PRO 1: Processing ABC—Part I

Chairs: Farah Sköld, Solex Thermal Science Inc., Canada; and Naudy Suarez, Richardson Oilseed Ltd., Canada

Advancements in Vegetable Oil Extraction and Oil Processing Mohammad S. Alam*, *Texas A&M University, USA*

Majority of vegetables oils are obtained by solvent extraction and by mechanical press. The main goal of the extraction process is to optimize the oil yield and to maintain the oil quality. Steps (cleaning, dehulling, flaking or cooking) are important as well for improved oil yield. Screw press is a decades old technique and is slowly being replaced by the solvent extraction process for reasons being cost effective and higher oil yield. However, screw press is still being used for small and medium sized mills. At present, the solvent extraction process is the most efficient method for producing high quality oils and meals. During the extraction/oil processing process considerable amount of energy is being consumed. Excessive energy consumption in oil mills are the major drawbacks and makes the business less profitable. In recent years there has been an extensive evolution in the new equipment designs, seed preparation techniques, new solvent extraction methods which has resulted in higher oil yields. These new developments has proven greater savings in energy, safety and in operation as well. The oil processing industry (refining, bleaching and deodorization) is equally important for producing high-quality oil. New technologies (cavitation, silica refining, enzymatic degumming) has largely contributed in saving times, producing high-quality oil and lower oil loses. In this presentation, a brief over view of the different methods of oil extraction, oil processing will be discussed.

Food and Feed Safety through Grain and Oil Seed Cleaning Daniele Lorenzi, *Buhler Inc., USA*

A short introduction to the importance and value of main ingredient cleaning.

Screw Press Technology for Oil Extraction Eric D. Stibora*, *Anderson International, USA*

In the 118 years since Mr. V.D. Anderson first invented the Expeller® press very little has changed in regards to the general design of the screw press. The majority of screw presses consist of a discontinuous screw encased in a perforated barrel with an electric motor and gear reducer to supply the power/torque and a choking mechanism to control the discharge pressure. While newer designs incorporate premium efficiency motors, more maintenance friendly layouts, and the latest hardened alloys, these changes have done little to change the recovery efficiency on oilseed applications. Instead, advances in the preparation of the oilseed over the years have had a far greater impact on increasing capacities and improving overall oil recovery when utilizing a screw press for mechanical recovery of the oil. Size reduction and heat treatment have the largest impact on improving the efficiency of the press. Traditional size reduction and heat treatment steps can reduce energy consumption in a screw press by over 30%. The development of the high shear extruder, combining both heat treatment and

size reduction in a single step, can reduce energy consumption in a screw press by over 50%.

Pilot Plant Concept "EthaNa" for Ethanolic Extraction of Dehulled Canola Seeds Gunter Börner* and Bernd Steinhauer, *B+B Engineering GmbH, Germany*

The presentation gives a summary and an outlook of current results and targets of various research activities in developing a new concept for a canola seed bio-refinery. EthaNa represents the aim and technological background: Ethanolic Native Extraction of Oilseeds. This concept, researched by a consortium of institutes and industry, includes process development and new products from processing of dehulled canola seeds. In order to prepare this new technology and its benefits for industrial use a pilot plant has been developed and will be constructed. First process step is a new dehulling process using fluidized bed technology with the goal of less than 3% hull content in the canola seed kernels. The innovative approach of using dehulled canola seed is a special treatment of breaking the cells of the seed followed by a direct extraction with ethanol. After separation of the different phases and the desolventation step, a high valuable canola-seed-kernel-concentrate will be obtained. The careful conditions of the process allow the extensive elimination of anti-nutritive substances from the canola-seed-kernel-concentrate without damaging the proteins. The extracted oil has an improved quality and can be considered as a pre-refined oil with significantly less expenditure on the refining process. The separated middle fraction contains various high added value components which can be fed to an additional isolation process gaining e.g. lecithin, tocopherols, polyphenols and further valuable components depending on the application of specific technologies. A prospective outlook for industrial use of the EthaNa-Concept will be presented.

Energy Optimization in Soybean Processing Mohamed Abid*, *Solex Thermal Science Inc., Canada*

Soybean processing plants are always evaluating options to maximize operating margins. Conditioning of Soybeans is an energy intensive process and is critical in achieving target efficiency in oil extraction plants. The availability of waste heat sources vary with plant layout, steam sources and meal cooling technology used. However the choice of a heat transfer technology to efficiently recover this low grade energy is critical to finding an efficient and economical solution. Plate Heat exchangers provide high density of heat transfer area within a compact design that allows for an efficient utilization of the recovered energy. The plate channels maintain high velocities which is essential to higher rates of heat transfer. The Bean Heater Modules allow existing installations to reduce steam consumption, with low capex and minimal shutdown time for installation. These modules can be installed on top of existing bean conditioners, allowing use of waste heat to increase capacity, improve overall conditioning or replace leaking tube sections. The modular design makes installation faster and less expensive. The modules can also be used for new conditioning application using a combination of waste heat and steam sources. The long life of the plates in this abrasive environment reduces maintenance costs associated with frequent replacement of tube sections and required downtime. The external flexible hoses connected

to manifolds allow for easy isolation. Any plate can be blocked off without opening the modules. Also each module has an access door that can be used to replace any plate with minimal disruption of production.

Oilseed Conditioning and Effects on Extraction William C. Morphew*, *Crown Iron Works, USA*

Proper conditioning prepares oilseeds for oil extraction by using temperature, time, and moisture to make the oilseeds more pliable for mechanical processing. For soybeans, this conditioning step can happen before or after dehulling, with the desired goal of producing a soybean crack that can be readily flaked to optimum thickness, while controlling for moisture and temperature. Other seeds also benefit from conditioning before extraction. We will look at conditioning in different preparation processes, and also evaluate the effect that conditioning has on the solvent extraction process.

Solvent Extraction Overview Timothy G. Kemper*, *Desmet Ballestra, USA*

This solvent extraction overview presentation will cover the microstructure of oilseeds, the impact of seed preparation on this microstructure, and the 7 parameters impacting the effectiveness of solvent extracting the oil. This presentation will also describe the main equipment and define the key operating parameters for the various unit processes involved in; solvent extracting the oil from the meal; desolventizing, toasting, drying and cooling the meal; evaporating and stripping the solvent from the oil; recovering the solvent; and capturing solvent from the effluent air stream.

BIO 2.2 / PRO 2: Advances in Enzyme Processing Technologies

Chairs: Xuebing Xu Wilmar (Shanghai) Biotech R&D Ctr. China;Flavio Galhardo USA;

Overview and Recent Developments in Degumming, Interesterification and Biodiesel Hans Christian Holm*, *Novozymes A/S, Denmark*

Abstract not available.

Design and Synthesis of New Lipid Molecules by Assemblying Nature Segments for Multi-functionalities. An Enzymatic Solution Zheng Guo*, *Aarhus University, Denmark*

Thanks to billions of years' evolution, the nature creates the largest biodiversity in both organism species and molecular structures, which afford a sustainable source of structural moieties in connection to activities and functionalities of interests. Over 90% of food ingredients or pharmaceutical excipients are fully or partially rom natural molecules; therefore, design and synthesis of new lipid molecules using natural molecular segments as building blocks is an important pathway to acquire novel properties or deliver new functions in food, cosmetic and pharmaceutical industries. In this talk, I will report the concept and strategy in our lab to design and synthesize ultra-long chain fatty acid alcohol esters for cosmetic application; dual-function phenolic-containing DATEM-analogues as excipients for delivery of functionality; and surface-confined molecules as the barriers for prevention of lipid oxidation. We will show enzyme a green technology could optimally serve as a tool contributing to this chemo-enzymatic approach to minimize environmental impact. Not limited to the structural identification of the synthetic structure by MS, FTIT, 1H/13C/31P NMR characterization and synthetic skills, an intensive discussion is given to the analysis of the structure-function relationship by investigation of molecular packing, EPR measurement and AFM imaging etc, to demonstrate how a scientist could use the knowledge in lipid technology and biotechnology solve the technical challenges presented in food and cosmetic industries.

Value and Potential of Phospholipase C Assisted Enzymatic Degumming in Vegetable Oils. Ying Zha[1], Arjen Sein[2], Steve Gregory[3], Greg LeFebvre[4], and Michael Jung[3], *[1]DSM, The Netherlands; [2]DSM Biotechnology Center, The Netherlands; [3]DSM, USA; [4]DSM Food Specialties, Inc, USA*

The Purifine® enzymatic degumming technology of DSM has been showing its value for the past decade. The technology, which uses phospholipase C to convert phospholipids into diglycerides (a form of oil) and a phosphate group, is economically beneficial and sustainable at the same time. It is economically beneficial, because the extra oil yield from diglyceride formation and reduced entrained oil; and it is sustainable, because it reduces the CFP of the entire degumming process. In more recent years, the application of Purifine® has been extending from expander soybean oil to canola, as well as flake soybean oil. Dedicated researches at different scales are constantly looking for process and product adaptations to match a specific application. One interesting process can be the sequential combination of PLC and PLA1, which is the enzymatic solution for both higher oil yield and low P value. As a consequence of increased oil yield, the enzymatic degummed gums have much lower volume. This means a less diluted

meal stream containing higher protein content. Besides, as these gums are rich in choline phosphate, they have probably higher nutritional value than the meal, and multiple potential applications.

Enzymatic Interestrification Chris Dayton*, *Bunge Limited, USA*

Interesterification is a process that allows the modification of the melting characteristics of a blend of oils without changing the Fatty Acid Composition (FAC) of the original blend. Utilizing interesterification allows the manufacturer to produce a functional product with less saturated fat and with essentially no trans fatty acid. The traditional method for interesterification was to use a strong chemical to rearrange the distribution of the fatty acids in a random fashion with many unwanted side reactions. Commercial enzymes are now available that allow the same rearrangement without the use of harsh chemicals. I will review the Chemical Interesterification (CIE) process and then compare it to the various patented commercial Enzymatic Interesterification (EIE) processes.

Enzymatic Modification of Menhaden Oil to Incorporate Caprylic and/or Stearic Acid Sarah A. Willett*[1], Casimir C. Akoh[1], and Silvana Martini[2], *[1]University of Georgia, USA; [2]Utah State University, USA*

Structured lipids (SLs) of menhaden oil with caprylic (C8:0) and/or stearic acid (C18:0) were produced enzymatically. The molar ratios of acyl donor substrates were 1:1, 3:1, and 5:1 for C8:0, and 1:1, 2:1, and 3:1 for C18:0. Total mol% incorporation of C8:0 or C18:0 were plotted against molar ratios. Linear interpolation was used to estimate molar ratios that would yield SLs with 20 or 30 mol% incorporation of C8:0 or C18:0. Enzymatic reactions were also conducted using different blend ratios of caprylic to stearic acid (40:60, 50:50, and 60:40) to produce low saturated fat SLs with melting point range of 25–35°C. Recombinant lipase from Candida antarctica, Lipozyme 435, and sn-1,3 specific Rhizomucor miehei lipase, Lipozyme RM IM, were compared as biocatalysts in these reactions. Total and sn-2 fatty acid compositions, triacylglycerol (TAG) molecular species, thermal behavior, volatile lipid oxidation products, and oxidative stability were compared. Medium-long-medium (MLM)-type TAGs were produced when C8:0 was the acyl donor, and the 3.03:1 and 4.58:1 ratios incorporated 20 and 30 mol% C8:0, respectively, for 24 h reaction time. A low saturated fat SL was produced when stearic acid was the acyl donor, and the 1.32:1 and 2.41:1 ratios incorporated 20 and 30 mol% C18:0, respectively, for 24 h reaction time. These SLs have the potential for use as nutraceutical due to the conservation of polyunsaturated fatty acids (PUFAs) at the sn-2 position. These SLs also may also be advantageous when formulating food products to reduce consumption of saturated fat and risk of cardiovascular disease.

