

## PRO 1: Processing from Seed to Refined Oil

*Chairs: Farah Skold, Solex Thermal Science Inc., Canada; and Hans Christian Holm, Novozymes A/S, Denmark*

### **Cleaning, Cracking, and Dehulling Soybeans** Bill Morpew\*, *Crown Iron Works Company, USA*

The preparation process can be defined as the first step to creating value added products when starting with soybeans. In the case of oil separation, whether by pressing or solvent extraction, it is necessary to prepare the soybeans for the downstream process to attain product quality parameters dictated by the market while maximizing the efficiencies within our power to control. Cleaning the soybeans is often performed before the soybeans arrive at the processing facility, but we will discuss the need for further cleaning at the inlet of the preparation process, along with the various benefits. Cracking can precede or follow conditioning, and will change a little depending on this choice. Process parameters for cracking will be reviewed as they relate to the type of soybean conditioning and the desired product quality. Dehulling is the process of removing hulls for the purpose of controlling the fiber content of the soybean meal, and also indirectly adjusting the protein content of the meal. In dehulling, we have to consider both the effectiveness and the efficiency of the separation step.

### **Energy Recovery Optimisation** Farah Skold\*, *Solex Thermal Science Inc., Canada*

Oilseed crushing plants endeavour to reduce overall energy consumption in the process plant by recovering waste energy and finding novel methods of utilization using high efficiency heat exchangers. Where these energy recovery loops provide valuable savings, they need careful evaluation with respect to pay back as well as operating flexibility. This paper takes a look at

various typical energy recovery loops found in Preparation plant and how to best optimise the use of these low grade energy loops.

### **Modern Pressing Technology** Jon Hanft\*, *HF Press + Lipid Tech, USA*

The history and development of oilseed processing is characterized by the implementation of a number of important innovations. Apart from the transition from batch-type to continuous operations with regard to pressing and solvent extraction, the pre- and full-pressing processes have been modified for higher energy efficiency and oil yield. Screw presses not only grew in size and capacity but also incorporate modern shaft and cage designs, highly wear resistant materials, and innovative drive systems leading to today's high performance, heavy-duty equipment. The implementation of "Industry 4.0" techniques combines preventive maintenance ideas and routines for forward-looking stock-keeping of parts for maximum availability of the entire processing system.

### **Solvent Extraction** Timothy G. Kemper\*, *Desmet Ballestra, USA*

Solvent extraction is the most widely used process for extraction of edible oil from oilseeds such as soybean, rapeseed, sunflower, cottonseed and peanuts. This review paper will briefly describe the history of solvent extraction leading to today's technology. The common unit processes in a solvent extraction plant will be outlined including; solvent extraction, meal desolventising, toasting, drying and cooling, miscella distillation, solvent recovery and the mineral oil vent recovery system. Typical operating costs will also be presented.

**Recent Developments in Degumming of Oils and Fats** Robert Zeldenrust\*, *GEA, Germany*

The degumming of oils and fats is one process steps where the oil quality as well as the financial yield can be highly influenced. Important is to remove the undesired components, possibly create valuable by-products and to achieve a high yield. The process has to be adjusted to the feed stock quality as well as to the further processing and product use. The presentation will give an overview on the process set-up for different feed stocks and the recent developments in degumming technology.

**Current State of Adsorptive Bleaching, Materials, and Processes** Chris Mitchell\*, *Clariant Corp., USA*

As part of today's modern edible oil refinery, the bleaching process plays a key role in the removal of color bodies and other various pro-oxidants, leading to top-quality finished oil with pleasing, stable appearance and absent of undesired odors/flavors. In this short presentation we will examine the properties of various adsorbents and their removal capacity of targeted unwanted contaminants. Also, we will look at the most commonly used filter arrangements to accomplish the most efficient use of your adsorbents.

**Deodorization.** William Younggreen, *Alfa Laval Inc., USA*

*Abstract not available.*

**Fat Modification Technologies—Fractionation and Interesterification** Gijs Calliauw\*, *Desmet Ballestra Group, Belgium*

The aim of edible oil modification processes is to substantially change the physical behavior and structural properties of oils and fats, and it is very often the last processing stage before the oil is processed into foods, such as margarines, frying oils, shortenings, confectionery... This explains why in these processing technologies especially, harsh conditions as well as the use of solvents & chemicals is currently increasingly discouraged for these practices are perceived as possibly harmful and polluting. Those technologies are progressively being traded for 'greener' and more sustainable techniques, leading to healthier and better products as well. Two modification technologies in particular that meet such demands are dry fractionation and enzymatic interesterification. While the principles of those processes are well-known in the industry, the presentation aims at elaborating on the possibilities in operation modes for bulk and/or specialty fats, and highlights the potential and the limits of each approach. It also touches upon the combination of both technologies in contemporary state-of-the-art oils and fats applications such as structured lipids and cocoa butter alternatives.

## PRO 2: Enzymatic Processes—A User Perspective

*Chairs: Xuebing Xu, Wilmar Global Research and Development Center, China; and Krish Bhaggan, IOI Loders Crokiaan, The Netherlands*

### Enzymes to Improve OER in Palm Oil Extraction

Chandrakumar L. Rathi\* and Saylee Pradhan,  
*Advanced Enzyme Technologies Ltd., India*

The process of Enzymatic Extraction to increase the oil recovery was adapted for existing palm oil mills. Based on the structural carbohydrate composition of oil palm mesocarp (*Elaeis guineensis*), the effect of different combination of enzymes and treatment times on the oil extraction rate (OER) were studied. The enzymes degrade the cell walls of mesocarp & reduce its viscosity and also de-emulsify the pressed oil giving higher OER. It can deliver fermentable sugar solution as well. The fresh fruit bunches (FFB) of right maturity are harvested and transported to the palm oil mill. The FFB are sterilized and stripped to separate the fruits. The digester converts the loose fruits into semisolid pulp. The digested mesocarp is mixed with enzymes and is subjected to temperature of 30 deg C to 70 deg C at its inherent pH for period of 20 to 30 minutes before final mechanical pressing. The oil-water mixture from the press is separated in a clarifier, followed by centrifuge. The oil is finally vacuum dried. The oil content and viscosity of pulp, FFA of oil and sugar in water layer was measured. About 1 – 3% increase in OER is observed. The clarification process is also very fast.

### Enzymatic Synthesis of Biodiesel from Waste

**Cooking Oils** Li Deng\*, Meng Wang, Kaili Nie, Fang Wang, and Tianwei Tan, *Beijing University of Chemical Technology, China*

Waste cooking oils (WCOs), as its potential food security issues, are mainly been used as raw materials for liquid biofuels. However, the complex origins of WCOs making the traditional transformation methods difficult,. Therefore,

how to efficiently transform WCOs to high quality liquid biofuels with low cost was the main problem for the development of the technology. In order to efficiently transform WCOs to biodiesel and lower the catalyst cost, a new enzyme catalyzed biodiesel synthesis process with *Candida sp.* 99-125 lipase and assistant of the lipase was developed. The optimal conditions were established. The use of assistant lower the cost of the process, and the process were scaled up to 200 L, 5,000 L, 10,000 L, with the yield of 90.8 %, 90.2 % and 89.2 %, respectively. Furthermore, a low cost treatment process for enzymatic solution without distillation was developed. The cost of non-distillation process was 25% of the original process, and the product was used in low speed diesel engine for over 100 hours successfully.

### Biocatalysts in Refining and Concentrating PUFA Oils and Producing Advanced Bioactives

Jari Kralovec\*, Paul Mugford, Erick Suarez, and Zhuliang Tan, *DSM Nutritional Products, Canada*

Polyunsaturated fatty acid (PUFA)-rich oils, and especially those with high levels of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) are relatively unstable substances. The presence of methylene-separated cis double bonds (bis-allylic system) makes these oils vulnerable to the oxidation and thermal damage, as well as renders them sensitive to the presence of strong acids and bases. Although relatively cheap and very effective traditional chemistries to refine and execute molecular transformations of these oils were discovered, there have been considerable efforts to replace the conventional methods with appropriate biocatalysts. Similar attempts have also been seen with respect to the concentrating of

these oils to achieve higher EPA and DHA levels. The presentation will deliver the state-of-the art and perspectives of the field and demonstrate examples of unique applications of enzymes in refining and concentrating omega-3 triacylglycerides. It will also illustrate the use of these devices to accomplish clean molecular transformations with focus on hydrolysis, esterification, and transesterification mainly to generate bioactive products belonging to a variety of lipid classes (partial glycerides, ethyl esters, phospholipids, etc.). A balanced picture will be given and thus not only the advantages of enzyme applications will be emphasized but also common issues surrounding the use of biocatalysts will be discussed.

