Universita degli Studi di Parma, Italy; [4]VELP Scientifica, Italy*

Oxitest (VELP Scientifica, Italy) is a reactor based on the use of high temperature and over-pressure of oxygen that allows to easily measure a sample oxidative stability by accelerating the oxidation process. It has been shown to be a very useful tool for testing many types of food. In this work, the use of Oxitest to test food quality during storage was explored, evaluating its usefulness for the assessment of the shelf-life of food stored in glass and in metallic cans. Tuna fish in olive oil was chosen as food model to measure oxidative stability. Samples stored for different periods were submitted to analysis. Olive oil is a largely consumed product in the Mediterranean diet, responsible for several positive health benefits. Its shelf-life is limited since important changes in the bioactive compounds are known to occur during storage, leading to a loss of quality in terms of organoleptic and also healthy properties. The main changes observed are linked to the lipidic and

polyphenolic fraction, and involve oxidative and hydrolytic degradation. Results showed a progressive decrease of the olive oil stability accompanied by a parallel increase of tuna fish oxidative stability with time. It can be supposed that the antioxidant compounds occurring in olive oil exert a protective effect on the soaked tuna fish fillets. An interesting conclusion is that tuna fillet shelf-life is prolonged after its storage in olive oil, and even increases its quality, in terms of oxidative stability.

12. **Effects of Filtration on Chemical Properties and Oxidative Stability of Turkish Monovarietal Olive Oils** Esmaeil Ghanberi Shendi[1], Dilek Sivri Özay*[1], Mücahit Özkaya[2], and Feyza Üstünel[3], *[1]Hacettepe University, Turkey; [2]Dept. of Horticulture, Ankara University, Turkey; [3]Doğal Ürünler Tur. Tic. San. A.Ş., Turkey*

The mobile olive oil processing unit (MOOPU) was used to produce monocultivar extra virgin olive oil (MEVOO) extracted from a local olive cultivar called as “Beylik”. In addition to quality characteristics, some minor and major components of the MEVOO have been determined before and after filtration and the changes were monitored during storage for 12 months. Free fatty acid, peroxide and UV absorbance values, moisture content and fatty acid composition were performed according to the method of International Olive Council (IOC). The total phenols were determined by the Folin&Ciocalteu reagent. Methanolic extracts of MEVOO obtained by solid phase extraction (SPE) and used for total phenols and phenol profiles analyses. Phenolic and tocopherols were detected by ultra high-performance liquid chromatography (UHPLC). Results showed filtered and unfiltered olive oil samples can be classified as extra virgin olive oil (EVOO) as declared by IOC trade standards. Free fatty acid value of both filtered and unfiltered Beylik were 0.2% at the end of storage. Generally color changed from green to yellow. Filtered MEVOO had higher α-Tocopherol and β-Tocopherol contents than unfiltered sample. Total phenol content of unfiltered MEVOO (696 ppm) was higher than filtered one. Luteolin was the most abundant phenolic compounds in both samples. Unfiltered sample had higher oleuropein content. The amount of oleuropein and other phenolics decreased during storage, while tyrosol, 4-hydroxy benzoic acid and hydroxy tyrosol contents increased. Results showed that filtration and storage had significant effects on chemical and physical properties of MEVOO.

13. **Value Utilization of Discarded Fish Livers for Production of Omega-3 Rich Oil** Ann-Dorit M. Sørensen*[1], Nina Skall Nielsen[2], and Charlotte Jacobsen[1], *[1]Technical University of Denmark, Denmark; [2]National Food Institute, Technical University of Denmark, Denmark*