Cold Enzymatic Degumming on Sunflower Seed Oil Ling Hua*, and Alexey Shevchenko, *Alfa Laval Copenhagen A/S, Denmark*

Physical refining is the most economical refining process in terms of losses and utilities consumption. Degumming is key to overall process performance. Any variation of “special", “deep”, “uni” degumming are quite demanding in terms of feed oil quality. Uniform feed oil quality with minimum variation is required for a stable process. Such a demand is less critical for an enzymatic degumming process. Even switching

feed from crude to water degummed oil does not require much change in the process. The process is still stable and easy to manage. That is a reason why enzymatic degumming becomes the preferred process for physical refining. For wax containing oils (sunflower, corn) dewaxing is required and in wet dewaxing (high speed separator-based process) this is combined with neutralization or washing after neutralization (cold neutralization or cold washing). There are some benefits with this process, but soapstock handling issues are often pushing the industry towards physical refining. Alfa Laval has successfully combined enzymatic degumming with the wet dewaxing process for sunflower oil. Prolonged contact time between enzymes and oil in cold conditions shifts the reaction to the "safe area" when P content after first separator is below 5 ppm and washing does not bring value. Min. 75% waxes are removed simultaneously with the gums. If prolonged cold test is required (36h and longer) a polishing filtration step can be installed after the degumming. Filter aid consumption for this is drastically reduced compared to dry dewaxing process.

How to Overcome the Barrier of Mucilage for Extraction of Omega 3 from Chia Oil?
Gwendoline Gravé[1], Sidrine Koumba[1], Jean-Francois Fabre[2], Eric Lacroux[3], Muriel Cerny[4], Romain Valentin[5], Othmane Merah[1], and Zéphirin Mouloungui[4], *[1]INP - ENSIACET, France; [2]LCA UMR1010 INRA-INP/ENSIACET, France; [3]Chimie Agro-Industrielle, France; [4]Laboratoire de Chimie Agro-Industrielle, France; [5]INRA, France*

Chia seed is an oleoproteaginous seed and it is also covered by mucilage. This layer of polysaccharide thickens the medium when seeds are immersed in water complicating oleosomes extraction. This work aims to develop a process to obtain mucilages, oil bodies and fibers from chia seeds. The first step consists of separating polysaccharides of seed's surface by sonication, in order to obtain mucilage1, and also to increase the accessibility to oleosomes. Once the polysaccharides layer removed oleosomes can be extracted grounding degumming seeds. Thanks to water and mechanical energy (high-shearing device and high pressure homogenizer) oil bodies as an emulsion2 are obtained. These emulsions, stabilized by phospholipids and proteins3, are mainly composed of triglycerides as omega 3 (63%) and omega 6 (19%). By the addition of a enzymatic lipolysis step, oleosomes become a source of fatty unsaturated acid for production of omega-3 and omega-6 mono-glycerides by glycerolysis or esterification4 reactions. Besides oil (20%), this process gives access to fibers (30%) and an aqueous phase rich in minerals (9%) and proteins (15%). These oleosomes reveal the potential for the generation of new platform of omega-3 biomolecules. All these compounds have properties for a wide range of applications (alimentation, health, pharmaceutical and cosmetics). 1. Castejón, N. et al. J. Agric. Food Chem. 65, 2572–2579 (2017). 2. Fabre, J.-F., et al. OCL 22, D607 (2015). 3. Deleu, M. et al. Colloids Surf. B Biointerfaces 80, 125–132 (2010). 4. Wallis, C. et al. Waste Manag. 60, 184–190 (2017).

Pilot Enzymatic Production of Medium- and Long-chain Triacylglycerols Using a Solvent-free Packed Bed Reactor Yong Wang*, *Jinan University, China*

Abstract not available.

Lipid Modification by Enzymes and Engineered Microbes Uwe T. Bornscheuer*, *University of Greiswald, Germany*

This lecture will provide an overview about recent developments for the use of enzymes as well as microorganisms in the modification of fats and oils [1,2]. Beside the well-established lipases and phospholipases, more recently also CoA-independent acyltransferases as well as isomerases/hydratases and decarboxylases became important for lipid modification. Furthermore, the combination of isolated enzymes in cascade reactions is relevant to make more complex products at higher overall yields. In addition to isolated enzymes, many microorganisms are used to produce biotensides such as sophoro- and rhamnolipids. The recent major developments in metabolic engineering enabled the creation of tailor-designed microorganisms useful for the production of e.g., EPA, wax esters or drop-in biofuels from fats/oil and glucose as renewable resources. [1] Bornscheuer, U.T., Ed. (2018), Lipid modification by enzymes and engineered microbes, AOCS press. [2] Bornscheuer, U.T. (2018), Enzymes in lipid modification, Ann. Rev. Food Sci. Technol. DOI: 10.1146/annurev-food-030117-012336

Recent Progress of Enzymatic Synthesis of Polymers Douglas G. Hayes*, *University of Tennessee, USA*

Hydrolases, particularly lipases, have been proven to be useful enzymes in oleochemistry, including the ability to form polyesters from multifunctional fatty acids and alcohols. The first papers came out in this area in the late 1980s. The research field has continued to expand, to take advantage of newer solvent systems such as ionic liquids and deep eutectic solvents to enhance miscibility, and newer multifunctional molecules such as phenolics (e.g., caffeic acid and cardanol), amino acids, functionalized fatty acids (e.g., via reactions across double bonds and metathesis), polyols, biodiesel co-products (e.g., glycerol carbonate and polyglycerol) and carbohydrates. In addition, the ability to add polymerizable groups to oleochemical synthons, such as the vinyl or acrylate group, via biocatalysis provides a broad platform for chemical synthesis of the modified monomeric units. Recent studies of enzymatic or chemo-enzymatic synthesis of polymers will be reviewed, with applications in lubrication, materials, foods, nanotechnology and other areas.

PRO 2.1: Environment, Health, Safety and Sustainability

Chairs: Richard Barton, N. Hunt Moore & Associates, USA; and William S. Minor, CHS, USA

Managing Environmental Challenges in Uncertain Times Michael J. Boyer*, *AWTMS, USA*

Environmental challenges in industry, in the US and other parts of the world, are constantly changing. Current demands are now dominated by various regulatory, political and mega impact factors that create much greater uncertainty for managing issues to meet the needs of all stakeholders. In addition new products and processes often create unintended consequences that must be dealt with. The solutions must also meet company and other sustainability objectives This paper will address major factors in oilseed and fats and oils processing.

Creating Triple Win Sustainability Programs William S. Minor*, *CHS, USA*

"Triple Win Sustainability" has been defined as being good for the environment, good for the customer and good for the producer. Designing and executing sensible sustainability programs that deliver solid results in all three areas is not difficult or time consuming. The average processor will find that work that is already being done for other reasons can be harnessed to deliver "Triple Win" results. For example, we all try to minimize energy consumption for cost control purposes—a win for us, the producers. Reducing energy consumption obviously is a win for the environment. Our reduction of energy use is a win for our customers as they can rightfully claim that they source from producers who are good stewards of the environment. This is but one example of many where things we are already doing can be the basis of a sound sustainability program. The intent of this oral presentation will be to share how to design and execute "Triple Win" sustainability programs with a minimum of additional resources.

Test Tube to Tanker: Stage Gate Engineering as a Guide to Process Development Cecil T. Massie*, *Wood, USA*

Stage gate engineering is commonly used to estimate the capital cost of a new technology. Experience teaches, however, that stage gate engineering can also direct process development to the most significant technical and economic issues of that process. This presentation discusses how FEL-1 and FEL-2 engineering can apply to prioritizing process development. By looking at commercial scale operations early, process development is directed to unit operations that are more robust and suited for commercial scale operation. Such awareness encourages the data collection needed for sizing commercial scale unit operation, and avoids unnecessary backtracking to collect data during the commercialization phase. The result is faster commercialization and lower development costs.

Challenges and Opportunities in Effluent Water Treatment for Oil Seed Processing Plants Rakesh Patel*, *ADF Engineering, USA*

Treating oily wastewater at oilseed plants can be challenging due to variability in flow, oil content, nature of dissolved oils (free vs emulsified) and the extent of dissolved and suspended solids. Frequent spills of oils, solvents, caustic and acids add to the complexity to the wastewater characteristics. Notices of violation, surcharges and fines from the city water

treatment plant or the state environmental agency are not uncommon if the wastewater treatment plant does not perform consistently and reliability. A lot of oil seeds plants also miss out on revenue stream from recovered oil if they do not adequately segregate and treat wastewater streams generated in different plant areas such as crush, extraction, refining and packaging. This presentation focuses on how oilseed plants can upgrade and install cost effective wastewater treatment solutions by using physical and chemical methods to overcome the complexities and variability in wastewater characteristics and oil concentrations.

Creating an Effective Dust Hazard Analysis for NFPA 652 Compliance Matthew Williamson*, *ADF Engineering, USA*

In September 2015, NFPA issued NFPA 652—Standard on the Fundamentals of Combustible Dust. This new standard integrates all of the older, industry specific standards, such as NFPA 61, into a single, unified, consistent set of rules. The key new requirement is that all facilities which handle or generate combustible dusts must complete a Dust Hazard Analysis (DHA) by September 2018. This critical safety document identifies the specific combustible dust risks and establishes a mitigation plan for the site. How can older grain facilities with limited documentation effectively develop a DHA in time? This presentation will discuss the required information and how best to obtain it, as well as a proper format and the appropriate information to include in the DHA itself. Additionally, cost effective and timely implementation of the DHA findings will be discussed.

Towards Sustainable Production of Structured Lipids for the Food Industry Suzana Ferreira-Dias*, *Universidade de Lisboa, Instituto Superior de Agronomia, Portugal*

The production of novel lipids with important medical and functional properties, i.e. structured lipids (SL), is rapidly increasing. Most of the SL can only be synthesized by sn-1,3 regioselective lipase-catalyzed reactions, which preserve the original 2-position of fatty acids in triacylglycerols (TAG). We have been working on the production of (i) human milk fat substitutes (HMFS), (ii) low calorie TAG of MLM type and (iii) interesterified fat blends rich in omega-3 PUFA for the margarine industry. HMFS and MLM have been produced by acidolysis. For the implementation of low-cost sustainable processes for SL production, reactions have been carried out in solvent-free media. Enzyme costs are the main constraints of process upscaling. The search for novel biocatalysts presenting both high catalytic activity and operational stability has been attempted to lower enzyme costs. Non-commercial sn-1,3 regioselective lipases namely the heterologous Rhizopus oryzae lipase (rROL), produced by F. Valero's group (UAB, Spain) immobilized in different supports and Carica papaya lipase self-immobilized in the latex, produced by G. Sandoval's group (CIATEJ, Mexico) were tested. Also, natural cheap oils and fats and oils extracted from agro-industry by-products (e.g., grapeseed and olive pomace) were used as raw materials to minimize costs. The obtained results were similar or even superior to those obtained with the expensive commercial immobilized lipases, showing the feasibility of the implementation of a sustainable production of SL for the Food Industry.

Challenges of the Palm Oil Industry Peter J. Clarke*, *The Tintometer Ltd, United Kingdom*

This presentation provides an insight into some of the challenges of the Palm Oil Industry and the processes and practices that could be established to minimize the effects. The increasing trend of oil usage worldwide and the resultant effect on price fluctuations is driving the need for more accurate analysis within the supply chain. Specific to this is the rising popularity of palm oil and its derivatives: non-repetitive analysis across refineries, however, is still an issue. Palm oil is unique in its fractionation – resulting in different melting points for different applications. This makes the sample temperature and sample heating to maintain the temperature during measurement more important since temperature and colour go hand in hand. Temperature and duration times must be accurately set to ensure heating to the full liquid phase for measurement, while bearing in mind that overheating can cause the oil to turn darker. Microwave heating is faster but may not result in uniform temperatures of the oil. Further, due to the high melt temperature, Palm Oil shortens very quickly: as soon as the temperature is removed, it starts to solidify, visually turning the sample increasingly whiter. We look at comparative data from various real-life applications and the resultant spectral data received from the measurements: analysing how improvements may be made for closer inter-company agreement. Colour is an important measurement of the quality and, hence, value of Palm Oil. Misinterpretation in the industry can result in rejection, antagonism between suppliers and costs.