**New Concepts in Oil Degumming** Fang Cong<sup>\*1</sup>, Ulrich Sohling<sup>2</sup>, Kirstin Suck<sup>2</sup>, Yuanyuan Gao<sup>1</sup>, and Xuebing Xu<sup>3</sup>, <sup>1</sup>Wilmar Global R&D Center, China; <sup>2</sup>Clariant, Germany; <sup>3</sup>Wilmar Global Research and Development Center, China

Two new concepts for oil degumming were investigated. One is based on the use of 1,2 propanediol as additive for aqueous degumming, acid degumming or enzymatic degumming with Phospholipase A. The second concept is based on the addition of  $\alpha$ -amylase in aqueous degumming or enzymatic degumming with PLA. The existing method of oil degumming is mostly water degumming. However, the phospholipids transferred to the water serve as an emulsifier for emulsifying a portion of the vegetable oil in the aqueous phase. This leads to considerable oil and financial losses on an industrial scale. Lowering the aqueous phase viscosity helps to release the oil from emulsified gum. The crude gum contains 6-7% carbohydrate impurities (dry basis), with a significant thickening action. Use of 1,2-propanediol or amylase-assisted degumming can lower the viscosity of the gum phase and release the entrapped oil. In soybean oil water

degumming, 0.2% addition of 1,2-propanediol can help to improve the yield of oil by 0.3% -0.9% and reduce the viscosity of gum and also leads to 0.32-0.42% yield increase in PLA1 degumming compared to PLA1 itself. Similarly, addition of  $\alpha$ -amylase can also assist in degumming process to increase the oil yield in water degumming by 0.1%-0.5%, and in PLA1 degumming by 0.2-0.5%. After the whole refining process, the quality and stability of the refined oil from new degumming process and conventional process was evaluated. There was no significant difference between two processes on the oil stability (AV/PV/color) and the level of contaminants (3-MCPD&GE).

**Plant Scale Enzymatic FFA-remediation of Rice Bran Oil** Steen Balchen<sup>\*1</sup>, Prasert

Setwipatanachai<sup>2</sup>, Yee Hong Seng<sup>3</sup>, CheeKeong Tan<sup>4</sup>, and Lars Harild<sup>5</sup>, <sup>1</sup>Alfa Laval, Denmark; <sup>2</sup>Surin Bran Oil, Thailand; <sup>3</sup>Novozymes, Malaysia; <sup>4</sup>Alfa Laval, Malaysia; <sup>5</sup>Alfa Laval Copenhagen, Denmark

Enzymatically catalysed FFA-remediation of Ricebran oil, conducted with the constraint that the content of partial glycerides in product should not exceed the feed content, was carried out in a 3-party collaboration. Lab results were used to design a plant comprising a 4 ton batch vacuum reactor. Removal of water was done by means of spray drying as well as nitrogen sparging. CALB liquid enzyme solution was immobilised at site and during reaction either kept in suspension under vacuum or deposited under pressure in a "fixed bed" pulse tube filter while formed water was transferred to the drying zone by high oil flowrate. Currently 60+ batches were produced and FFA was reduced from 15+% to typically 6-7% within a 24 hr batch cycle time using a low enzyme concentration corresponding to 2% carrier. The enzyme lifetime is better than expected (30+ batches). Less than 3% residual FFA content can be obtained but requires extraordinary process conditions. The process economy would in many cases be attractive but the

significant production of high-melting glycerides (converted into stearin in case of winterisation) and geographically related legal and labelling issues have to be considered.

**Enzymatic Synthesis of Functional Lipids-phytosterol Esters with Controllable Fatty Acid Composition** Mingming Zheng\*, *Oil Crops Research Institute, CAAS, China*

**Objectives:** The fatty acid composition linked with phytosterol esters (PEs) could influence the activity of sterol esters significantly and has gained much attention. The objective of this study is to develop a strategy for esterification of phytosterols with different beneficial fatty acids (FAs) to produce PEs with controllable FA composition. The immobilized lipase, which employs hyper-crosslinked polymer-coated silica (HPCS) particles as carrier, were prepared, characterized and employed as the biocatalyst. **Methods:** esterification conditions: 80 mmol/L FFA, 160 mmol/L phytosterols, immobilized lipase (15 mg/mL), and solvent (isooctane, 20 mL) were added into a vial. The vial was placed in a shaking incubator at 50°C with a shaking speed of 200 rpm for 6 h. **Results:** Six PEs with conversions above 92.1% and controllable fatty acid composition were obtained. The prepared PEs possessed a low acid value ( $\leq 0.86$  mg of KOH/g), peroxide value ( $\leq 3.3$  mequiv/kg), and high purity ( $\geq 97.8\%$ ) and fatty solubility ( $\geq 28.9$  g/100 mL). The CRL@HPCS could be used 10 times with 80.6% of its original catalytic activity still retained. **Conclusions:** The immobilization of CRL on HPCS resulted in a highly stable and active immobilized lipase. The preparation of PEs with controllable FA composition was successfully achieved. All the characteristics favored the wide application of PEs with controllable fatty acid composition in different fields of functional food.

**Chemo-enzymatic Synthesis and Characterization of a Novel Array of Phenolic-containing Emulsifiers: A Physico-chemical Study** Zheng Guo<sup>1</sup>, Bianca Perez<sup>2</sup>, and Sampson Anankanbil\*<sup>2</sup>, <sup>1</sup>Aarhus University, Denmark; <sup>2</sup>Dept. of Engineering, Aarhus University, Denmark

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

**Enzymatic Production of Specialty Fats CBE and HMF Substitute – SOS/OPO** Krish Bhaggan\* and Jeanine Werleman, *IOI Lodders Croklaan, The Netherlands*

Specialty fats belong to a unique category of fats. They are substitutes for other types of fats

and can also be grouped as structured lipids, which can be classified as any modified fat obtained by any modification techniques. Cocoa butter (CB) is a natural fat extracted from the cocoa bean. CB can be replaced by CBEs which have strong similarity of physical characteristics as CB and is fully compatible with CB. Conventional CBEs can be produced by blending different types of Sat-O-Sat fats. The main Sat-O-Sat type of triacylglycerol (TAG) in CB is POSt, StOSt and POP, while conventional CBEs contain StOSt and POP. Different enzymatic processes have been developed to produce Sat-O-Sat type of fats and even matching the TAG composition of CB. Products produced via these processes met all the physical characteristics of CB and is fully compatible in all ratios. HMF is the main source of energy for breastfed infants, contributing more than 50% of the total energy intake. Major fatty acids reported in HMF were oleic-, palmitic-, linoleic-, stearic-, myristic and lauric acid in which more than 60% of the palmitic acid is esterified at the sn-2 position resulting in the main TAG molecule in HMF of the type 1,3-dioleoyl-2-palmitoyl-sn-glycerol (OPO). This type of TAG molecule can be produced enzymatically and when blended with other oils, the desired intramolecular structure of HMF can be mimicked. Different enzymatic processes have been developed to produce these Sat-O-Sat and OPO type of specialty fats and will be discussed.

### **Oil Yield Increase in Enzymatically Assisted Degumming of Vegetable Oil in a 1-step Process**

Nina Schroegel-Truxius\*, *AB Enzymes, Germany*

**Objective:** Phospholipase A2 used for enzymatic degumming has resulted especially in higher oil yield, lower gum phase and lower phosphorus contents. This effect is caused by the phospholipase A2 action, which releases a fatty acid from Phospholipids, changing them into Lyso-phospholipids. Therefore the emulsification properties are changed and thus reduces oil yield loss during enzymatic degumming. Typically, enzymes for subsequent Phospholipid to Lyso-phospholipid conversion show a pH optimum more neutral than the low pH of the water phase during acid treatment. This requires a 2-step process. **Solution:** Adding a Phospholipase that can act at a low pH (PL A2, optimum pH: 3.8-4.2) the simultaneous addition of acid and enzyme along with the water phase is possible, which also shows a reduction in insoluble calcium salts. There is no need for further addition of caustic during the degumming process. **Conclusion:** The addition of a phospholipase A2 into a vegetable oil leads to an improved performance for the enzymatic oil degumming process in terms of oil yield increase, gum phase decrease as well as overall phosphorus decrease. The proposed 1-step enzymatic degumming process can be implemented into existing processes.