The intake of long chain (LC) omega-3 polyunsaturated fatty acids (PUFAs), especially eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), has been associated with several health beneficial effects. Thus, there is a demand for new methods to obtain high quality omega-3 rich oils and applications with omega-3 to increase the population’s intake of the healthy omega-3 LC PUFAs. Most of the fish caught in Denmark are slaughtered and rinsed immediately after catch, when the fishing vessel is still at the sea. When the fish is rinsed, the liver is discarded in to the sea. However, this practice has now been prohibited in the new EU legislation. Liver from fish has a high content of omega-3 LC PUFAs, i.e. EPA and DHA. This liver could be stored and used for production of oil rich in omega-3 and thus, create value from waste material. The quality of the livers will affect the quality of the oil produced. Thus, a good quality of the waste material has to be preserved from

catch to oil production. Parameters that can affect the quality of the liver from catch to oil production are storage condition and initial oxidation stage. The aim of this study was to evaluate the effect of storage conditions (iced and -18 oC) on board the fishing vessel on the oxidative quality of different cod species. Additionally, a systematic evaluation of seasonal variation in oil content, oxidation status and fatty acid composition was performed on different cod species.

14. **Natural Antioxidants Derived from Seaweed Material** Ditte B. Hermund*[1], Randi Neerup[2], Susan Holdt[3], and Charlotte Jacobsen[1], *[1]Technical University of Denmark, Denmark; [2]Danish Technological Institute, Denmark; [3]National Food Institute, Technical University of Denmark, Denmark*

Natural antioxidants derived from seaweed have a high content of bioactive components with potential for improving oxidative stability of lipids in food systems. Furthermore, some of these compounds can be used as functional ingredients in skin care products, against aging and inflammation of the skin. Seaweed cultivation is a rather new discipline in Denmark, and the utilization potential of the biomass is being studied. In a new research project the value chain from seaweed cultivation to final product will be investigated and implemented. In this project bioactive compounds from the seaweed biomass are extracted and high value products for the food and cosmetic industry are developed. The poster will contain the background and aim of the work and preliminary results on antioxidants derived from seaweed.

15. **Oxidative and Physical Stability of Fish Oil-in-Water Emulsions Stabilized with Sodium Caseinate and DATEM** Betül Yesiltas*[1], Pedro J. Garcia-Moreno[2], Ann-Dorit M. Sørensen[2], and Charlotte Jacobsen[2], *[1]National Food Institute, Technical University of Denmark, Denmark; [2]Technical University of Denmark, Denmark*

An optimization study was carried out in order to evaluate the physical and oxidative stability of high fat (50-70%) omega-3 delivery fish oil-in-water emulsions stabilized with combinations of sodium caseinate (CAS) and commercial diacetyl tartaric acid ester of mono- and diglycerides (DATEM). It is hypothesized that combined use of emulsifiers and surfactants provides less viscous emulsions which makes it easier to incorporate delivery emulsion into food products. The influence of 3 factors related to emulsion composition (fish oil content: 50, 60 and 70%; total amount of CAS and DATEM: 1.4, 2.1 and 2.8 %; and ratio between CAS and DATEM: 0.4, 1.2 and 2) on physical (droplet size, viscosity and zeta potential) and oxidative (peroxide value and volatile oxidation products) parameters were evaluated. After executing a Box-Behnken design, statistical modelling and optimization was performed. The best performing recipe was determined to be 70% fish oil, 2.8% total emulsifier and a ratio of 2 for the combination of CAS:DATEM. Results of optimization study showed that creaming decreased with increasing fish oil and total emulsifier content, whereas, droplet size was affected by all variables. Viscosity decreased with the decreasing fish oil content, however no significant effect of variables was found on peroxide value. Volatile compounds were formed in lower amounts with the increased ratio of CAS to DATEM, confirming good antioxidative effects of CAS.