The Compliance Audit — A Challenge to Your Process Safety Management Program John Mulholland*, *N. Hunt Moore & Associates, USA*

The Compliance Audit A challenge to your Process Safety Management program Every three years the Process Safety Management Standard 29 CFR 1910.119 requires that processes covered by the standard complete a compliance audit which verifies that the practices and procedures developed under the standard are adequate and being followed. This sounds like a fairly straightforward task, however, due to the interrelationship of the 14 elements of PSM, coupled with the fact PSM is performance based standard and the many interpretations of the standard this can be a daunting undertaking. During this session we will look at an overview of the regulations and based on training and experience identify some of the areas plants succeed and fail in meeting the standard with emphasis on how to stay in compliance.

PRO 3: By-product Processing

Chairs: Norman J. Smallwood, The Core Team, USA; and Samia Mezouari, FoodScience - R&D-qc, Canada

Current Situation for Spent Bleaching Earth Disposal Norman J. Smallwood*, *The Core Team, USA*

Due to the rapid oxidation of the retained oil, spent bleaching earth is vulnerable to spontaneous combustion. Finding a safe, inexpensive and environmentally sustainable solution for disposal has long been pursued. For integrated oilseed and edible oil processing facilities, adding spent bleaching earth to the solvent extractor feed or to the solvent extractor discharge meal stream is used in some cases. Independent solvent extraction of the oil in spent clay has rarely been found to be economically viable. Solid waste disposal involving prompt coverage with soil has and continues to be the dominant practice worldwide. By mixing spent bleaching earth with sufficient feed-grade salt and/or lime to eliminate the spontaneous combustion hazard and adding other desired ingredients, high-energy nutritional products can be produced for livestock and poultry feed at an especially competitive cost.

Processing of Atlantic Salmon (*Salmo salar*) Heads for the Extraction of Proteoglycans Subin R. C. K. Rajendran*[1], Aishwarya Mohan[2], Zied Khiari[3], and Beth Mason[2], *[1]Department of Chemistry, Dalhousie University, Canada; [2]Verschuren Centre for Sustainability in Energy and the Environment, Canada; [3]Lethbridge College, Canada*

Fish processing industries generate large amounts of low value by-products and wastes (viscera, frame, head, skin etc.). Glycosaminoglycans along with collagen peptides are the most valuable biomolecules derived from these wastes with several prophylactic activities along with immunomodulatory and cosmetic applications. Current processes for the preparation of glycosaminoglycans are unsustainable as these depend on harsh chemical/physical treatments or costly enzymatic treatments. The present study describes the development and optimization of economically and environmentally sustainable alternate processing approaches for the extraction of glycosaminoglycans from salmon heads. Multiple proteoglycan extraction, liberation and separation strategies were developed based on the activation of endogenous proteases present in the salmon tissue, facilitating extraction without additional commercial or purified enzymes and moisture. Following hydrolysis, soluble peptides and glycosaminoglycans were centrifugally separated from fat and partially decalcified bones. Peptides are then separated from glycosaminoglycans through ultrafiltration with a molecular weight cut-off membrane. Different processing strategies were compared for their effectiveness in the recovery of glycosaminoglycans from salmon heads. The results from this study direct towards the effective application of processing approaches for valourization of by-products from fish processing industries.

Oil Recovery from Palm Kernel Meal using Subcritical Water Extraction in a Stirred Tank Reactor Johnnys A. Bustillo Maury[1], Andres F. Aldana Rico*[1], Jerry W. King[2], Cindy L. Garcia Pinto[1], Ingrid N. Hernandez Medina[1], Jose Urbina[3], Juan C. Urueta[3], Marco E. Sanjuan Mejia[4], and Antonio J. Bula Silvera[1], *[1]Universidad del Norte, Colombia; [2]Critical Fluid Symposia, USA; [3]PALMACEITE SA, Colombia; [4]msanjuan@uninorte.edu.co, Colombia*

Water at subcritical conditions, experiences significant changes in dielectric constant and ionic product, making it capable of solubilizing non-polar products such as oils. The objective of this research was to study the extraction of remaining oil on palm kernel meal in a stirred tank reactor. After the extraction quality analysis was performed on the obtained oil to determine if it was suitable for mixing with standard extracted product. The experimental apparatus consisted of a 250ml stainless-steel stirred tank reactor with a temperature-controlled heater and a cooling water circuit were the meal-water mixture was poured. Head space addition of Nitrogen did initial pressurization up to 6Bar. Later, pressure started to increase according to temperature (150C). After the desired process time, mixture was cooled down. Mixture was taken out from the vessel and centrifuged; oil layer was quantified and used for analytical tests. Mass yield was calculated by comparing the extracted oil amount with the produced by a standard Soxhlet extraction. Free fatty acid determination was done by ASTM-D5555 method and fatty acid identification was done by gas chromatography with FID detection. A central composite design defined process conditions with three factors and five levels. Factors and levels were meal mass (9–23g), reaction time(8–16min) and particle size (0.212–2mm). Extraction yield reached a 40% (w/w) without another meal modification different to the oil reduction. Also, no catalyst was applied, free fatty acid content didn't increase over 3% and fatty acid profile was maintained with Lauric acid as predominant species.

Use of Lipids for Enhancing the Nutritional Value, Digestibility and Health Safety of Hay William J. Hausmann*, *The Core Team, USA*

Hay fed to livestock has three significant deficiencies: low fat content for metabolizable energy; rough texture that causes digestive tract inflammation, especially for young animals; and toxic mold dust if exposed to excessive moisture before use. Treatment of hay with a broad array of edible lipids can eliminate or significantly reduce these hay deficiencies. A variety of methods can be used to effectively apply the broad range of lipids to hay. This use of lipids creates an enormous new market. The market can be particularly valuable for the by-product class of lipids including acidulated soapstock, gums from the enzymatic degumming process, and degraded lipids not acceptable for human use but acceptable for livestock. Application for an international patent for this lipid use has been made in the major hay-producing countries.

Investigation of the Selective Extraction of Non-polyisoprenic Compounds from Parthenium argentatum Biomass with Pressurized Fluids Teerasak Punvichai*[1], Daniel Pioch[2], Serge PALU[2], and Eric Tardan[2], *[1]Prince of Songkla University, Thailand; [2]CIRAD, UR 114 Biowooeb, TA-B 114/16, 34398, Montpellier, France, France*

P. argentatum (guayule) is a dry climate bush producing polyisoprene (PI) having properties close to hevea's, and resin, a complex mixture of terpenics and lipids. Chemical analysis (GC, TLC,

HPLC, IR, NMR) which needed setting-up new extraction protocols, coupled to imaging techniques, shows the important variability among the 26 biomass samples of same genotype (age, place): resin content in biomass parts and composition. The ratio of unsaponifiable / saponifiable fractions is always found larger than 1; taking into account that this last fraction contains 65% of cinnamic and para-anisic acids issued from the saponification of guayulins (sesquiterpenes esters), fatty acid-containing lipids are thus minor components in resin. The saponifiable fraction shows ten identified fatty acids (C10 to C21, mainly saturated), about the double of reported fatty acids in literature (C16 and C18, mainly unsaturated).

Orange Oil Based Highly Flame-retardant Rigid Polyurethanes Foams for Industrial Applications Sanket Bhoyate, Mihail Ionescu, Pawan Kahol, and Ram Gupta*, *Pittsburg State University, USA*

Flammability of polyurethane foams is a global issue as it limits their wide applications in different fields such as in construction, automobile, and household appliances industries. We have used orange oil-based polyol along with reactive flame retardants for the preparation of flame retardant polyurethane foams. Reactive flame-retardants based on aliphatic and aromatic structured bromine compounds were synthesized and used to prepare bio-based polyurethanes using limonene dimercaptan, a derivative of orange oil. The foams containing aromatic flame-retardant compounds showed higher closed cell content (average 97% and 100%) and compressive strength (230 and 325 kPa) to that of aliphatic flame retardant containing compounds. The horizontal burning test confirms that aromatic flame retardants are more effective than aliphatic flame-retardant compounds. At 5% of bromine contents, a foam containing aromatic flame retardant displayed a burning time of 12 seconds while aliphatic compound containing foam showed a burning time of 26 seconds. Polyurethane foams without any flame-retardant compounds showed a burning time of 74 seconds. The percentage weight loss for neat polyurethane foam was 27%, while foams containing 5 wt% bromine in aliphatic and aromatic compounds displayed weight loss of 11 and 14%, respectively. Our results suggest that orange oil-based polyol along with reactive flame-retardant compounds could be used for the preparation of rigid polyurethane foams with high flame-retardant properties.

Impact of Heat and Pressure on the Polyphenol Composition of Plant Extracts Michael Eskin and Usha Thiyam*, *University of Manitoba, Canada*

The use of high-temperature and high-pressure based extraction and processing has resulted in extracts rich in polyphenolic compounds. In canola, thermal treatment of sinapic acid can produce canolol which is known for its anti-mutagenic properties. Thus, food processing can generate novel active molecules with enhanced properties compared to the original molecules, e.g., dimers and oligomers of caffeic acid, cocoa procyanidins. Once thought to be a simple routine procedure, targeted extraction can often have dramatic effects. It can change the structure of a phenolic molecule, causing a marked change or substantially enhancement of its functions. Several unidentified polyphenols were detected as a result of heat and pressure processing using Accelerated Solvent Extraction generating

catechin, cannabisin, and two unknown lignamides in hemp extracts. The impact of these novel processes on sinapates (sinapic acid derivatives) and hemp-based lignamides will be presented, as part of our characterization of plant-based phenolic compounds.

Challenges of Extraction and Downstream Processing of Polyunsaturated Oils from Microbial Biomass Thushan S. Withana-Gamage* and Udaya N. Wanasundara*, POS Bio-Sciences, Canada*

Micro-algae are a diverse group of photosynthetic organisms which convert solar energy more efficiently as compared to vascular plants. Microalgae produce wide variety of components including proteins, lipids, carbohydrates, vitamins, enzymes, or carotenoids. The lipids and lipid-soluble bioactives are the major bio-products in the micro-algae. Recent advances in molecular biology and biotechnology lead to higher oil content and enhanced the fatty acid composition with high polyunsaturated fatty acids (high ALA, DHA, DPA, and EPA strains) in both phototropic and heterotropic growing conditions. However, disruption of cell wall and extraction oil from algae continues to have a significant challenge in producing pure and quality oil with high recovery. Furthermore, extracted polyunsaturated oils from microbial sources contain considerable amounts of non-triacylglycerol (non-TAG) substances. (i.e., free fatty acid, colored substances, phospholipids, glycolipids, sterols, etc.). Presence of non-TAGs decreases storage stability and shelf life of the oil. Therefore, the processing of crude algal oils comprising a series of discrete steps such as degumming, alkali refining, bleaching and deodorization (RBD) is carried out to remove these impurities. At POS Bio-Sciences (Canada), we are capable to extract (bead milling and high-pressure homogenizer) and process highly unsaturated crude oil into stable RBD oil in small- or large-scale quantities via different techniques. At POS Batavia Bio Processing plant (USA), we are capable of fractionation and enrich the polyunsaturated algal oil using different fractionating columns and short path distillation columns. This presentation focuses on our process capabilities and new methodologies that we are able to provide.