### PRO 3: Refining—Basic and New Technologies

*Chairs: Jim Willits, Desmet Ballestra, USA; and William Younggreen, Alfa Laval Inc., USA*

#### **AquaHy—An Aqueous Extraction of Oil Crops**

Stefan Kirchner\* and Jörg Heidhues, *GEA Westfalia Separator Group GmbH, Germany*

The aqueous recovery of oil from crops such as palm fruits and olives is an established technology since years, whereas seed oils such as soybean, rapeseed and sunflower are recovered by expeller presses and/ or solvent extraction. The AquaHy-Process offers an interesting alternative for seed oils with a higher oil content. The main challenges for the aqueous extraction of oil seeds is to break up the oil containing cells of the plant material and release the oil by using a water based extraction. This lecture describes the trials which have been conducted on a pilot plant scale for an aqueous extraction with different plant materials, e.g. Jatropha and Shea nuts. The process has been optimized in order to reduce the water consumption by achieving best yields and oil qualities. Performance data of the process will be described as well as the qualities of the oils. Concepts for the recovery of special native oils as well as oils for biofuels will be presented. The oils extracted by the AquaHy-Process offers excellent crude oil quality, e.g. extremely low phospholipids content, which make them ideally suited as basis for simple refining to edible oils or oleo chemical products.

#### **Expanding the Enzymatic Degumming Toolbox with a New Phospholipase C** Hanna M. Lilbæk\* and Per Munk Nielsen, *Novozymes, Denmark*

Enzymatic degumming has become an established technology in vegetable oil refining, offering improved process economics and sustainability for crushing and refining plants. The optimal choice of enzyme depends on the degumming process and specifications of the

degummed oil. Phospholipase A enzymes are particularly suited for deep degumming, thanks to their unmatched efficiency to reduce phosphorus to very low level, while at the same time improving overall process economy via reduced oil losses and chemical usage. Novozymes' newest generation of PLA-1 is suited for operation at acidic environment and higher operating temperature increasing the process robustness and economics even further. In water degumming of crude seed oils, the primary benefit of enzymatic process lies in the substantial increase of oil yield. Due to the reduction of total gums formed, the lower dilution of the meal protein is also an appreciable consequent benefit. While both phospholipases A and C cut oil losses via reduced amount of neutral oil entrapped in gums, phospholipase C also generates diglycerides which add to the oil yield. Novozymes has developed a new phospholipase C product that is optimally suited for enzymatic assisted water degumming, generating significant yield improvement without increasing the FFA content of oil. In this presentation we will discuss the properties and application of the new PLC.

#### **Oil Modification: Solution or Problem for 3-MCPD/GE Mitigation** Marc J. Kellens, Wim De Greyt\*, Véronique Gibon, and Jeroen Maes, *Desmet Ballestra Group, Belgium*

In its recent published scientific opinion, EFSA confirms the potential health concern of 3-MCPDE and GE and highly recommends a further (significant) reduction of 3-MCPDE/GE levels in (infant) food fats. In anticipation to expected European regulatory limits, oil processors are obliged to accelerate the implementation of new mitigation technologies to further reduce 3-MCPDE/GE levels, especially in refined palm oil

(fractions). While mitigation is mostly achieved during (post-)refining, the impact of various oil modification processes on final 3-MCPDE/GE levels in food oils and fats may certainly not be overlooked. 3-MCPDE are enriched in liquid olein fractions during dry fractionation. As a result, palm super olein can have 50% higher 3-MCPDE levels than the feedstock RBD palm oil. This will make 3-MCPD mitigation during palm oil refining even more challenging knowing that nearly all palm (super) olein is obtained from fully refined feedstock and that there's no economical, industrial process (yet) for the removal of 3-MCPDE from refined oils. On the other hand, 3-MCPDE are degraded to nearly non-detectable levels during chemical interesterification (CIE). At the same time, the very alkaline conditions during CIE give also formation of (very) high levels of GE. Careful post-refining consisting of a post-bleaching with (non-HCl) activated bleaching earth and mild deodorization is required to obtain a fully refined CIE fat with very low 3-MCPDE/GE levels. Contrary to CIE, 3-MCPD/GE levels are nearly not changing during enzymatic interesterification (EIE) because of the much milder process conditions (lower temperature, no alkaline conditions).

**Micronutrient Recovery in Deodorization.** Steen Balchen, Alfa Laval, Denmark  
*Abstract not available.*

**How Silica Can Improve Quality and Reduce Operating Cost for Enzymatic Interesterification**  
Jim Willits\*, Desmet Ballestra, USA

By proper use of a specific silica, marked reduction of operating cost for enzymatic interesterification can be achieved. The use of silica as a pre-treat agent can also improve the finished quality of the interesterified oil. The presentation will outline the process modifications needed to an existing system as well as a design flow for a new system.

**3-MCPD/GE—Current and Future Technologies for Mitigation.** Danilo Lima\*, Alfa Laval, Brazil  
*Abstract not available.*

**New Developments in the Enzyme Processing of Oilseeds** Steve Gregory\*, DSM, USA  
*Abstract not available.*



**BIO 3.1 / PRO 3.1: Biodiesel from Low-quality Feed Stocks**

*Chairs: Casimir Akoh, University of Georgia, USA; and Per Munk Nielsen, Novozymes, Denmark*

**Lipase-mediated Biodiesel Production and Its Commercialization Progress** Dehua Liu\*, *Tsinghua University, China*

Although it is well recognized that enzymatic process has tremendous advantages versus alkali/acid-based catalytic process, the low stability (poor operational life) and the high cost of the lipase have been the main hurdle to the industrialization of lipase-catalyzed biodiesel production. Tsinghua University has been engaged in enzymatic process for biodiesel production for more than 10 years and great breakthrough technologies/equipments including integrated use of different lipases, on-line water removal, membrane technology for recovering lipase as well as development of a novel air-lift bioreactor have been developed/achieved successfully, with which the lipase's operational life is improved greatly, leading to significant reduction in lipase cost. This enzyme-mediated biodiesel production has been commercialized with a capacity of 50,000 tons/y biodiesel in China and a bigger one (200,000ton/year) is being under construction. This process has also been demonstrated successfully in Brazil. The successful running of this technology is attracting attention worldwide, and companies from the United States and European countries are exploring the potential of licensing this technology for large-scale biodiesel production based on low quality oil feedstocks. In 2016, a biodiesel plant (with a capacity of 50,000 tons/y) from Texas, US decided to use this enzymatic process for biodiesel production (the company originally use alkali-based catalysis) and currently they are modifying the related equipment and shifting the production from alkali-based process to lipase-mediated process.

**Industrial Applications in Continuous Enzymatic Biodiesel Processing** Brent Chrabas\* and Stu Lamb, *Viesel Fuel LLC, USA*

The ability to optimize process inputs in a continuous system has lead to the development of the Continuous Enzymatic Biodiesel Process. Working in collaboration with Novozymes, Viesel Fuel, LLC is pioneering the use of enzymes in their Continuous Enzymatic Biodiesel Process in Ft Myers, Florida. Initially brought to pilot scale out of the laboratory in 2014, the construction of the full scale Continuous Enzymatic Biodiesel Process was completed in 2016. The Industrial Application of the Continuous Enzymatic Biodiesel Process being demonstrated by Viesel Fuel, LLC at its South West Florida production facility was retrofitted from an idled biodiesel plant which utilized traditional chemical catalysts and has a nameplate capacity of 9 MMGY. The laboratory capabilities and process controls required to support production will be highlighted as the general process flow of the Continuous Enzymatic Biodiesel Process is outlined in this presentation.