16. **Comparison of Antioxidant Activities of Fucoidan Isolated from Four Species of Brown Algae from Danish Coast** Sabeena Farvin Koduvayur Habeebullah[1], Surendraraj Alagarsamy[2], and Charlotte Jacobsen*[3], *[1]Environmental and Life Science Research Center, Kuwait Institute for Scientific Research, Kuwait; [2]Kuwait Institute for*

*Scientific Research, Kuwait; [3]Technical University of Denmark, Denmark*

Fucoidans are fucose-rich sulfated polysaccharides found in brown macroalgae. These polysaccharides differ in their sugar backbone composition, structure and sulfation patterns according to the species of origin and the extraction techniques used. In the present work Fucoidan was extracted from four species of brown algae viz, F. vesiculosus, F. serratus, A. nodosum and L. digitata. The extracted fucoidan was tested for their antioxidant activity both in in vitro assays and in 5% oil-in-water emulsion and were compared with Sigma@Fucoidan. F.serratus showed high iron chelating activity and antioxidant activity in liposomes. Sigma@Fucoidan showed high reducing power and radical scavenging activity. Surprisingly when tested in 5% fish oil in water emulsion, fucoidan from L.digitata showed high protection against lipid oxidation in spite of the low antioxidant activity in the in vitro assays. The fucoidans from L.digitata have the highest content of sulfate when compared to other fucoidans and this may be the reason for the high antioxidant activity in 5% oil in water emulsion.

18. **The Role of Phenolic Compounds Extracted from Germinated Pulse Crops on the Oxidative Stability of Oil-in-Water Emulsions** Minwei Xu*, and Bingcan Chen, *North Dakota State University, USA*

Germinated pulse crops produced from North Dakota, including chickpea, yellow pea and lentil, were used as the substrate for extraction of natural antioxidants. Dynamic changes of phenolic compounds during germination were recorded. Phenolic compounds in free and bound form were extracted at different germination time. The in vitro antioxidative activity of phenolic fractions were characterized using 2, 2-diphenyl-1-picrylhydrazyl (DPPH) and 2, 2'-azinobis 3-ethylbenzothiazoline-6-sulfonic acid (ABTS) radical scavenging capacity assay, and oxygen radical absorbance capacity (ORAC) assay. The antioxidant role of free and bound phenolic compounds was further investigated in stripped flaxseed oil-in-water emulsions stabilized by tween 20 at pH 3 and 7 on the basis of lipid hydroperoxides and hexanal formation. The impact of free and bound phenolic fractions on the physical properties of emulsions was also assessed. The results suggested that germination time is critical for phenolic compounds production. The form of phenolic compounds, i.e., free and bound, will influence the antioxidative activity both in vitro assay and in real food systems. The findings could be applied to develop novel natural antioxidants to effectively prevent lipid oxidation.

19. **Influence of Food Additives and pH on the Oxidative Stability of Crackers** Thanh P. Vu*, Lili He, David J. McClements, and Eric A. Decker, *University of Massachusetts Amherst, USA*

Crackers are low moisture food and one of the top saturated fat contributors in American diet. Understanding lipid oxidation mechanism in cracker system helps to develop strategies to replace saturated fat by unsaturated fat and, at the same time, prolong shelf-life and reduce food waste. To get a better understanding of factors influencing lipid oxidation in a model cracker system, gluten was added and the pH was varied from 4.2 to 9.3. Crackers were incubated at 55°C in the dark and maintained at a water activity of 0.22 or 0.68 during incubation. Oxidation was evaluated based on hydroperoxides and hexanal lag phases. Gluten was added at concentrations of 0, 2.5, 5, and 10% (endogenous gluten was 10%). At low water activity (0.22), gluten showed no effect on lipid oxidation in cracker; however, at intermediate water activity (0.68), high gluten concentrations resulted in slightly longer lag phases. Low pH promoted lipid oxidation, presumably due to enhancing mobility of iron. Crackers were most

oxidative stable when pH values were close to pH 7.