BIO 3.1/PRO 3.1: Biodiesel

Chairs: Per Munk Nielsen, Novozymes A/S, Denmark; Casimir C. Akoh, University of Georgia, USA; and Anders Rancke-Madsen, Novozymes, Denmark

Improving Pre-treatment Efficiency of Oil Feedstock using Adsorbent Filter Aids David Gittins, Li-Chih Hu, and Nathan Dias*, *Imerys Filtration Minerals Inc., USA*

Physically refined vegetable oil and mixed feedstock are often pretreated to remove contaminants which cause issues in downstream production of biodiesel or edible oil. Adsorbents are typically used in conjunction with filter aid in a dry washing filtration step to reduce contaminants. On their own, adsorbents do a great job of removing phospholipids, soaps, metals, and other contaminants but often suffer the effects of short filtration runs or require a lot of adsorbent or filter aid to meet contaminant reduction and throughput needs. The purpose of this talk is to describe how hybrid adsorbent filter aids combine unique filtration kinetics and surface chemistry to reduce contaminants, reduce overall powder use, and increase filtration cycle in oil pre-treatment.

Online Real-time Quality Control of Biodiesel using Near-Infrared Spectroscopy Paul Dawson[1], Yosra Allouche[2], and Fabiano de Melas[2], *[1]BUCHI Corporation, USA; [2]BUCHI NIR-Online GmbH, Germany*

Quality control of Biodiesel (B100) is of utmost importance and key parameters to be analyzed are commonly defined in ASTM D6751 and EN-14214. However, the chemical analysis of Biodiesel expensive, time-consuming and generates waste products. Here, we introduce a by-pass based online solution of non-invasive near-infrared (NIR) technology for simultaneous, real-time measurements of e.g. acid value, ester content, methanol, monoglyceride, total glycerol and water content. In general, analytical equipment to be utilized directly in a process stream needs to be capable of coping with harsh industrial environments such as vibrations or extreme climate conditions in case of outdoor installations. To circumvent these challenges NIR spectrometers based on robust diode array technology without any moving parts have been developed. Light emitted from a halogen source is transflected from the sample, collected and diffracted on a stationary grating. Spatially separated light is then detected by means of a diode array followed by automated chemometric analysis. Predicted values are displayed in the control room. Operators may therefore correct process deviations in real-time greatly reducing safety margins or out-of-specification batches leading to an increased overall production capacity Moreover, the described analytical solution is in full compliance with ATEX II 2G Ex px II T4 and may thus be installed in potentially gas explosive environments.

FFA Reduction and Production Control Frankie Mathis* and Bo Munc, *Tactical Fabrication LLC, USA*.

Tactical Fabrication (TF) has for more than a decade been using Resin Technologies to improve the Biodiesel production. In the past 4 years this has included the functionality of Resin Acid Catalyzed Esterification (RACE) of Free Fatty Acid (FFA) after the Novozymes Eversa enzymatic process. This add-on technology can be used in

any production, but become essential when working with ultra-high FFA feedstock like Brown Grease (BG) or Palm Fatty Acid Distillate (PFAD). The water produced in a BG (FFA >85%) reaction compare to UCO (FFA

Integrating Conventional and Enzymatic Approaches Towards Industrial Biodiesel Production Marcelo Cantele* and Jose Vladimir de Oliveira, *Tranfertech Gestão de Inovações LTDA, Brazil*

Currently, large scale production of biodiesel has been mostly based on homogeneous, alkali catalysis. Nevertheless, biotechnological production of biodiesel with lipases has received growing consideration and compared to conventional alkali-catalyzed production, the enzymatic process is less energy intensive and produces higher-purity product with less downstream operations. Different from the conventional base-catalyst route, the enzymatic process is very tolerant to high acid and water contents hence allowing the use of unrefined, less expensive, high FFA, lower-grade oils and fats, such as soapstock, acid oils and grease strap. The use of the commercial low-cost, soluble free-lipase Eversa Transform 2.0, recently launched by Novozymes, has been tested and shown to be cost-efficient and sustainable at industrial level. However, plant integration of conventional and enzymatic approaches may be much more advantageous than stand-alone production units as relatively low acid charges (~ 10 wt%) may be pre-treated by enzymatic esterification allowing then direct inlet through alkali-catalyzed reactor and also olein generated in the basic-transesterification route would be welcome in the soluble free lipase-catalyzed reactor. The rationale of infrastructure for both routes should be then explored to assure the best strategy towards keeping profit margins at the highest levels. This talk illustrates such scenario, taking into account a biodiesel production unit of around from ~ 100 ton/day, discussing a flow chart of an integrated base and enzyme-catalyzed processes. It is shown that the integrated plant approach may constitute a promising alternative to biodiesel manufacture and can contribute to make the enzymatic biodiesel feasible.

Enzymatic Esterification to Handle the FFA in Biodiesel Production Per Munk Nielsen*, *Novozymes, Denmark*

Enzymatic esterification to handle the FFA in biodiesel production P.M.Nielsen, A.Rancke-Madsen, H.C.Holm Free fatty acids (FFA) is a separate issue in the production of biodiesel. This goes for both the chemically and the enzymatically catalyzed biodiesel. The chemically biodiesel needs to treat the raw material to obtain a feed stock low enough for the Na-methylate process. This can be physical removal or esterification with acid catalyst. Both ways do have some significant drawbacks. For the biodiesel produced using enzymes as catalyst it is normal with 1-3% FFA in the crude biodiesel coming out of the enzyme reactor. There is one process—enzymatic esterification—that can esterify the FFA whether it is coming with crude oil or it in the crude FAME after the transesterification reaction. The way of controlling the process is by managing the chemical equilibrium which mainly must do with the methanol/water content in the reaction mixture. Several methods for making a production setup have been suggested over the years. This presentation will discuss the

alternative process options and emphasize the pros et contra of the processes and concluding which process is the optimum for esterifying the FFA to FAME.

Liquid Lipases for Enzymatic Refining: Technical Advantages Beyond Green Technology Zheng Guo*, *Aarhus University, Denmark*

Benefited from tremendous in genetic/protein engineering and fermentation technology, production of liquid enzymes could be achieved at high titer and high purity in low price, which promotes enzyme engineers and industries rethink the strategy and choice of free (liquid) enzymes for industrial application; particularly for production of low value but large quantity products. This work examined catalytic specificity and fatty acid selectivity of five liquid lipases C. antarctica lipase A and B (CAL-A/B), and lipase TL (T. lanuginosus), Eversa Transfrom and NS in ethanolysis of fish /microalgae oils with the aim to concentrate n-3 PUFAs into monoacylglycerols (MAGs) products. Lipase TL, Eversa Transform & NS entail a much faster reaction and produce higher MAGs yield (>30%); whearas CAL-A obtains the highest concentration of n-3 PUFAs/DHA/EPA into MAGs products (88.30%); followed by lipase NS (81.02%). 13CNMR analysis indicates that CAL-B and lipase TL are sn-1,3 specific; but CAL-A and lipase Eversa Transform are non-regiospecific or weak sn-2 specific; which plausibly explians high enrichment effect of the latter two lipases. All liquid lipases are observed reusable for a certain times (lipase Eversa Transform up to 12 times), demonstrating their competitive advantage over immobilized form for industrial application because of their higher activity and cheaper operation cost. For a complete utilization of fatty acyls, a further process is carried out after MAG enriched with high content PUFAs is isolated by short path distillation; where the side products are converted into biodiesels at high yield by the same liquid lipase system but different reaction conditions. Thus a complete value chain is created by liquid enzyme system.

A New Enzymatic Biodiesel Polishing Process Based on Esterification of FFA into Methyl Esters Anders Rancke-Madsen*, *Novozymes, Denmark*

The use of liquid Thermomyces lanuginosus lipase in biodiesel production has been a break-through as liquid lipase can handle crude feed-stocks with any content of free fatty acids at low economical risks. However, equilibrium level around 2-3% FFA requires caustic polishing step and recycling of 7-8% of fatty matter to avoid compromising yields. A next generation FFA polishing process is based on a liquid version of Candida Antarctica lipase B. This lipase can operate at low water activity making it excellent for converting FFA into methyl esters and thus reaching low equilibrium levels of FFA, thus no or limited caustic FFA polishing is needed. The process is based on conventional technology only including reactor design and water removal techniques. Reaction conditions in lab on crude PFAD based methyl esters are 0.75 kg enzyme/ ton oil, 10% w/w dry glycerol, 3.5% w/w dry methanol and 13 hours of reaction time at 104°F/40°C and 250 rpm. After reaction, heavy phase is separated off by centrifugation and methyl ester phase is added dilute caustic for 30 minutes followed by separation by centrifugation, 2% w/w water washing step and drying. The biodiesel yield is 99% with FFA < 0.1% in final biodiesel. Glycerol and methanol is recovered from heavy phase and reused. The

presentation will discuss the different process scenarios and key process parameters and document the overall process robustness with data from lab.

Soapstock Acidulation using Carbon Dioxide

Rusty Sutterlin*, *Inventure Renewables, USA*

The Inventure Carbonate Process converts soapstock, refinery byproduct lipid, Fatty Acid Distillate (FAD), Lecithin or any other fatty material into Free Fatty Acids (FFA) using heat and carbonic acid, as opposed to the traditional mineral acid pathway. The FFA produced is then upgraded to the valuable Olein/Stearin products via downstream purification. The waste water from this process can be concentrated and sold as an animal feed additive. The feedstock undergoes a continuous pre-treatment step prior to the acidulation with carbon dioxide. The material is then pumped to the acidulation batch reactor where carbon dioxide is introduced at medium pressures. After the reaction takes place, it is allowed to settle where any water present is decanted off and sent through a flash drum to recover carbon dioxide. The material is then routed to a combination of distillation/crystallization to product the final olein/stearin products.

LOQ 3a/PRO 3.2a: Effect of New Processing Technologies on Lipid Oxidation

Chairs: David Johnson, Kalsec Inc., USA; and Antonios Papastergiadis, Desmet Ballestra, Belgium

Oxidative Stability of Tomato-based Matrices Enriched with n-3-LC-PUFA Derived from Microalgae Lore Gheysen[1], Nele Lagae[1], Jolien Devaere[2], Koen Goiris[4], Luc De Cooman[2], and Imogen Foubert[1], *[1]Katholieke Universiteit Leuven Kulak, Belgium; [2]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. Fruit and vegetable-based products could be valuable matrices to enrich with n-3-LC-PUFA, since they are important carriers of nutrients and fibers and essential in a healthy diet. This study investigates the impact of processing on tomato-based matrices enriched with the microalga Nannochloropsis compared to commercial fish oil. Intact biomass, disrupted biomass and extracted oil were screened as different delivery systems of Nannochloropsis. For each delivery system of Nannochloropsis as well as for commercial fish oil, a tomato-based matrix was made containing 80 mg n-3-LC-PUFA/100 g suspension. The impact of high pressure homogenization, pasteurization and sterilization on the n-3-LC-PUFA enriched matrices was followed in a dual way. First, the matrices were characterized before and after each (mechanical and thermal) process step for their amount of n-3-LC-PUFA, free fatty acids, carotenoids, polyphenols, vitamin C and tocopherols. In addition, all the samples were stored for 12 weeks at 37°C to follow up their oxidative stability at different time intervals (0, 2, 4, 8 and 12 weeks). The results showed a limited impact of processing and an influence of delivery system. In this study, promising results for the use of microalgal biomass in tomato-based products as an alternative source of n-3 LC-PUFA were observed.