**Biodiesel Produced from Oil Recovered from Waste Water Plants** Frankie Mathis\*, *Tactical Fabrication LLC, USA*

Tactical Fabrication has in the past years been gathering data on unit operations to clean oily waste (Fats, Oils and Grease/FOG) from waste water treatment plant (WWTP). The goal is a purity of the oil where the Novozymes Eversa enzymatic process can produce biodiesel from the convertible material. Any industrial technology for producing biodiesel from FOG have until now been focused of using acid esterification and caustic

transesterification, in an array of different setups. The main issues being high free fatty acid and catalyst side reactions. After the ban on high sulfur (500ppm) fuel, things have slowed down. The FOG harvester is not just a piece of equipment, but a mobile lab and design team, who will verify and prove in pilot scale the functionality of a specific self-sustaining piece of equipment for a location. The main challenge of the FOG harvester has been to identify the enzymatic process' key parameters. Just removing water and solids is not enough; pH-control and demulsification are also key, resulting in a feedstock for enzymes and not just clean oil. Any carry over of suspended water has the risk of no conversion and a loss of all catalyst. Along with the development of the FOG harvester, Tactical Fabrication has been a key player in the development and improvement of a downstream design, which solves the challenges arising from the use of enzymatically catalyzed biodiesel production and removes the sulfur from the crude biodiesel. A separate issue, which does not practically impact the FOG harvester, is the calculation of yield, and therefore a major challenge in getting the technology implemented. The oily feedstock from a specific WWTP will have a target level on convertible material, so to optimize reactor space, but the biodiesel production will need to handle waste streams, just as it is the case with other oil raw materials. The enzymatic process with distillation currently offers 80% low sulfur biodiesel relative to the convertible material in the FOG. We expect to gather more real-life production data, which will both allow us to optimize the yield and understand the degree of variation associated with WWTP locations. In this presentation we document how high sulfur FOG have been collected from waste water and converted to ASTM biodiesel by an enzymatic biodiesel process.

**Industrial Enzymatic Biodiesel from Low-cost Feedstocks** Marcelo Cantele\*, *Tranfertech Gestão de Inovações LTDA, Brazil*

Currently, large scale production of biodiesel has been mostly based on homogeneous, alkali catalysis. However, biotechnological production of biodiesel with lipases has received growing consideration in recent years and is undergoing a rapid development. Compared to conventional alkali-catalyzed production, the enzymatic process is considered a "green route" because it is less energy intensive and produces higher-purity product with less downstream operations. In addition, the enzymatic process is very tolerant to high acid and water contents present in waste oils and increases the biodiesel yield by avoiding the typical soap formation due to alkaline transesterification. Edible oils with less than 1 wt% free fatty acids (FFA) have been used as feedstock for industrial biodiesel production despite the relatively high cost of the raw material, which is nowadays one of the most significant factors affecting the economic viability of biodiesel production. In order to make the production of enzymatic biodiesel competitive compared to petroleum-derived diesel, feedstock for long-term supply and at the lowest price possible must be pursued, such as soapstock, acid oils, deodorized distillates, grease trap fat. The use of unrefined, less expensive, high FFA, lower-grade oils and fats would result in a dramatic reduction of the overall costs of enzyme-catalyzed biodiesel production. Liquid formulations of lipases can provide a highly competitive option for the conversion of oils and fats to biodiesel. In this context, the performance of a commercial, low-cost, soluble free-lipase (Eversa Transform 2.0 from Novozymes) is documented in this talk. Low-cost feedstocks such as yellow grease and tallow used in the enzymatic hydrolysis followed by esterification reactions for fatty acid methyl esters (FAME) production, both in

laboratory scale and pilot plant units (1 ton/batch) will be discussed. Results of biodiesel production from an industrial (~ 100 ton/day) plant is also described and details discussed based on a complete flowsheet of the large-scale industrial processing, from the raw materials processed, reaction system, washing, phase separation and purification to give on-spec biodiesel storage. Finally, local biodiesel market is discussed bringing future challenges and perspectives for the field.

### **Customized Solutions Through Modular Engineering of Renewable Biodiesel Production Plants**

Gijs Calliauw\*, Wim De Greyt, Dario Altera, and Marc J. Kellens, *Desmet Ballestra Group, Belgium*

While extensive R&D has convincingly demonstrated the potential of enzymatic biodiesel production to become a credible and economically attractive process, the next challenge is to extend and improve the engineering and safe operation of such plants at an industrial scale. Compared to chemical catalysts, enzymes allow the use of a wider spectrum of feedstocks, containing higher amounts of free fatty acids, water and oxidation products. This advantage can extend the applicability and profitability of the total process, but it also requires that the actual plant can effectively deal with this large variety. Also, an industrial biodiesel plant involves more processing steps than only the methylation section, and if any enzymatically based technology is to be commercially viable, it requires the flexibility to be integrated into existing biodiesel plants as well. Hence, a successful industrial design of the enzymatic biodiesel process is one that can handle the feedstock variability as well as be able to blend in seamlessly with current practices in existing installations. Desmet Ballestra now approaches this challenge through a 'modular design' in which the sequence of processing the oil feedstock to in spec

biodiesel, as well as the side stream handling, is broken down in many processing steps. This facilitates engineering and design of customized installation and the understanding of the sensitivity of each processing step. Such approach is also of crucial importance in assessments of the return-on-investment, the impact of feedstock changes on yields, and the importance of waste stream valorization. Modular engineering thus helps to understand the sensitivities and impact of various process choices on the final feasibility of an enzymatic biodiesel project, how it compares to existing competing chemical technologies, and therefore contributes to building better plants in response to the customer's specific demands.

### **Enzymatic Biodiesel from Distiller's Corn Oil.**

**Experiences from Full Scale Production** Anders Rancke-Madsen\*<sup>1</sup>, Mark Bollinger<sup>2</sup>, Hans Christian Holm<sup>3</sup>, and Per Munk Nielsen<sup>1</sup>, <sup>1</sup>*Novozymes, Denmark*; <sup>2</sup>*Novozymes, USA*; <sup>3</sup>*Novozymes A/S, Denmark*

The use of liquid enzymes in biodiesel production has been a break-through as liquid enzymes can handle crude feed-stocks with any content of free fatty acids at low economical risks. However, complexity of re-using the enzyme and lack of robust polishing technologies has been major challenges. A next version liquid enzymatic biodiesel process is based on a new more stable variant of the *Thermomyces lanuginosus* lipase, single time use of the enzyme and a polishing neutralization step called "the one reactor process". The new process has over the last two years been validated in large scale and has proven robust due to simplicity, low process variations and excellent separation performance. Reaction conditions on distiller's corn oil are 3 kg enzyme/ton oil, 2% water, 1.2-1.5 equivalents of methanol and 24-30 hours of reaction time at 104°F/40°C. After reaction, dilute caustic is mixed

in to the reaction mixture for 30 minutes. After a few hours of settling time at 130°F/60°C, the FAME phase is separated from the heavy phase, washed with 2% water, dried and distilled. The biodiesel yield is 93% and the glycerol heavy phase has relatively low salt and methanol content. The presentation will discuss the process parameters in detail and document the overall process robustness with data from lab as well as full scale production.

**Eurofins QTA, AOCS Ck2-09 Solution for the Quality/Process Control in Enzymatic Biodiesel Production**

Nan Wang<sup>1</sup>, and Kangming Ma\*<sup>2</sup>,  
<sup>1</sup>*Eurofins Analytical, USA*; <sup>2</sup>*Eurofins QTA Inc., USA*

Process control is critical to produce high quality biodiesel in the enzymatic process.

Traditional analytical methods are based on the wet chemistry or chromatographic methods. These methods require tedious sample preparation and lengthy analytical time. The operators' error can also make the results unreliable. AOCS ck2-09 is based on the patented technology utilizing the FT-MidIR instrumentation. It analyzes multiple parameters without sample preparation. All results are generated within two minutes. The calibration database has covered all the feedstock available in the market and monitored constantly. The rapid results can be used closely to monitor the enzymatic process from the feedstock, transesterification, to the polishing stage, ensuring the quality of the B100. The presentation will demonstrate the operation of QTA system and the best practice for enzymatic biodiesel production.

## PRO 4: Environment and Regulatory

*Chairs: David Selig, Louis Dreyfus Co., USA; and Eduardo Mualem, Bunge Southern Cone, Argentina*

### **Valorizing Waste Streams by Integrated Biorefining Process** Jingbo Li\* and Zheng Guo, Aarhus University, Denmark

Wet gum, deodorizer distillate, spent bleaching earth, and meal are the main by-products generated by classic oil refining of rapeseed oil. To valorize the by-products, innovative and greener technologies need to be developed as there are still technology challenges to be addressed. Here we present two pieces of related processes to demonstrate how to add value to the by-products. The potential candidate responsible for the taste and flavor of rapeseed lecithins was identified as sinapic acid. The content of sinapic acid was tracked following typical lecithin refinery processes including drying, acetone deoiling, and ethanol fractionation. It was found that the acetone deoiling process removed a large part of sinapic acid from lecithin. The sinapic content was below detection limit when the deoiling process was performed for three times. Sinapic acid also showed good antioxidant activity as we measured. Therefore, the controllable sinapic acid content in the product could meet the needs of different applications. Additionally, we developed a one-step process to produce more canolol from rapeseed meal directly. Several works have studied the formation of canolol by two-step process: heat of rapeseed meal followed by extraction; or extraction of phenolics followed by transformation. In our work, the two steps were integrated into one, namely, concurrent extraction and transformation. The pressurized solvent extraction system was employed to conduct this process. Selection of solvent and effect of temperature, treatment time, and catalyst were investigated extensively. Higher canolol content than ever reported was obtained, suggesting the effectiveness of this process.