21. **The Sensorial Properties of Olive Oils Extracted from Edremit Cultivars Harvested in Different Regions** Alev Y. Aydar*, *Manisa Celal Bayar University, Turkey*

In this study, the effect of region on sensorial properties of olive oils extracted from Edremit variety olives were studied. The sensory evaluations were performed by 10 trained panelists. Panelists evaluated the overall acceptability of each oil sample (taking into account any positive and negative taste), using a numerical scale 1–5 (1 = not acceptable, 5 = extremely good), as well as bitterness and fruity etc. (1 = no bitterness, 5 = extremely bitter). The panelists perceived no negative attributes such as rancid or musty-humid taste for all oils extracted from Edremit varieties harvested in Mut and Akhisar regions. The oils coded as 133 and 254 were evaluated fruitier than other oils, however pungent and bitter properties of oils were not significantly different. Oils extracted by 10 min ultrasound at 50 °C and 40 min of malaxation (sample:154) were evaluated as lowest fruity oils. After 12 month storage of olive oils at room temperature, all olive oils extracted by ultrasound showed sensory profiles belonging to EVOO category. Therefore, it can be concluded ultrasound had positive effect on sensory properties of oil not only after extraction but also after long term storage.

22. **Novel Method for Fast and Straightforward Determination of the Oxidation Stability of Fats and Oils** Carolin Edinger*, *Anton Paar ProveTec GmbH, Germany*

The quality of fats and oils strongly depends on their oxidation stability. In this contribution a new method for evaluating the oxidation stability by determining the induction period is introduced. Under accelerated conditions (elevated temperature and pure oxygen pressure) a sample of 5 mL is examined in a sealed stainless steel test chamber. Typical conditions of the method are temperatures between 120 °C – 140 °C and an initial oxygen pressure of 700 kPa. Application of these conditions allows for initiation of a rapid oxidation process, which is monitored by recording the pressure until a predefined pressure drop. It was found that the elapsed time until the pressure drop is directly related to the oxidation stability of the sample. In order to compare our new approach to the AOCS method Cd12b_92_13, we have studied seventeen edible oils at 120 °C and 140 °C. Comparison to the data collected using the abovementioned AOCS method reveals a linear relationship. Due to defined oxygen volume in the closed test chamber, the oxygen consumption can be calculated. Furthermore, we found Arrhenius behaviour with regard to the applied temperature, enabling the user to determine the activation energy of a specific oxidation process. The significantly reduced measurement time and a high repeatability of the method represents the major advantages of our method, allowing for quick and direct measurement of the oxidation stability for research, process and test bench control.

23. **Effects of Polar Compounds from Oidized Palm Oil on Lipid Metabolism and Glucose Tolerance in Kunming Mice** Peirang Cao*, Xiaodan Li, Xiaoyan Yu, Yuanpeng Wang, and Yuanfa Liu, *Jiangnan University, China*

In this study, effects of oxidized palm oil, and its polar compounds generated from the frying process of palm oil, on animal effect of lipid and glucose metabolism were investigated. Kunming mice were fed with high-fat diet containing oxidized palm oil or purified polar compounds for period of 12 weeks. The effect on animal health including liver functions, serum biochemistry and

glucose tolerance were analyzed. Our results revealed that the consumption of polar compounds was positively related to the change of lipid deposition in liver and whole body and glucose tolerances in Kunming mice. Correspondingly, decrease of gene expression involved in lipid metabolism of PPARa, Acox1 and Cpt1a in liver suggested that the polar compounds could facilitate the fatty acid oxidation in peroxisomes and suppress the lipid oxidation in mitochondria. Furthermore, glucose tolerance test (GTT) revealed that high polar compound consumption impaired glucose tolerance, indicating its effect on the glucose metabolism in vivo as well. Our results provided critical information on the effects of polar compounds generated from oxidized palm oil on animal health specially on liver functions and lipid metabolism, which is important for the biosafety of frying oil.