Oxidation and Hydrolysis of Lipids in Marine Edible Shellfishes During Hot Drying Process Dayong Zhou*[1], Zhongyuan Liu[2], Kaiqi Gang[3], Fereidoon Shahidi[4], and Tong Wang[5], *[1]Dalian Polytechnic University, China; [2]College of Food Science & Technology, Dalian Polytechnic University, China; [3]School of Food Science and Technology, Dalian Polytechnic University, China; [4]Memorial University of Newfoundland, Canada; [5]Iowa State University, USA*

Objective: Hot-air drying techniques are widely utilized to decrease the water activity and extend the shelf-life of shellfish. So far, little information is available about the impact of the drying process on the lipids in shellfish in which the n-3 LC-PUFA are present primarily as phospholipid. Therefore, the objective of the present study was to evaluate the hydrolysis and oxidation of lipids in marine edible shellfishes such as clams and whelks upon hot air drying. Methods used: Marine shellfishes were processed by hot air drying, and the lipid content, lipid classes, PL classes, fatty acid composition, acid value (AV), peroxide value (POV), thiobarbituric acid-reactive substances (TBARS) and total oxidation (TOTOX) value were

determined. Furthermore, oxidation (OXITEST) method was used to evaluate the oxidation level and stability of the dried shellfish. Results: The drying processing reduced percentages of triacylglycerols, phosphatidylcholine and phosphatidylserine but increased percentages of lysophosphatidylcholine and lysophosphatidylethanolamine, indicating the hydrolysis of the lipids. Interestingly, the POV, TBARS and TOTOX all decreased after the hot air drying process. However, the significant decline of the induction period for the dried shellfish at elevated temperatures indicated their higher oxidation level, poor oxidative stability and reduction of shelf-life. Conclusion: The traditional oxidation measurements, such as AV, POV, TBARS even TOTOX are inadequate for evaluating the level of lipid oxidation in shellfish upon hot air drying process. In contrast, OXITEST method is shown to be an effective tool for estimating lipid oxidation level for hot air dried shellfish.

Effect of Spray-Dried Flavonoid Microparticles on Oxidative Stability of Methyl Linoleate as Lipid Model System Manuel J. Palma*[1], Gloria Márquez-Ruiz[2], Paula García [3], Francisca Holgado[4], Cristina Vergara [3], Begoña Giménez[5], and Paz S. Robert[6], *[1]Universidad de Chile, Chile; [2]Instituto de Ciencia y Tecnología de Alimentos y Nutrición (ICTAN-CSIC), Spain; [3]Departamento de Ciencia de los Alimentos y Tecnología Química, Facultad de Ciencias Químicas y Farmacéuticas, Universidad de Chile, Chile; [4]Instituto de Ciencia y Tecnología de Alimentos y Nutrición (ICTAN-CSIC); [5]Departamento de Ciencia y Tecnología de los Alimentos, Facultad Tecnológica, Universidad de Santiago de Chile, Chile; [6]Universidad de Chile, Chile*

The objective of this work was to evaluate the antioxidant action of microencapsulated flavonoids with specific differences in chemical structure, namely, quercetin (Q) and epicatechin (E) in bulk methyl linoleate (ML) under oxidation conditions at 60ºC, in a Rancimat apparatus. Microencapsulated Q and E were prepared by spray-drying using inulin (IN) as encapsulating agent (Q–IN and E–IN) as well as with Capsul (C) as channelizing agent (Q–IN–C and E–IN–C). Microparticles were added to ML and results showed that Q microparticles markedly improved its oxidative stability by increasing the induction period values and delaying the formation of oxidation compounds, as determined by high-performance size-exclusion chromatography, with respect to E microparticles, thus suggesting the importance of flavonoid C-ring substitution. Remaining levels of Q in the lipid system throughout oxidation of ML added with Q microparticles seemed to show two releasing zones: the first one corresponds to the equilibrium zone, when Q released from microparticles replaces Q that is being degraded; the second zone corresponds to the degradation of Q, when the release rate of the encapsulated Q is slower than its degradation rate. In contrast, E microparticles showed only one zone corresponding to the release of surface E. The end of the induction period was in line with the exhaustion of Q and E and the initiation of formation of advanced oxidation products (polymers). In conclusion, Q microparticles have a potential application to extend the shelf-life of lipid matrixes.

The Impact of Diacylglycerol on Association Colloids Formation and Lipid Oxidation

Mizue Ouchi*[1], Eric A. Decker[2], and D. Julian McClements[2], *[1]Kao Corporation, Japan; [2]University of Massachusetts Amherst, USA*

Refined oil contains small amount of water and various kinds of surface active components such as phospholipids. In previous studies, our group reported association colloids such as reverse micelles are formed by these surface active components and affect lipid oxidation. Diacylglycerols (DAG), one of the minor components in refined oils, have different physicochemical properties from triacylglycerols (TAG) due to its structure. There are several studies on the influence of DAG on the oxidation stability. However, little is known about the impact of DAG on association colloids formation, and very few attempts have been made to retard oxidation using minor components for inhibiting association colloids formation. This study focused on how the DAG affect the ability of 1,2-dioleoyl-sn-glycero-3-phosphatidylcholine (DOPC) to form association colloids and lipid oxidation. The critical micelle concentration (CMC) and the mobility of phospholipid in different polar oils were measured by using TCNQ solubilization technique, interfacial tension, and NMR measurement. Higher mobility of phospholipid was confirmed in high DAG concentration oils suggesting the possibility of DAG to inhibit association colloids formation. Oxidative stability was confirmed that DOPC at concentrations above its CMC was prooxidative regardless of the presence of DAG, while high DAG concentration oil could slightly retard oxidative rate when the CMC of the oil was above DOPC concentration in the oil sample. To find out the role of minor components would contribute to develop new strategy to improve oxidative stability.

PRO 4: Processing ABC—Part II

Chairs: Bruce Patsey, Oil-Dri Corporation of America, USA; and Kris Knudson, Crown Iron Works Co., USA

The Use of Controlled Flow Cavitation to Improve the Performance of Degumming, Refining and Biodiesel Operations Darren J. Litle[1], Peter Reimers[1], and Oleg Kozyuk[2], *[1]Arisdyne Systems, Inc., USA; [2]ASI Chief Technology Officer, Ukraine*

An overview of the application of controlled flow cavitation and compression-decompression jet atomization phenomenon for the intensification of chemical processing applications is presented. For vegetable oil acid degumming and/or neutralization reactions, the reasons for enhanced performance of the refining operation, reduced environmental impact, observed reduction in necessary acid and/or caustic addition as well as decrease in oil loss, potential savings in steam consumption and decrease in maintenance opex is discussed and industrial scale examples given. The efficient removal of residual soaps, phosphorus, ffa and metals while minimizing and in some cases even eliminating the need for water washing or silica addition is also described. Finally, the power of controlled flow cavitation to reduce catalyst consumption, increase throughput, and reduce monoglyceride content in finished biodiesel is also described.

Centrifuge Equipment in Fats and Oils Processing William Younggreen*, *Alfa Laval Inc., USA*

Abstract not available

Composite Adsorbent - Filter Aid for High Performance Oil Purification Li-Chih Hu*, David Gittins, and Nathan Dias, *Imerys Filtration Minerals Inc., USA*

Oil for food or oleochemical processes often contain impurities including soap, free fatty acid, metals (natural or catalyst), phospholipids, etc. These impurities need to be removed to meet the quality and stability requirements downstream. In this paper, we describe a technology platform in which filter aid substrate, adsorbent coating and reactant (loaded to react with impurity) are combined. In an adsorbent treatment and filtration process, they provide synergy of high impurity removal efficiency, longer filtration cycle time, higher flow rate, oil yield, as well as reduced material usage and waste disposal. Examples of edible oil, biodiesel feedstock and byproduct, and frying oil purification will be discussed.

Low Color Bodies During Bleaching Impacting Refining Cost Jorge Bello*, *EP Engineered Clays, USA*

During the bleaching the importance of removing efficiently color bodies play an important role in the total cost, many times this cost scenario depends on the installed capacity of processing for the different unitary operations. An extended bleaching and filtration capacity could potentially unlock the associated cost of processing during the deodorization step,

particularly in soft oils. In this abstract, industrial and lab results are evaluated to determinate the cost of bleaching and how a low red color specification carries positive side effects across the whole process and cost of refining

Removal of Biofuel Feedstock Trace Elements by Bleaching Clays David Brooks* and Sajo Naik, *Oil-Dri Corporation of America, USA*

Studies were carried out treating two biofuel feedstocks comprised of waste tallow and waste vegetable oil with montmorillonite and palygorskite mineral based bleaching clays (1.0 to 2.0% g clay /g waste oil). Trace elements including iron, calcium, magnesium, and phosphorus were monitored using inductively coupled plasma spectrophotometry to compare adsorptive performance using methods adapted from official AOCS methods. Bleaching performance was positively impacted by the percent free moisture present in the feedstock blend at time of sorbent addition. No significant trends were established between typical clay characteristics and trace element reduction across the feedstock blends at constant dosage. The most impactful trend was observed within the palygorskite clays between total element reduction and observed pH acidity.

Dry Condensing System; Why all this Sudden Interest in this Technology? Sascha Wenger-Parving*, *GEA Process Engineering, Denmark*

Vacuum system in the oil deodorization process is often an underestimated part of the whole process, impacting the refined oil quality and very much production and operation costs due to increasing energy costs Today's main objectives for a vacuum system 1. Remove the stripping steam from the deodorization process 2. Remove the air load from the deodorization process equipment 3. Create the necessary vacuum level Tomorrow's main objectives for a vacuum system 1. Operational cost efficiency 2. Minimize steam consumption for ejector system 3. Reduce stripping steam in the deodorizer 4. Reduce deodorization temperature 5. Reduce waste water and other effluents 6. Reduce formation of Glycidol Esters and 3-MCPD Esters One topic that has gained a lot of attention recently when addressing deodorization process is the formation of GE's and 3-MCPD's. This topic is widely discussed in the edible oil production and intensively in the dominant Palm Oil producing countries in SAE This paper explains one simple way to combine all the above points under one single cost-effective solution with an attractive return of investment of approximately 3 years. First, stripping steam along with the FFA's carry over from the scrubber are cooled and frozen in a vacuum vessel at ca -30°C. With this step, the massive volume normally going to the traditional vacuum system is removed from the process and only air from the leakages is left. Those air leakages, also called non-condensable are afterwards been taken care by small vacuum system either by ejectors or by mechanical pumps This alternative technology can save up to 9/10 of the original steam amount compared to a traditional barometric solution In case of waste water, the amount to be treated will be the same amount of evacuated stripping steam Focusing on the oil quality, by lowering the absolute pressure, or creating higher vacuum, refineries will be able to either lower the striping temperature or the amount of stripping steam for the deodorization process. Eventually a bit of both.

What is Your Best Deodorizer Choice? Developments in Deodorization Process Leon Pablo Espinosa*, *Desmet Ballestra, USA*

Deodorization is a crucial refining stage that impacts refined oil quality. It has three main objectives 1. Stripping of volatile components such as free fatty acids (in case of physical refining), valuable minor components (tocopherols, sterols, etc.) and contaminants (pesticides, light PAH, etc.); 2. Removal of different off-flavors; 3. Thermal destruction of pigments (so-called heat bleaching). There are many factors affecting the final oil quality that are critical before starting the refining process. These factors are important to consider when choosing to purchase a new deodorizer. Today, the food oil industry is under a lot of scrutiny. With the advancement in analytical technology, we can measure many components that we couldn't in the past, and consumers are looking for healthy food free of contaminants. The food being produced is analyzed in detail and “new” components like PAH, Dioxins, 3MCPD, GE’s, and mineral oil are being found in food oil. This is leading to consumers asking for new regulations. Some of these contaminants are coming from the environment, while others are formed during the processing of the seeds and the refining process. In an effort to meet the needs of the consumer, new process conditions, and in some cases, new technologies will be required by the industry. . One topic that has gained a lot of attention recently is the formation of GE’s in oil. This paper offers some mitigation strategies to treat GE’s in the deodorization process.