### **Waste Heat Recovery in Soybean Processing** Mohamed Abid\*, Solex Thermal Science Inc., Canada

The conditioning of soybean or rapeseed is an important step in preparation for extraction process. It is also an energy intensive process with a high waste

heat recovery potential which can reduce the overall steam consumption of the processing plant. Innovative plate technology provides solutions that allow for efficient heat recovery. Low-grade energy that is otherwise wasted or not economically recoverable can be a source of cost savings by reducing steam consumption. It is essential to utilize high heat transfer rate area to enhance heat transfer efficiency when dealing with a low-grade heat from multiple sources: water, condensate, or flash steam. The heat recovery loop can be optimized for maximum energy savings along with design that allows for a wide range of operational flexibility. It is critical for plant operators to understand and grasp the importance of a waste heat recovery system that maximises energy saving and quickly pay back on capital investment. This paper covers fundamentals that are critical when designing a waste heat recovery system in a crushing plant.

### **Impact of New NFPA 652 Combustible Dust Standard on Oilseeds Crush Plants** Matthew Williamson\*, ADF Engineering, USA

In September 2015, the National Fire Protection Association (NFPA) issued NFPA 652, Standard on the Fundamentals of Combustible Dust. This new standard ties together the myriad of industry specific codes and standards related to combustible dusts, including NFPA 61 and 654, under one overarching standard that establishes minimum requirements for managing dust hazards across all industries. Most importantly, this standard is retroactive, meaning that all facilities that handle combustible dusts must meet these new criteria by the deadline of September 2018. This new standard impacts every oilseeds preparation and crush facility, regardless of age or size, as well as every grain elevator and seed facility in the United States. What are the specific requirements of this standard and how can you ensure compliance quickly and with minimal cost or disruption? This presentation will explain the testing and documentation requirements and discuss the newly required Dust Hazard Analysis (DHA) in order to prepare

your facility for compliance with NFPA 652.

**100% Contact Extractor: Thicker Flakes and Low Residual Oil** Adolfo T. Subieta\*, *Desmet Ballestra North America Inc., USA*

In a solvent extraction operation, thin flakes (< 0.012 inch or 0.30 mm) are recommended for low residual oil in the spent meal (or “white flakes” residual oil). However, how easy is to keep thin flakes 24/7 in a solvent extraction operation? A more practical approach is presented, accepting thicker flakes while maintaining low residual oil in the extracted meal. This requires an extractor that consistently maintains 24/7, one hundred percent (100%) contact between the miscella and the flakes during all the washing stages in the extraction operation. The Reflex® extractor is discussed.

## PRO 5: General Processing

*Chairs: Kerry Staller, BSI Engineering, USA; and Nurhan Dunford, Oklahoma State University, USA*

### **Immersion and Percolation Extraction for Solvent Plants and Specialty Applications**

Richard W. Ozer\*, *Crown Iron Works, USA*

Fines in extraction plants hinder percolation and reduce recovery of oil. Fines are generally removed during Prep and leave the plant with hulls. Immersion Extraction was developed to work with finer products that sink in the solvent. Small Scale Percolation Extractors are also available for specialty applications such as Cannabis or Nutraceuticals where percolation is appropriate. This paper will discuss applications such as the processing of fines from solvent plants or nutraceuticals such as cannabis, echinacea, algae etc. using hexane or other solvents. This paper will also review data from installations where fines were screened out prior to the Main Plant Extractor and processed in immersion extractors. improved percolation in the main extractor resulted in better recovery and overall plant production rates.

### **Lipolytic Stability During Wet Storage of**

**Autotrophic Microalgae** Lieselot Balduyck\*<sup>1</sup>, Sebastiaan Bijttebier<sup>2</sup>, Charlotte Bruneel<sup>1</sup>, Griet Jacobs<sup>2</sup>, Stefan Voorspoels<sup>2</sup>, Koenraad Muylaert<sup>1</sup>, and Imogen Foubert<sup>1</sup>, <sup>1</sup>*Katholieke Universiteit Leuven Kulak, Belgium*; <sup>2</sup>*VITO, Belgium*

Microalgae contain substantial amounts of lipids, which can be used in biodiesel, food, pharmaceuticals, and cosmetics. For these applications, lipolysis reactions, taking place during processing and storage, and the accompanying formation of free fatty acids (FFA) are detrimental for lipid quality. This study contributes to a better understanding of the underlying mechanisms leading to lipolysis during storage of wet microalgal biomass and the dependence of this process on storage temperature and microalgal species. Therefore, wet biomass of *T-Ischrysis lutea* and

*Nannochloropsis oculata* was stored at 20°C, 4°C and -20°C during three weeks. The extent of lipolysis was followed by analysis of the FFA content and by separation of the extracted lipids in lipid classes by ultrahigh-performance liquid chromatography–accurate mass spectrometry (UHPLC-amMS). In *T-Ischrysis*, problematic amounts of FFA were formed very rapidly during wet storage at 20°C, and 4°C. A significantly higher lipolytic stability was observed in *Nannochloropsis* than in *T-Ischrysis*, as the FFA formation in *Nannochloropsis* was negligible during the first days of wet storage. It was hypothesized that this difference was caused by a much stronger cell wall in *Nannochloropsis* than in *T-Ischrysis*. Therefore, an additional experiment was performed in which the wet biomass was disrupted by high pressure homogenization (HPH) and compared to a control sample that was not disrupted. As the disrupted *Nannochloropsis* batch showed a very rapid increase in FFA content, in contrast to the control batch, this confirmed the hypothesis that the strong cell wall of *Nannochloropsis* (partly) protects against lipolysis.

**Effect of Extraction Method on the Composition and Oxidative Stability of Omega-3-rich *Camelina sativa* Seed Oil** Henok D. Belayneh\*, Ozan N. Ciftci, Randy L. Wehling, and Ed Cahoon, *University of Nebraska-Lincoln, USA*

The main objective of this study was to investigate the effect of extraction method on the content of bioactive compounds and the oxidative stability of *Camelina sativa* seed oil which is an underutilized oil source rich in omega-3 fatty acids, tocopherols, and phytosterols. *Camelina* seed oil was extracted with screw press, hexane, supercritical carbon dioxide (SC-CO<sub>2</sub>), and ethanol-modified SC-CO<sub>2</sub>. SC-CO<sub>2</sub> extractions were carried out at varying temperatures (50 and 70 °C) and

pressures (35 and 45 MPa), and the ethanol-modified SC-CO<sub>2</sub> extractions were carried out at varying ethanol concentrations (0-10%, w/w) at the same SC-CO<sub>2</sub> extraction conditions. Nonisothermal differential scanning calorimeter (DSC) oxidation kinetics of the oils were studied at different heating rates (2.5, 5, 10, and 15 °C/min). The highest oil yield (37.6%) was obtained at 45 MPa/70 °C/10% ethanol. Apparent solubility of camelina seed oil in SC-CO<sub>2</sub>, determined using the Chrastil model, ranged from 0.0065 kg oil/kg CO<sub>2</sub> (35 MPa/50 °C) to 0.0133 kg oil/kg CO<sub>2</sub> (45 MPa/70 °C). Phospholipids and phenolic content of the oils increased significantly with ethanol-modified SC-CO<sub>2</sub> (p<sub>2</sub> containing 10% ethanol. Oxidative stability of the oils depended on the type and content of the polar fractions, namely, phenolic compounds and phospholipids. Phenolic compounds acted as natural antioxidants, whereas increased phospholipid contents decreased the oxidative stability. The study showed that oxidative stability of camelina seed oil can be improved at the extraction stage and this may eliminate the need for additive antioxidants.