24. **Decoding the Rancid Off-flavor of Olive Oil Using the Sensomics Approach** Anja Neugebauer*, Michael Granvogl, and Peter Schieberle, *Technical University of Munich, Germany*

Olive oil is a favored edible oil around the world for hundreds of years due to its valuable nutrients and its unique flavor profile. Due to the fact that the aroma is one of the most important criteria for consumers' buying behavior, it is crucial to gain more knowledge about the aroma profile of olive oil and the possible off-flavor development during its production and storage. Using the sensomics approach, including comparative aroma extract dilution analysis (cAEDA) by gas chromatography-olfactometry (GC-O) and identification experiments via gas chromatography-mass spectrometry (GC-MS), extra virgin olive oil, eliciting the desired aroma properties, and a rancid off-flavor olive oil were compared. The most potent aroma compounds were quantitated by stable isotope dilution assays (SIDAs) and odor activity values (OAVs; ratio of concentration to respective odor threshold) were calculated. Finally, aroma simulation experiments were carried out to validate the analytical data. Fourteen odorants with an OAV ≥ 1 were detected as key aroma compounds in extra virgin olive oil including 1-penten-3-one, (*Z*)-3-hexenal, trans-4,5-epoxy-(*E*)-2-decenal, acetic acid, 2-isobutyl-3-methoxypyrazine, (*E*)-2-hexenal, and (*Z*)-3-hexenylacetate. The rancid off-flavor olive oil showed significantly higher OAVs for acids, such as acetic acid, butanoic acid, pentanoic acid, and hexanoic acid. Further, saturated aldehydes (hexanal, heptanal, octanal, and nonanal), monounsaturated aldehydes ((*E*)-2-octenal and (*E*)-2-nonenal), and diunsaturated aldehydes ((*E*,*E*)-2,4-nonadienal and (*E*,*E*)-2,4-decadienal) were key odorants of the off-flavor oil. In summary, the rancid off-flavor was characterized for the first time on a molecular level using the combined approach of instrumental and sensorial analysis.

25, **An Investigation into the Stability of Flavour Compounds in Flavoured Fish Oil During Oxidation** Emily Harris[1], Suzanne M. Budge[1], Jenna Sullivan Ritter[2], and Wei Xia*[1], *[1]Dalhousie University, Canada; [2]Nature's Way of Canada, Canada*

The p-anisidine (pAV) test is used to measure the concentration of aldehydes in marine oils and marine oil supplements. However, flavours added to these products to enhance their palatability contribute to the measured pAV, leading to inaccurate results. For this reason, the Global Organization for EPA and DHA Omega-3s (GOED) recommends a modified method to measure the pAV of flavoured marine oils that is predicated on the assumption that the flavour compounds themselves do not oxidize or degrade during oxidation. Here, we proposed to test the null hypothesis, as followed by GOED, that the contribution of flavour to the measured pAV does

not change during storage. We performed stability studies to compare oil samples to which flavour had been added before and after oxidation, respectively. The pAVs and the 1H NMR signal responses of the aldehydic flavour signals in these oils were compared between sample types to determine the extent of flavour degradation over the course of oxidation. The pAVs of oils to which flavour was added before oxidation were significantly lower than the pAVs of oils to which flavour was added after oxidation for several sampling points in both chocolate-vanilla and lemon flavoured oils, indicating that these flavours degrade during oxidation. Additionally, in chocolate-vanilla flavoured oil, the rate of oxidation was faster in unflavoured than flavoured oil, suggesting that compounds in the chocolate-vanilla flavour may be acting as antioxidants. These findings have ramifications for the applicability of the GOED recommended method for measuring the pAV in flavoured oils.

26. **Effect of Antioxidants on Changes in Edible Oils During Frying** S.P.J. Namal Senanayake*[1], and Neeti Chavan[2], *[1]CFS North America, USA; [2]Camlin Fine Sciences Ltd., India*

The effect of antioxidants on the stability of edible oils under frying conditions can be assessed via increase in peroxide value and anisidine value, measurement of viscosity and changes in antioxidant levels during frying. The Oil Stability Index (OSI) or Rancimat results can also be utilized to assess the stability of the frying oils. Both synthetic and natural antioxidants may be used to enhance the stability of frying oils. In this presentation, the performance of various antioxidants in edible oils under frying conditions will be discussed.