New Developments in Physical Refining and Removal of Contaminants in Edible Oils Perry Alasti*, *Artisan Industries Inc., USA*

Edible oils are processed to remove impurities and contaminants to produce a final product that meets regulatory standards and possess acceptable palatability and shelf life. Crude oils normally contain some non-triglyceride components and other impurities that are removed at each processing step. These components include free fatty acids (FFA's), phospholipids, color components, peroxides, carboxylic compounds, metals and toxic environmental contaminants such as PCB's, dioxins, furans and pesticides. Most Edible oils undergo four basic processing steps: Refining/Neutralization, Bleaching and Deodorization. The last step in edible oil processing is usually deodorization, used mainly to remove undesirable odors or flavors and other contaminants such as peroxides, free fatty acids and partial removal of environmental contaminants. A new “thin-film” processing method is developed that combines deodorization and removal of toxins such as PCB's and dioxins to produce a highly pure oil

An Alternative Filtration in Bleaching Tony Dinsbach*, *Filtration Group BV, The Netherlands*

A cricket filter as alternative filtration technique in the bleaching application. For many year a pressure leaf filter is used in the bleaching application for edible oils. A cricket filter is used for many years in many other applications. The cricket filter is used already in edible oil as polishing filter and as hydrogenated step. The cricket filter can create cost savings and a more

effective filtration in bleaching step. We will present the differences between the systems and how end users can benefit from this other filtration technique.

Glycidyl Ester Formation and Mitigation
Linsen Liu*, Stephen Lumor, and Tri Le, *IOI Loders Croklann, USA*

Glycidyl ester is one of the process contaminants in refined oils. This presentation reviews its formation conditions and mitigation methods.

Results of Experiments to Reduce MCPD and Glycidyl Esters in Edible Fats and Oils by Ionic Liquid Treatment
Frank Pudel*[1], Jennifer Heymann[2], and Bertrand Matthäus[3], *[1]Pilot Pflanzenöltechnologie Magdeburg e.V., Germany; [2]Evonik Creavis GmbH, Germany; [3]Max-Rubner-Institut, Germany*

After the first communication of MCPD esters in edible fats and oils in 2007 and later of glycidyl esters in 2008 a lot of research has been done to elucidate the reaction pathway(s) and to find mitigation strategies to reduce the formation of these contaminants during oil processing. The mitigation strategies can be divided into a) avoiding or removing of precursors during all steps of the value chain up to refining, b) process changes during refining and c) removing of the formed esters after refining (post refining processes). Nowadays, a multitude of different methods to reduce MCPD and glycidyl esters are known and partially installed in oil mills and refineries, like—use of suitable crude oils, low in DAG and ffa—use of chemical refining instead of physical refining - avoiding low pHs—use of significant increased amounts of bleaching clay—use of natural bleaching clays—use of low temperatures in deodorizing—use of 2-step deodorizing - use short path distillation - post bleaching with different adsorbents - double bleaching/deodorization. Beyond that, Evonik Creavis developed a new process to reduce precursors in crude oil by ionic liquid treatment, leading to very low MCPD and glycidyl esters in refined palm oils. The results of experiments in laboratory scale will be presented and discussed.

PRO 5: New Technologies for Oil Processing

Chairs: Darren J. Litle, Arisdyne Systems, Inc., USA; and Gijs Calliauw, Desmet Ballestra Group, Belgium

Enzymatic Degumming with Expanders for Maximum Oil Yield Steve Gregory*, *DSM, USA*

Enzymes are the current cutting-edge technology for vegetable oil degumming. When enzyme degumming is paired with an expander for oilseed preparation, the economic results are synergistic. Expanders have many benefits for oil mills but the yield gain they provide when used with enzyme degumming is not widely recognized. This presentation will discuss the additional economic and processing advantages of using enzyme degumming with expanders as well as the individual advantages of both types of processes.

Recent Developments in Hydrodynamic Nanocavitation in Oil Refining: The Next Best Thing Marc J. Kellens*, *Desmet Ballestra Group, Belgium*

Nutritional oil quality and food safety have become the key factors in determining the optimal processing route for a given oil or fat. The increasing range of unwanted minor-components found in edible oils, together with a continuous lowering of their acceptance limits, are challenging the refining industry to constantly seek for new solutions and continuously adapt its processing route. On top, environmental and economic concerns drive refineries to become more sustainable and cost-efficient. With a vegetable oils and fats market being dominated by palm oil and soybean oil, it is quite evident that most new solutions are tailored on those two major oils. The shift from conventional chemical to physical refining is further stimulated with the introduction of new and more performing enzymes, especially in degumming. But at the same time, novel technologies like hydrodynamic nanocavitation are favouring the chemical route, especially when oils of varying quality are to be processed or very mild refining conditions are needed, to prevent or minimize side reactions typically linked to high temperature treatments. The soapstock treatment is today still a big concern for most chemical refineries, also but also here new eco-friendly and cost-efficient solutions are being developed to convert soapstocks into more value-added oil streams, which can then be further processed into biodiesel or other oleochemicals. Hydrodynamic cavitation in chemical oil refining has become today a well proven state of the art technology. New applications for this intriguing technology are being developed and expected in other oil processing stages soon.

Elimination of MCPDs and GEs in Edible Oils Joseph Rongione*, *Stepan Company, USA*

MCPDs (3-monochloropropane-1,2-diol and its esters) and glycidyl esters (GEs), have been a cause of concern for global regulatory bodies such as EFSA and WHO. In response to this Stepan has developed a process to eliminate these species from oil streams. This technology removes MCPDs and GEs by converting these species into glycerides. This technology has been used for several years in MCT (medium chain triglycerides) production. In this process, the MCPDs and GEs are eliminated during the

production of MCTs. More recently, this technology has been utilized to treat vegetable and marine oils as part of the overall oil refinement process. A review of this chemistry will be presented, focusing on MCPD/GE levels, stability of positional distribution and effects on double bond integrity.

Dry Condensing in Oil Refining: Latest Improvements to Further Enhance Oil Quality, Plant Safety, Process Efficiency and Overall Sustainability John A. Weston*[1], Jelle L. Nijdam[2], and Pieter Jellema[2], *[1]Desmet Ballestra, USA; [2]Solutherm, The Netherlands*

Increasing demands for higher quality, healthier oils require mild deodorisation temperatures, in combination with lower pressures in the deodoriser. These demands, along with a growing need to minimise energy and environmental impact of processes, have resulted in a clear tendency towards dry condensing systems for vacuum preservation in deodorisers. All available dry condensing (DC) systems make use of a refrigeration plant for the discharge of process heat at a low temperature. DC systems generally use ammonia as a refrigerant, which is in some areas not permitted for safety reasons. Other refrigerants could usually not be applied in dry condensing systems, for technical reasons. Recently a new, highly versatile, DC system has been developed and introduced to the market, which has been improved from various perspectives, all aiming at higher quality, healthier oils, in combination with the highest possible demands for safety and energy efficiency. This so-called 2G Sublimax DC system is suitable for all commonly applied industrial refrigerants, with the lowest possible refrigerant mass. The vertical condenser orientation permits safe refrigerant drainage, by gravity. The process vapours enter the shell of the condenser, initially in-line with the pipes and without any obstructions, minimising pressure losses. The new condenser has been designed for high fat depositions, permitting the omission of an intermediate scrubber, in case of mild (post) refining of temperature sensitive oils in a ultra-low pressure deodoriser, at pressures down to 0.5 to 1 mbar. This provides a new economically attractive alternative to short path / wiped film evaporators.

New Applications for Controlled Flow Cavitation in the Refining of Edible Oils Dr. Peter Reimers*[1], Oleg Kozyuk[2], and Darren J. Litle[1], *[1]Arisdyne Systems, Inc., USA; [2]ASI Chief Technology Officer, Ukraine*

An overview of newly developed applications of controlled flow cavitation and compression-decompression jet atomization phenomenon for the intensification of refining processing applications is presented. For vegetable oil enzymatic degumming, the reasons for enhanced performance of the refining operation, reduced environmental impact, observed reduction in necessary retention time as well as reduction in necessary enzyme addition, potential savings in steam consumption and decrease in maintenance OPEX is discussed and lab scale examples given. The efficient removal of phosphorus and metals while minimizing and in some cases even eliminating the need for silica addition is also explained. Finally, the power of controlled flow cavitation to reduce toxic components in refined palm oil is also described.

Soy and Other Protein Concentrates Alexander M. Danelich*, *Crown Iron Works, USA*

Soy Protein Concentrate (SPC) has been available to the market since the early 1960's for Human Consumption and Specialty Feeds through the use of Hydrous Ethanol Extraction. In the early 2000's, SPC for aqua feed became a growing market. Since then this market has grown to over 600,000 metric tons per year with plans to expand further. Profit margins for SPC are generally significantly higher compared to those for soybean meal. This paper will discuss overview of SPC, uses of SPC, importance of SPC for aqua feed market, as well as the Hydrous Ethanol process to manufacture SPC for Human & Feed applications. Discussion will also include some of the important considerations and challenges in plant design as well as design considerations that should be taken for specific pieces of equipment. The paper will also discuss the possibility of applying the Hydrous Ethanol Process to other oilseeds.

Enzymatic Gums Deoiling Antonios Papastergiadis*,*Desmet Ballestra Group, Belgium*

Abstract not available.

PRO 5.1: General Processing

Chairs: Mohammad Alam, Texas A&M University, USA; and Richard Clough, Texas A&M University, USA

Selectively Extracting Triglycerides by Chain Length and Saturation from Microalgae with Supercritical Carbon Dioxide Thomas A. Kwan*, Qingshi Tu, and Julie B. Zimmerman, *Yale University, USA*

Objective/Hypothesis: Development of clean technologies presents significant opportunities for economic growth as well as environmental and societal benefit by advancing a renewable chemicals industry. Triacylglyceride (TAG) lipids are attractive for biorefining as they are used for biofuels, renewable chemicals, and dietary supplements based on carbon chain lengths and degrees of unsaturation. Supercritical carbon dioxide (scCO2) has been heralded as a green technology for lipid processing because it is non-toxic, has high selectivity for TAG, and is suitable to produce edible extracts. scCO2 can be tuned to simultaneously extract, fractionate, and enrich different classes of TAGs, including mono- and poly-unsaturated fatty acid glycerides. Methods: TAG from microalgae was continuously extracted and collected using scCO2. Samples were analyzed using LC-MS and GC-FID for composition and software was used to predict energy needs for additional purification. Results: Solubility's of TAG can be enhanced or diminished in the presence of dissimilar TAGs due to solute to solute interactions and scCO2 densities. These modified solubility parameters provide a control handle to enrich extraction mixtures based on saturation and chain length. Additionally, energy impacts for downstream purification were modeled and provide insight to the potential trade-offs of over and under enriching certain TAGs during the biomass extraction process. Conclusion: Both the experimental and modeling results demonstrate the potential of scCO2 to be used as a green technology whereby the associated energy and economic costs of downstream TAG processing can be reduced. This study provides a significant step towards a viable biorefinery and ultimately a biobased economy.

Formation of 3-MCPD and Glycidyl Esters in Palm Oil using Different Laboratory-scale Refining Units Biow Ing Sim and Chin Ping Tan*, *Universiti Putra Malaysia, Malaysia*

Laboratory-scale refining processes are often used in research and development projects including 3-MCPD esters (ME) and glycidyl esters (GE) studies by oil industry. The present study involved a comparison of both micro (0.1 kg) and macro laboratory-scale (1 kg and 3 kg) refining units on formation of ME and GE formations as well as the final oil quality: free fatty acids (FFA), peroxide value (PV), oxidative stability index (OSI), and colour. Both refining units were capable of producing oil with acceptable quality in terms of colours, FFA and PV. The micro-scale refining unit produced oil with lower ME and GE contents compare to that produced by a macro-scale refining unit. Besides, the oil from the micro-scale refining unit was found to have a lower oxidative stability due to high ratio of surface to volume in contact with the glass vessel surface which catalyses the oxidation of oil.