#### **Effect of Ultrasound on Extraction Yield and Quality Characteristics of Extra Virgin Olive Oil**

Alev Y. Aydar\*, *Manisa Celal Bayar University, Turkey*

In our study, the influence of ultrasound on the extraction yield and quality characteristics of Extra Virgin Olive Oils extracted from of Edremit, Uslu and Gemlik cultivars were studied. The extraction yield, free acidity, peroxide value, p-anisidin value, total oxidation value, K232 and K270, sensorial properties, total phenol compound, the content of total chlorophyll and total carotenoid compounds were determined. Oil obtained from Edremit olive pastes treated with 4 and 8 minute ultrasound improved oil yield, by 0.59% and 0.60% and the oil extractability by 3.32% and 3.40%, respectively. Moreover, the content of chlorophylls and carotenoids were highest in samples subjected to 8 minutes sonication treatment whereas they were

lowest in olive oils subjected to 12 minute ultrasound. Oils extracted by 8 minute sonication showed higher oxidative stability as proven by their lower FFA, p-AV, PV and TOTOX values than those extracted by 4 and 12 minute sonication. The total phenol content of EVOOs was decreased as ultrasound time increased. The results showed that applying sonication prior to malaxation is an effective technique for extra virgin olive oil extraction and the benefit of this technique include higher extraction yield and lower extraction time with no negative effect in quality and antioxidant activity of virgin olive oil.

#### **Influence of Adsorption Parameters on Physical Refining of Sunflower Oil Using a New Mesoporous Silica Based Adsorbent** Ecem Tiryaki\*, Tulay Merve Temel, Burcu Karakuzu, and Sevil Yucel, *Yildiz Technical University, Turkey*

Sunflower oil has gained special significance because of its widespread all over the world and health benefits. Due to the presence of linoleic acid, oleic acid, palmitic acid, and stearic acid, sunflower oil has an impressive fatty acid profile. Crude sunflower oil is obtained from the seeds of sunflower (*Helianthus annuus*). Nevertheless, the refining process is necessary to enhance quality, stability and shelf life of crude oils. Especially, there is still a need for novel efficient adsorbents to be used in oil refining. In the present study, the efficiency of a new mesoporous silica based adsorbent has been evaluated for refining of sunflower oil. The adsorption of crude and neutralized sunflower oil was carried out at different temperatures and adsorbent dosages (1-3%) for 30 minutes. Adsorption studies have been performed with a combination of sunflower oil in a ratio of %1-3 in weight of mesoporous silica, bentonite, commercial silica, bentonite+mesoporous silica and bentonite+commercial silica. The results revealed that silica based adsorbent was very effective for the removal of free fatty acids at 90°C with 3% adsorbent dosage. In bleaching process, which has



been applied to neutralized oil the mixture of %1 bentonite+mesoporous silica gave the most effective results. *This research is supported by The Scientific and Technological Research Council of Turkey (TUBITAK) (Project No:115M469).*

**The Effects of Oilseed Processing on Bioactive Compounds in Edible Canola Oil: A Case Study Involving Australian Processing Plants** Clare L. Flakelar<sup>\*1</sup>, David J. Lockett<sup>2</sup>, Julia A. Howitt<sup>3</sup>, Gregory Doran<sup>3</sup>, and Paul D. Prenzler<sup>3</sup>, <sup>1</sup>Charles Sturt University, Australia; <sup>2</sup>Graham Centre for Agricultural Innovation, Australia; <sup>3</sup>School of Agricultural and Wine Sciences, Charles Sturt University, Australia

Conventional and novel approaches to industrial oilseed processing may have quite different effects on the retention of minor components in edible food oils. To our knowledge, no published study has been conducted that directly compares bioactives in multiple seed and oil samples obtained from different processing plants, at an industrial scale. This study investigated the impacts of current techniques on bioactive compounds, and determined the potential for enhancement of oil quality on a basis of these nutrients. Tocopherols, carotenoids and sterols are classes of bioactive compounds present in canola seed and oil that each possess health beneficial properties. During this study, canola seed and oil samples from multiple stages along the processing chain were acquired from four Australian oil processing plants, and concentrations of tocopherols, carotenoids and sterols determined. Samples including those from: one solvent-expeller refinery, one expeller-physical degumming process, an expeller-chemical degumming process and two cold pressing plants. High abundances of tocopherols and sterols were discovered in the refined oil samples, indicating high efficiency of current techniques. Additionally, the different seed pre-treatment techniques used were found to have a considerable effect on bioactive concentrations. Results indicate that the

current techniques result in valuable products accumulating in end-product oil, however a proportion remains in waste by-products, and strategies for retention or purification should be sought.

**Applying Different Filtration Parameters on Crude Canola Oil with Metal Doped Nanoporous Silica Adsorbent** Tulay Merve Temel<sup>\*1</sup>, Burcu Karakuzu<sup>1</sup>, Pinar Terzioglu<sup>2</sup>, and Sevil Yucel<sup>1</sup>, <sup>1</sup>Yildiz Technical University, Turkey; <sup>2</sup>Muğla Sıtkı Koçman University, Turkey

Crude canola oil includes 95-99% triacylglycerols and 1-5 % minor compounds. Some minor compounds are not desired because of their bad influence on oil quality and harmful to the body. Hence, crude oil is refined for the purpose of removal this compounds. The fatty acids found in the oil is removed by use of alkali in the neutralization. Various bleaching earths, silica and combination of these are used in the bleaching. In the industry, a qualified oil can be obtained by use of double adsorbent (bleaching earth and commercial silica) in the bleaching which provides color and phospholipid removal. In the recent years, researches have been conducted for the improvement of various adsorbents to remove impurities of oil. However, efficient removal of impurities such as polar compounds, phospholipids, free fatty acids and heavy metals is not impossible because bleaching earth is preferred due to its low cost only in the bleaching step. For this reason, there is a need of new adsorbents to remove other impurities. Silicates generally have the adsorption capacity for polar lipids (free fatty acids, partial glycerides etc.) of oil. In this study, metal doped nanoporous silica adsorbent was used for crude canola oil filtration in different temperatures (30, 60, 90°C) and adsorption times (10, 20, 30 minutes). Metal doped nanoporous silica have 230 m<sup>2</sup>/g surface area, 0.19 g cm<sup>-3</sup> tapped density. Consequently, the most suitable conditions for adsorption was determined to be 90°C and 30 minutes and filtrated canola oil's

oil acid change % was found 21%. *This research is supported by The Scientific and Technological Research Council of Turkey (TUBITAK) (Project No:115M469).*

**Development of an Up-grading Process to Produce MLM Structured Lipids from Sardine Discards**

Rocio Morales-Medina\*, M. Munio, A.M. Guadix and E.M. Guadix, *University of Granada, Spain*

The aim of the work was to produce MLM structured lipids with caprylic acid (M) as medium chain fatty acid and concentrated polyunsaturated fatty acids (L) from sardine discards (*Sardine pilchardus*) in the central bond of the glycerol. To that end, the following steps were conducted: (i) fish oil extraction, (ii) Omega-3 free fatty acids (FFA) concentration (low temperature winterization), (iii) two-steps enzymatic esterification and (iv) triacylglycerols (TAG) purification (liquid column chromatography). The resultant purified triacylglycerol accomplished with the oxidative state: (peroxide and anisidine value, PV and AV) required for refined oils. As enzymatic treatment, the concentrate FFA was esterified with dicaprylic glycerol employing Novozyme 435. This process presented high regioselectivity with ~ 80 mol% of concentrated fatty acids esterified at the sn-2 position.

**PRO-P: Processing Poster Session**

*Chairs: Ozan Ciftci, University of Nebraska-Lincoln, USA; and Junsu Yang, University of Nebraska-Lincoln, USA*

**1. Comparison of Different Solvents for Extraction of Krill Oil from Krill Meal: Lipid Yield, Phospholipids Content, Fatty Acid Composition, and Minor Components** Dan Xie\*<sup>1</sup>, Jun Jin<sup>1</sup>, Jiang Sun<sup>2</sup>, Xingguo Wang<sup>1</sup>, and Qingzhe Jin<sup>1</sup>, <sup>1</sup>Jiangnan University, China; <sup>2</sup>Zhonghai Ocean (Wuxi) Marine Equipment Engineering Co., Ltd., China

Krill oil has been gaining great research interests owing to its unique composition and function in recent years. The present study is to investigate the effects of seven different extraction solvents (ethanol, isopropanol, acetone, ethyl acetate, isohexane, n-hexane, subcritical butane) on lipid yield and quality of extracted oil from krill meal. Phospholipids (PL), fatty acid and minor components including sterol, astaxanthin, vitamin A and tocopherol in the extracted krill oil were analyzed. The results reflect that ethanol and isopropanol led to comparatively higher lipid yields (16.33% and 14.52%, respectively) and PL contents (39.2% and 38.7%, respectively), but lower content of minor components in the extracted krill oils. The krill oil extracted with acetone contained the lowest PL content (20.63%), but achieved more astaxanthin (206.74 mg/kg), vitamin A (27.84 mg/100g) and sterol (39.00 mg/g). Moreover, high levels of n-3 fatty acid were observed in the extracts with high PL contents, because 23.65-28.1% of EPA and 16.71-21.03% of DHA were distributed in PL, while only 2.83-3.48% and 1.40-1.74% in triglycerides (TG). Also, subcritical butane was revealed to be an alternative to n-hexane and isohexane as they extracted krill oils with similar qualities. Based on our research, it can be concluded that the extraction solvent should be considered carefully for krill oil industrial production. The information of this work can help manufacturers to select solvent or mixed solvents in krill oil industry more appropriately.

**2. Development of a Cocoa Butter Improver by Chemical Interesterification and Thermal Fractionation** Glazieli M. de Oliveira\*<sup>1</sup>, Ana Paula B. Ribeiro<sup>1</sup>, and Theo G. Kieckbusch<sup>2</sup>, <sup>1</sup>University of Campinas, Brazil; <sup>2</sup>ICT-Institute of Science and Technology, UNIFAL, Brazil

Seasonal variations in prices and/or oleaginous crop failures demand special processing efforts in order to standardize product formulations containing lipids. Cocoa butter (CB) is especially vulnerable to these disturbances and its chemical composition is also a strong function of the latitude of the farming, compromising the production of chocolates and confectionary goods. The oscillating chemical and physical characteristics of CB can be modified or customized through addition of cocoa butter improvers (CBIs). This research considered the development of CBI to be applied in CB. Lipid sources like high oleic sunflower oil (HOSO) and fully hydrogenated canola oil (FHCO) were contemplated as raw materials. A blend containing 45% of HOSO and 55% of FHCO were prepared and submitted to chemical interesterification (CIE). The CIE led to the production of a sample with 39.9% of disaturated TAGs (S2U), which included 28.1% of StOSt/StStO (stearic-oleic-stearic acids). The heavy trisaturated (S3) TAGs were separated by dry (thermal) fractionation with the runs conducted at 36 and at 53°C showing the best yields. At last, the olein obtained at 36°C was submitted to solvent (acetone) fractionation runs for the removal of the triunsaturated (U3) TAGs. The fractionation at 12°C produced a stearin fraction with a total of 78.7% of S2U, including 63.4% of StOSt/StStO. The crystallization behaviors by DSC were investigated and the solid fat content determined.

### 3. Application of Imidazolium-based Ionic Liquids as Co-solvent for Extraction of Oil from Nahar Seeds Using Sonoreactor

Adeeb Hayyan\*<sup>1</sup>, Mohamed E.S Mirghani<sup>2</sup>, Shahidah N. Rashid<sup>3</sup>, Maan Hayyan<sup>4</sup>, M. Y. Zulkifli<sup>4</sup>, and Fazrizatul S. Sani<sup>3</sup>, <sup>1</sup>University of Malaya, Malaysia; <sup>2</sup>Dept. of Biotechnology Engineering, Faculty of Engineering and International Institute for Halal Research and Training, International Is, Malaysia; <sup>3</sup>University of Malaya Centre for Ionic Liquids (UMCIL), Kuala Lumpur, Malaysia; <sup>4</sup>Institute of Halal Research University of Malaya, Malaysia

Mesua ferrea L. (Nahar) known in Malaysia as penaga lilin or commonly known as the iron wood tree is plant species available widely in Sri Lanka, Nepal, India and Malay Peninsula. In this study, oil was extracted from Nahar seeds using n-Heptane as organic solvent. Ultrasonic bath was used as reactor for extraction of oil from Nahar seeds. Different operating parameters were optimized such as mass ratio of seeds to solvent, extraction time and extraction temperature. Different types of imidazolium-based ionic liquids (ILs) were used and screened as co-solvent to enhance the oil extraction process. 1-Ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide (EMIM TFSI) was recommended for further optimization. The highest oil yield was obtained under these optimum conditions (1:6 mass ratio of seeds to n-Heptane, 30 minutes and 60°C). When IL used as co-solvent, almost same yield was obtained using mass ratio of 1:3:3 (seeds:n-Heptane:IL). IL can reduce the usage of volatile organic solvent such as n-Heptane.

### 4. Lowering the Temperature Improves Enzymatic Prefractionation of EPA and DHA

Ryosuke Hoshina\*<sup>1</sup>, Yomi Watanabe<sup>2</sup>, Kazumi Katagiri<sup>1</sup>, and Hideaki Kobayashi<sup>1</sup>, <sup>1</sup>Kewpie Corporation, Japan; <sup>2</sup>Osaka Municipal Technical Research Institute, Japan

Health benefits on the intake of  $\omega$ -3 polyunsaturated fatty acids (PUFA) have been widely known. Among them, pure 20:5 (EPA) ethyl

ester is approved as medicine in Japan for hyperlipidemia and arteriosclerosis obliterans, while 22:6 (DHA) concentrates are used as supplements to prevent cardiovascular diseases, dementia, cognitive disorders, etc. EPA and DHA could be fractionated and purified by MPLC or by the rectification, for example. Prefractionation step to roughly separate the two in the material oil might improve the efficiency of the final purification steps. Lipase treatment is good to this end. Thus, sardine oil was treated by 10 wt% of lipase RMIM (Novozymes) and 2 wt% water with 3 mols equivalent of ethanol, added stepwise. After 24 h reaction at 35°C, nearly 60% transesterification was reached. The content of EPA and DHA in FAEF fraction was 15.4 and 3.4% (EPA/DHA ratio, 4.5). By decreasing the reaction temperature to 15 and to 5°C, the EPA/DHA ratio increased to 7.1 and to 14.6, respectively, where the degree of transesterification was 52 and 62% respectively. Thus, it was suggested that the fractionation of EPA, recovered in FAEF fraction, and DHA, recovered in glyceride fraction, is more effective by lowering the enzymatic reaction temperature. This method might contribute to improve the production efficiency of fractionated EPA and DHA.

### 6. Influence of Active Carbon in Bleaching Process in Coconut Oil

Jin Sup Shin\*<sup>1</sup>, Minyoung Kim<sup>2</sup>, Dongjin Yu<sup>2</sup>, Eunseok Jang<sup>2</sup>, Yoonchang Kang<sup>2</sup>, and Bongchan Kim<sup>2</sup>, <sup>1</sup>Samyang Corporation, Korea; <sup>2</sup>Samyang Co., South Korea

Various methods were researched in an attempt to remove the benzopyrene of oil from edible oils. A coconut oil, commercially extracted from coconuts, was refined in the plant with degumming, bleaching, and deodorization. These oils are widely used as commercial oils. Crude coconut oils can contain more than 50 $\mu$ g/kg benzopyrene. After refining coconut oils, these oils have a little benzopyrene. Even if these benzopyrene is small amount to satisfying regulation, I try to get rid of benzopyrene until not

detected using active carbon increasingly during bleaching process. Not only removing benzopyrene, but oxidation stability was also observed. To remove benzopyrene, active carbon with fuller's earth was applied. As increasing active carbon dose, the benzopyrene content is decreased even though fuller's earth does not effect the benzopyrene content. When 0.1%(total weight) of active carbon was applied during bleaching the coconut oil, benzopyrene is not detected. When the amount of benzopyrene is decreased, oxidative stability of coconut oil is decreased too. This phenomenon may be caused by applying active carbon has absorbability of functional substance. Additional study is necessary for manufacturing oxidative stable coconut oil and other commercial oils.