Reduction of Toxins in Fish Oil from ppm to ppb in a Specialized Passive Stripper at Micron-level Vacuum Caitlin A. Davis*, *Artisan Industries Inc., USA*

Artisan Industries manufactures gravity-fed disc-and-tray stripping columns which are used to remove undesirable trace organics in oil products. In most cases, residual concentration specifications in the ppb range can be met with the normal equipment design. In the case study to be presented, relatively low vapor pressure toxins were successfully removed from fish oil by modifying the stripper design for continuous operation at 0.1 torr, permitting trace contaminant removal at temperatures below the threshold at which decomposition becomes a risk. The equipment employed was an Artisan Evaporator/StripperTM modified with low pressure drop chimneys, along with a Jet-Vac® multi-stage steam jet vacuum system. Neither the stripper nor the vacuum equipment have any moving parts and both are easy to clean, providing the combined advantages of low product loss, extended campaign times, and fewer maintenance intervals.

Synthesis of Esters from Chia Seeds Mucilage and Saturated Fatty Acids of Irvingia gabonensis Kernels Sidrine Kerthy Koumba Ibinga[1], Gwendoline Gravé[1], Jean-Francois Fabre[2], Eric Lacroux[3], Muriel Cerny[4], Romain Valentin[5], Raphaël Bikanga[6], and Zéphirin Mouloungui[4], *[1]INP - ENSIACET, France; [2]LCA UMR1010 INRA-INP/ENSIACET, France; [3]Chimie Agro-Industrielle, France; [4]Laboratoire de Chimie Agro-Industrielle, France; [5]INRA, France; [6]USTM, France*

Chia seeds (Salvia hispanica) and Irvingia gabonensis (IG) kernels, offer a wide range of food, pharmaceutical and industrial applications thanks to their specific chemical composition. For instance, the mucilaginous chia seed is rich in polysaccharides, polyunsaturated fatty acids and proteins and the kernel of IG has a high content of saturated fatty acids (SFA). The aim of this work is to valorize carbohydrates of chia seed mucilage (CSM) and SFA from IG by esterification leading to fatty esters. CSM is extracted by ultrasonic cavitation in water [1] and characterized. CSM contains approximately 81% of polysaccharides, 6% of proteins and 9% of minerals. The oil content of IG determined by soxhlet extraction is 63.78±0.19%. SFA content is 97.60±0.045% and the fatty acid profile shows that myristic (51.87±0.18%) and lauric (38.48±0.09%) acids are the most abundant. Esterification feasibility of sucrose with SFA [2,3] of IG with an optimized process developed in our laboratory predicts the esterification of carbohydrates from CSM with SFA from IG. Products were characterized by IR and NMR spectroscopy and chromatographic methods. Sucrochemistry of carbohydrates of CSM by O-acylation with SFA of IG kernel could lead to the synthesis of new compounds that would find applications as emulsifying, texturing, complexing agents... Keywords: Chia, Irvingia gabonensis, ultrasounds, mucilage, carbohydrates esters, surfactant. 1. Fabre, J.-F. et al. Ind. Crops Prod. 65, 354–360 (2015). 2. Lemaire-Claverie V. et al., US Patent No 6,706,877 B1 (2004) 3. Mouloungui, Z. et al. La lipochimie. In La chimie verte, 305-356 (2006).

PRO-P: Processing Poster Session

Chairs: Ozan N. Ciftci, University of Nebraska-Lincoln, USA; and Ali Ubeyitogullari, University of Nebraska-Lincoln, USA

1. **Enzymatic Preparation of Food and Medical Materials from Fish Oil.** Kazumi Katagiri[1], Yomi Watanabe[2], Ryosuke Hoshina[1], and Hideaki Kobayashi[1], *[1]Kewpie Corporation, Japan; [2]Osaka Research Institute of Industrial Science and Technology, Japan*

The purified C20:5 (EPA) ethylate is the only PUFA approved as medicine in Japan for hyperlipidemia and obstructive arteriosclerosis, whereas C22:6 (DHA) are supplied as food. In general, the two are concentrated and purified from FFA or FAEE chemically prepared from fish oil, where the separation of EPA and DHA by the rectification is not very efficient due to the similar properties of the two. In addition, DHA should be reconverted to glyceride form in order to supply as food. Therefore, an enzymatic process was studied to recover DHA as glycerides and EPA as ethylate. Treatment of sardine oil (EPA and DHA contents: 18 and 12%) with ethanol using lipase RMIM (Novozymes) at 5˚C for 24 h reached nearly 65% conversions. The resulting oil was distillated. FAEE with EPA content of 19% (recovery, 65%) and glycerides with DHA content of 33% (recovery, 92%) were recovered. The glyceride fraction consisted mainly of DAG and MAG (80%). To the DHA rich glyceride, middle chain FA, that is known to easily be digested, was introduced to reconvert to TAG by the same lipase under vacuum. After the removal of unreacted MCFA by distillation, glycerides consisted of 85% TAG, 10% DAG, and 5% MAG were obtained. MCFA and DHA contents of the glycerides were 18 and 22%, where 60% of DHA was located selectively at *sn*-2position by the regiospecific analysis of FA distribution using *Candida antarctica* lipase.

2. **High Intensity Ultrasound and Bubble Dynamics does not Affect the Oxidative Stability of Soybean Oil.** Juhee Lee[1], Roberta Claro da Silva[2], Peter Birkin[3], Tadd Truscott[1], and Silvana Martini[1], *[1]Utah State University, USA; [2]North Carolina A&T University, USA; [3]University of Southampton, United Kingdom*

The aim of this study was to examine the effect of high intensity ultrasound (HIU) on the oxidative stability of soybean oil by measuring peroxide value (PV) and oil stability index (OSI). Samples ([5, 100, and 250] g) were sonicated at 25 ºC using various sonication conditions: power level (setting 1, 5, and 9), tip size (diameters of [12.7, 3.2, and 2] mm), and pulse duration ([5, 10, and 60] s). A range of absolute power ([0 to 123] W), power density ([0 to 14] W/cm3), and power intensity ([0 to 1100] W/cm2) values were obtained based on the volume of the samples, power setting, and tip size. Highest power density and power intensity values were obtained with the highest HIU setting and with the 3.2 mm tip. No significant difference ($p < 0.05$) was observed in PV nor OSI among the various sonication conditions tested. PV of samples was 0.22 +/–0.02 mEq/kg and OSI was 10.05 +/– 0.04 h at 110 °C. The IDF standard method was used to determine the PV, and the AOCS official method (Cd 12b-92) was used to determine OSI. Bubble formation and dynamics under these sonication conditions were monitored using a hydrophone and a laser

scattering set up. Bubble formation was affected mainly by power level and not by pulse duration under the conditions employed. These results indicate that the oxidative stability of soybean oil was not affected by HIU, under these conditions, even when highest power levels, densities, and intensities were used to change the bubble dynamics in the system.

3. **Method of Miscella Zenith Deacidifying Process Research.** Zhao Chenwei[1], Weiguo Qin[2], and Jinfeng Qi[3], *[1]State Key Laboratory of Food Science and Technology, School of Food Science and Technology, Jiangnan University, China; [2]COFCO Engineering & Technology Co., Ltd., China; [3]Jiangsu University of Science and Technology/Jiangnan University, China*

This experiment combined zenith refining with miscella refining together in order to illustrate that through zenith refining method we can get high acid value oil as well, simultaneously possessing the advantages of miscella refining method which has features of lower rate of neutral oil saponification, fewer oil soap stock entertainment and better effect of refining, etc. By single factor experiment, we have studied not the impact parameters during processing of deacidefication and reaction yield, but optimizing the processing conditions which are in terms of two indicators - the acid value of product and the yield of reaction, the optimal conditions were determined by orthogonal experiment as follows: reaction temperature 65°C height 1400mm, miscella concentration 60%, alkali concentration 0.1 mol/L, feed aperture 0.5 mm, puckering feed flow rate 1.0mL/min. Under this condition, the acid value of the product was 0.27mgKOH/g, reaction yield was 92.0%, acid value of cottonseed oil reached two standards.

4. **Effects of Heat Pretreatment of Wet-milled Corn Germ on Physicochemical Properties of Oil.** Liyou Zheng[1], Jianhua Huang[2], Jun Jin[3], Xingguo Wang[3], and Qingzhe Jin[3], *[1]State Key Laboratory of Food Science and Technology Synergetic Innovation Center of Food Safety and Nutrition School of Food Science and Technology, China; [2]School of Food Science and Technology, Jiangnan University, China; [3]Jiangnan University, China*

Objective Wet-milled corn germ is a value-added co-product generated during the starch production as it contains over 30% oil. Traditionally, such oil is removed from the milled germ using a conditioning (heating) process prior to oil extraction. The present study aims to investigate the effect of oven roasting (OR) and microwave (MW) radiation on crude oil as a pretreatment. Methods Used Three OR temperatures (125, 150, and 175°C) were used with 60 min exposure, and MW pretreatments were established by combining two powers (440 and 800 W) and three pretreatment times (4, 6, and 8 min). Chemical composition like fat compositions, triacylglycerol species, and tocopherol and phytosterol levels were determined. Results Significant increases were observed in the levels of 1,3-diacylglycerol and total diacylglycerolincreased substantially ($P < 0.05$), while triacylglycerol experienced a significant decrease ($P < 0.05$) during OR. There were no significant differences among triacylglycerol compositions following OR and MW treatments. Both heat pretreatments dramatically increased ($P < 0.05$) the total tocopherol content. δ-Tocopherol showed minimal changes, while β-tocopherol progressively increased during the heat treatments. No significant differences ($P > 0.05$) of phytosterols content were observed among

most samples tested, along with it first slightly increased and then decreased during pretreatments. Conclusions Proper roasting temperatures or MW radiation times could enrich the content of individual tocopherol and phytosterol. Data analysis revealed greater alterations occurred in a conventional oven, compared to heating by MW.

5. **Quantitative Isolation of Cannnabinoids from Plant Material.** Albert J. Dijkstra and Robert W. LaChance, *FAOCS, France*

Quantitative isolation of cannabinoids from plant material Current cannabinoid isolation processes use extraction solvents like butane, carbon dioxide or alcohol. Besides cannabinoids, these solvents extract many other compounds from the plant material such as terpenes, sesquiterpenes, oils, waxes, colouring compounds, sugars, and many other, as yet unknown compounds. Accordingly, the extracts obtained by current extraction processes require extensive purification. Purification means additional costs, especially because of product loss. A novel isolation process has been developed that ensures near quantitative isolation of cannabinoids from plant material at much higher rates than all popular methods. In addition, the cannabinoids thus obtained have a higher purity than those obtained by the current processes. A patent has been applied for. The novel process extracts the cannabinoids from the plant material by using a low-volatility solvent like vegetable oil. Subsequently, the oil containing the cannabinoids and other non-polar plant material constituents is subjected to a vacuum stripping process while the oil is being heated. This stripping process causes volatile compounds to be removed from the oil in order of their volatility. So water is removed first, followed by terpenes and finally the cannabinoids. Separate collection of the fractions ensures their purity. There is no need to continue the stripping process to very low residual cannabinoids levels in the oil since the oil can be recycled after the plant material has been removed by filtration. Accordingly, any cannabinoids still present in the oil will be retained in the system for future recovery by vacuum steam stripping.

6. **CLA-Rich Vegetable Oil Production by Adapting a Small Scale Hydrogenation Reactor.** Chaun Min Ruan[1], and Andrew Proctor*[2], *[1]University of Arkansas, USA; [2]Dept. of Food Science, University of Arkansas, USA*

The various health benefits of conjugated linoleic acid (CLA) and its use in polymer materials have been studied for many years. There have been many methods proposed to produce CLA fatty acids, including fermentation and alkali oil treatments. Production of CLA-rich triacylglycerides has been proposed by transesterification, homogeneous catalysis and heterogeneous catalysis. However, many of these methods are time consuming expensive and tedious. The objective of this study is to develop a rapid, simple to produce CLA-rich soy oil using an oil hydrogenation reactor and commercial nickel hydrogenation catalyst, in the absence of hydrogen. A series of experiments were conducted with a model 4530 Parr small scale hydrogenation reactor, Pricat 9925 nickel catalyst and once refined soy oil with variable amounts of formic acid and cysteine. A 20% CLA-rich oil was produced from 500mL of oil with 0.5% catalyst, 0.25% formic. 0.01% cysteine for 2 hours at 210°C with a nitrogen pressure of 3 atmospheres

and 1200 RPM mixing speed. However, increasing the catalyst dose increased both CLA levels and trans-fatty acids but trans-fat formation occurred at a much slower rate. A 25% CLA-oil was produced with >10% trans-fat from 220 ml of oil by using a longer processing time of 5 hours at a lower temperature of 175° C with a stirring rate of 900 RPM under nitrogen. While the study shows in principle the production of CLA-rich oil in small scale hydrogenation reactor, larger batch production in industrial reactors is needed to show commercial viability.

7. **Large Scale Purification of Minority Fatty Acids by Liquid Chromatography: The n-3 docosapentaenoic Acid Case.** Gaëtan Drouin, Etienne Guillocheau*, Daniel Catheline, Vincent Rioux, and Philippe Legrand, *Agrocampus-Ouest, France*

Some fatty acids of interest present in natural or synthesized sources are not commercially available at high purity in sufficient amounts, or at affordable prices to allow in vivo studies of these molecules. The purpose of this study was to purify on the scale of a few tens of grams of n-3 ethyl docosapentaenoic acid (n-3 DPA) in the laboratory. The purification was performed by crystallization following by flash and preparative liquid chromatography. The optimization of the n-3 DPA source, the columns used, the elution, the volume and the injection method, the purification process and the associated logistics were carried out. Several tens of grams of n-3 DPA were obtained with a purity and an efficiency greater than 99% and 80%, respectively. Starting from a high Omega-3 content marine oil, one step of crystallization and seven successive steps of liquid chromatography coupled with an UV-detector were necessary. 20 ml of oil mixed with 15% methanol were injected over an original system consisting in two-coupled C18 flash columns (45 µm with high capacity, and 15 µm with high performance). The elution was operated at 120 ml/min (constant flow rate), with an isocratic methanol/water mode which proportions depended on the purification step. These conditions allowed successive injections without waiting for the end of the chromatogram. This method could be applicable to the separation of all polyunsaturated and monounsaturated fatty acids.

8. **Temperature Controlled Pulsed Light Treatment: Impact on Aflatoxin Level, and Quality Parameters of Peanut Oil.** George Baker[1], Basheer Iqdiam, Manal Abuagela, and Andrew MacIntosh, *University of Florida, USA*

Peanuts are economically significant crops that are prone to contamination by aflatoxin (AFT) producing fungi. Common methods to treat oils often utilize high temperatures (> 100°C) to thermally degrade AFTs, however, high temperatures often negatively impact product quality. In this study, pulsed light (PL) was assessed as a possible method to degrade AFTs in peanut oil. A pilot-scale PL applicator was used to treat 5 mL samples of peanut oil 10 mm thick. Samples were treated for 1 to 10 min with and without temperature control (TC). Ice was used to maintain temperatures less than 50°C in TC samples. The quality of treated peanut oil was assessed through chemical quality indices (peroxide value, free fatty acid, and acidity value), color, and oxidative stability index (OSI), in comparison to controls. Results show that significant AFT destruction was achieved using PL. There was a significant difference between

the reduction achieved when TC was employed, likely due to the combined effect of high temperature with PL. TC samples treated for 4 min resulted in a reduction of 44% while those treated 6 and 10 min experienced a reduction of 80% and 84%, respectively. The measured quality parameters of all TC samples showed no significant difference from controls in the quality indices, color parameters, and OSI for all treatment times. This novel method resulted in comparable toxin reduction, without loss of quality typically associated with traditional thermal methods. At industrial scale, PL may offer an economical treatment of AFT contaminated oil.

9. **Development of an Extraction and Purification Process to Obtain High Purity Wax from Sorghum.** Junsi Yang, Loren Isom, Curtis Weller, and Ozan N. Ciftci, *University of Nebraska-Lincoln, USA*

There is a growing demand for alternative natural wax sources due to growing industrial, food, pharmaceutical, and cosmetic applications of waxes, and lower wax supply. It is projected that global wax demand will grow at a rate of 1.5% until 2019, whereas wax supply growth will be only 0.4%. Sorghum is a promising alternative natural wax source. However, there is no specific processing method to obtain high purity sorghum wax that meets natural wax industry specifications. Without such methods, sorghum wax's potential will remain untapped. Therefore, the objective of this study was to develop an improved processing method to obtain a high purity wax from sorghum. Sorghum kernels were extracted with hexane and supercritical carbon dioxide (SC-CO_2) and the extracts were purified by a simple ethanol washing process. Wax yield was 0.11 wt.% from hexane extraction, whereas it was 0.39 wt.% from SC-CO_2 extraction at 30 MPa/70ºC. Following hexane extraction, the melting point of the sample after purification by degumming and winterization was 75°C; however, it improved to 80°C with the ethanol wash. Moreover, our ethanol washing process improved the hardness (needle penetration method) from 40 to 2, which is the hardness value of Candelilla and Carnauba waxes. Ethanol wash decreased acid and iodine numbers (USP 401) from 20 to 3.3, and from 59.4 to 23.6, respectively. Our simple process minimizes processing steps, reduces solvent and energy requirements, and generates superior quality sorghum wax, which may reduce U.S. dependence on non-renewable petroleum-derived waxes or natural wax imports.

10. **Concentration of Lignan from Defatted Sesame Meal by Supercritical Carbon Dioxide Two Step Process.** No Young Kim[1], Heejin Kim[2], Nakyung Choi[1], Jong Hun Choi[3], Chulyoung Lee[3], and In-Hwan Kim[1], *[1]Korea University, Republic of Korea; [2]Dept. of Public Health Sciences, Graduate School, Korea University, Republic of Korea; [3]R&D Center, Nongshim Republic of Korea*

Lignan, a powerful antioxidant in sesame oil was successfully concentrated from defatted sesame meal by fractional supercritical carbon dioxide (SC-CO2) in two-step extraction. In general, lignan content of commercial sesame oil is ca. 0.6%. However, in this study sesame oil with 3% lignan content was obtained in first two fractions by fractional SC-CO2 extraction at the pressure of 6500 psi and the temperature of 40°C. To further enrichment of lignan, the sesame oil with 3% lignan content was converted to fatty acid ethyl ester (FAEE) by lipase-catalyzed esterification. The esterified sesame oil was

extracted by SC-CO2 using different solubility between FAEE and lignan in SC-CO2. Lignan was effectively concentrated in residue by removal of FAEE from the esterified sesame oil. The effect of pressure and temperature was investigated between 1600-1800 psi and 40–60°C. Consequently, lignan-rich sesame oil with 25% lignan content was achieved in residue under the optimum condition.

11. **Quality Characteristics of Cold-pressed Flaxseed Oils from Cultivars in China.** Qianchun Deng, Xiao Yu, Qingde Huang, Fenghong Huang, Xiaoshan Wei, Xixi Zang, Peng Chen, and Luxi Meng, *Oil Crops Research Institute, Chinese Academy of Agricultural Sciences; Hubei Key Laboratory of Lipid Chemistry and Nutrition; Key Laboratory of Oils, China*

Objective: This study aimed to compare the quality characteristics of flaxseed oil from cultivars in China.

Methods: Thirty-two representative flaxseed cultivars were collected from the major planting areas in China. The bioactive compounds and in vitro antioxidant activities of cold-pressed flaxseed oil were evaluated according to variety and geographical origins using a chemometric study. Moreover, the antagonistic effect of optimized cold-pressed flaxseed oil against obesity-induced insulin resistance was investigated.

Results: Differential α-linolenic acid (35.83–58.91%), total phenolic acids (7.01–21.95 mg/100g), secoisolariciresinol (ND–0.300 μg/g), tocopherols (372.41–553.00 μg/g) and phytosterols (322.99–1029.71mg/100g) contents were observed among the thirty-two cold-pressed flaxseed oil samples. Concomitantly, the in vitro antioxidant activities varied from 1.73 to 6.15 mg TE/100g, 4.21 to 7.67 mg TE/g, 1.95 to 6.95 mmol TE/g as determined by the DPPH, FRAP and ABTS assays, respectively. Significant correlations were manifested between the in vitro antioxidant activities and contents of total phenolic acids, tocopherols and phytosterols ($p<0.01$). Cluster analysis allowed the flaxseed oil samples to be grouped based on the antioxidant activities and contents of bioactive components mainly in relation to variety specificity. Moreover, the in vivo studies indicated that the optimized flaxseed oil enriched with endogenous micronutrients greatly alleviated obesity-induced endoplasmic reticulum unfolded protein response inactivation, mitochondrial dysfunction and defects in mitophagy, paralleling with improved insulin resistance.

Conclusions: The selected flaxseed oil samples showed different biochemical properties due to genetic specificity, contributing to the differential modulation effect on obesity-induced insulin resistance.

12. **Quality and Safety Control Technology in Wood-pressed Rapeseed Oil.** Youfeng Zhang[1], Ruijie Liu[2], Qingzhe Jin[3], and Xingguo Wang[3], *[1]School of Food Science and Technology, Jiangnan University, People's Republic of China; [2]Jiangnan University/Cornell University, China; [3]Jiangnan University, China*

Wood-pressed rapeseed oil belongs to fragrant oil with wood pressing which is a traditional Chinese pressing craft for edible oil production. Manual roasting and natural static settlement are commonly used in wood-pressed rapeseed oil production without further industrial refining process, resulting in the rich fragrance and smooth taste of the final oil products. At present, there are few systematic studies available on the profile of wood-pressed rapeseed oil. Quality of wood-pressed rapeseed oil was systematically studied. In addition,

contamination sources of polycyclic aromatic hydrocarbons (PAHs) in the raw material, oil production and storage processes of wood-pressed rapeseed oil were investigated in this study. The results showed that the quality of wood-pressed rapeseed oil differed greatly. 75% samples' benzo[a]pyrene (BaP) and sum of BaP, benzo[a]fluoranthene, benzo[b]fluoranthene and chrysene (PAH4) concentrations exceeded the allowable limits set by the Commission Regulation of Europe (Eu). The main sources could be attributed to the over-roasting, pollution of high PAHs content contact substances and roasting fume. BaP and PAH4 levels declined slightly during the storage. Prevention and control measures of PAHs in wood-pressed rapeseed oil were proposed and implemented, which could make the final products meet the Eu.

13. **Study and Modeling of Oilseeds Continuous Pressing.** Houcine Mhemdi, *University of Technology Compiègne, France*

Continuous screw pressing is widely applied for mechanical extraction of oil from oilseeds. Despite significant recent advances in the field of press design and automation, the prediction of performance based on theoretical approaches remains difficult. This work is devoted to better understand, characterize and model the solid/liquid behavior in a screw press by combining experimental and computational approaches. For this purpose, a pilot (0–40 kg/h) screw press (Reinartz, Germany) was instrumented by installing 16 pressure sensors, 4 temperature probes and 2 balances. Canola seeds were used for investigation. The pressure, the temperature and the oil flow rate were measured and recorded experimentally throughout the press. Additionally, samples of press cake were taken in the different sections and analyzed (porosity, residual oil content). The experimental results showed the presence of successive sections of compression and mixing. An agreement was found between oil flow rates and pressure values in the compression sections. The total oil yield increased with lower rotation speed and the maximal oil yield was approximately 90%. The residence time distribution in the press was studied and showed an important oil recirculation and axial dispersion of mass flow through the screw press. These phenomena were taken account for oil expression kinetics modeling based on the filtration/consolidation theory. The model allowed the computation of the compressibility module, the consolidation coefficient and the specific resistance of the cake. Finally, the applicability of the model to predict the extracted oil flow and yield was verified and validated on a laboratory scale on the studied press.