**7. Impact of Roasting on Specific Phytochemicals in Perilla Seed Oil Extracted Using Supercritical Carbon Dioxide** Hyo Jung Cho\*<sup>1</sup>, Nakyung Choi<sup>1</sup>, and In-Hwan Kim<sup>2</sup>, <sup>1</sup>*Korea University, South Korea*; <sup>2</sup>*Korea University, Republic of Korea*

Perilla oil, a beneficial vegetable oil to human health, has one of the highest proportion of  $\alpha$ -linolenic acid (ca. 54-64%). Changes in the phytochemical content of oils prepared from perilla seeds roasted at different roasting temperatures (160, 200, and 240°C) and roasting times (10, 20, and 30 min) were investigated. Specifically tocopherol, squalene, phytosterol, and policosanol were examined. The oil from perilla seed was extracted using supercritical carbon dioxide (SC-CO<sub>2</sub>) at a pressure of 6000 psi and a temperature of 50°C. The tocopherol content of the oils prepared from roasted perilla seeds were significantly higher than those of the oils obtained from unroasted perilla seeds. The squalene content increased markedly as the roasting temperature and time increased. In particular, the squalene content of the oils prepared at early stages of SC-CO<sub>2</sub> extraction was significantly higher than those of the oils prepared at later stages. Meanwhile, no significant changes in the phytosterol and

policosanol contents of perilla seed oils prepared at different roasting conditions were observed.

**8. Novel Technology to Produce High Quality Ester and High Purity Vitamin E from Deodorizer Distillate** Kousuke Hiromori\*<sup>1</sup>, Tomoya Watanabe<sup>1</sup>, and Naomi Shibasaki-Kitakawa<sup>2</sup>, <sup>1</sup>*Dept. of Chemical Engineering, Tohoku University, Japan*; <sup>2</sup>*Graduate School of Engineering, Tohoku University, Japan*

The by-product obtained during edible oil refining, such as deodorizer distillate and fatty acid distillate, mainly contains free fatty acids (FFA) and glycerides. Those from rice bran and palm also contain bioactive compounds such as vitamin E (tocopherol and tocotrienol) with a few weight percent. Many researchers have been proposed the methods of converting FFA and/or glycerides to fatty acid ester by esterification and/or transesterification. On the other hand, other researchers have been proposed the methods of recovering vitamin E by molecular distillation. However, there is no technology to simultaneously produce high quality fatty acid ester and high purity vitamin E from the distillate. We have proposed a novel reaction and separation technology using three kinds of ion-exchange resins at 50°C and atmospheric pressure. It consists of 1) esterification of FFA by strongly acid resin, 2) adsorption of vitamin E on the strongly base resin and transesterification of glycerides by the resin, 3) desorption of vitamin E from the resin and 4) purification of vitamin E by weakly base resin. Using this technology, both FFA and glyceride in the distillate were completely converted to fatty acid ester and vitamin E was recovered with a purity of over 95wt% and a yield of over 85%. Therefore, this novel technology succeeds to simultaneously produce high quality fatty acid ester and high purity vitamin E from the distillate, and allows economic utilization of the by-product obtained during edible oil refining.

**9. Comparison of the Newly Formulated Silica Adsorbent Bleaching Capacity with Traditional Adsorbents** Burcu Karakuzu\*, Sevil Yucel, and Tulay Merve Temel, *Yildiz Technical University, Turkey*

Removed color, phospholipid, soap residue, and some oxidation products from oil is obtained with pairs of adsorbent (bleaching earth and silica) using in the industrial oil bleaching step. Using of the bleaching earth in refining process can be increased some trace elements in edible oil due to bleaching earth contain some trace elements. In earlier studies have been reported that silica adsorbents can be adsorbed these trace elements. The amount of trace elements in edible oils directly relates to oil quality. Even if Fe, Cu, Ni, and Zn as metals is at trace level, they catalyze the autoxidation reaction. Autoxidation causes the formation of peroxide, aldehyde, ketone and radical in oils and decreasing oil's quality parameters. In this study, newly formulated silica adsorbent was produced by the precipitated method and was used in the removing of color pigment in crude sunflower oil. Adsorption experiment was performed at 90°C for 30 minutes. Lovibond tintometer was used to determine color changes in the oil. Adsorption efficiency of newly formulated silica was compared to commercial silica and bleaching earth. Bleaching efficiency of newly formulated silica was found better than commercial silica and similar to bleaching earth. *This research is supported by The Scientific and Technological Research Council of Turkey (TUBITAK) (Project No:115M469).*

**10. Determination of Pore Size and Lignin Distribution in Coconut Shells Residue by Using Stain Technology** Prashant Katiyar\*<sup>1</sup>, Shailendra K. Srivastava<sup>1</sup>, and Vinod K. Tyagi<sup>2</sup>, <sup>1</sup>Sam Higginbottom Institute of Agriculture, Technology and Sciences, India; <sup>2</sup>Harcourt Butler Technological Institute, India

In the present study, we try to investigate the porosity determination of lignocellulose biomass of coconut shells before and after treatment of

biomass. Using stain technology, the determination of pore size distribution and its surface area of biomass of coconut shells is possible. Here, we are employing two types of stains: Safranin and  $KMnO_4$  to analyze the lignin deposition in biomass before and after treatment.

Along with stains, some mordant are used here: Formic acid specific for Safranin and oxalic acid specific for  $KMnO_4$  stain. Before applying the treatment to coconut shell biomass, the biomass will be kept at over-night incubation at 100°C in a hot air oven. The method utilized for the purpose of treatment of coconut shell biomass: Combine treatment with citric acid and hot salt water under varied conditions of pressure and temperature conditions in a vacuum oven. After the treatment, biochemical analyses were done on UV-spectrophotometer to analyze the sugar exposure and lignin reduction. In general, these techniques clearly indicate that the pretreated coconut shell has more pore size distribution and specific surface area as compared to non-treated sample.

**11. Ricinodendron heudelotii Oil: An Original Source of  $\alpha$ -eleostearic Acid,  $\beta$ -eleostearic Acid, and Linoleic Acid** Diakaridja Nikiema\*<sup>1</sup>, Muriel Cerny<sup>1</sup>, Eric Lacroux<sup>2</sup>, and Zéphirin Mouloungui<sup>1</sup>, <sup>1</sup>Laboratoire de Chimie Agro-Industrielle, France; <sup>2</sup>Chimie Agro-Industrielle, France

*Ricinodendron heudelotii* is a tropical tree that is found in West and Central Africa. These oilseeds are rich in  $\alpha$ -eleostearic acid, a C18: 3 n-5 fatty acid. Ricinodendron oil is a siccative oil analogous to Tung oil. Oil fatty acids profile was analysed in GC-FID: 55%  $\alpha$ -eleostearic acid, 25% linoleic acid, 10% oleic acid, 5% stearic acid, and 5% palmitic acid. Obtaining enriched lipids fractions was done according to two approaches, with or without chemical modification of the oil. Stability study of Ricinodendron oil was achieved without chemical modification of triacylglycerols: influence of water, temperature and light radiation. This study shows possibility of isomerization of  $\alpha$ -eleostearic acid (C18:3 n-5; 9c, 11t, 13t) into  $\beta$ -eleostearic acid

(C18: 3 n-5; 9t, 11t, 13t). Indeed, while heat treatment oil causes an increase in linoleic acid content, a photochemical treatment exhibit enrichment in  $\beta$ -eleostearic acid. The lipolytic capability of LypolyveCC lipase is used for the modification of Ricinodendron triacylglycerols in fatty acids (monitoring carried out by HPLC-ELSD). Hydrolysis led to isolate lipids-enriched fractions such as: 55%  $\alpha$ -eleostearic acid fraction, 80% of  $\beta$ -eleostearic acid fraction or a 35% linoleic acid fraction. Physicals and chemicals treatments allow a reasoned and sequenced fractionation of Ricinodendron oil into fatty acids of interest enriched fractions, whatsoever as triacylglycerols or free fatty acids. Ricinodendron oil is definitely an important source for the following tryptic of fatty acids of interest:  $\alpha$ -eleostearic,  $\beta$ -eleostearic, and linoleic.

**12. Stabilization of Meat Products Using Functionalized Pork Fat Produced by Enzymatic Glycerolysis and Short Path Distillation** Eleonora Miquel Becker\*, Bjørn Alexander S. Hansen, Maria Barmar Larsen, and Mia Fiilsøe Falkeborg, *Danish Technological Institute, Denmark*

Large amounts of lipid-based by-products are generated in the food industry each year. In this work, partial glycerides were formed through enzymatic glycerolysis of rendered pork fat. The target product was a mixture of tri- and diglycerides (TAG and DAG), and the by-products (monoglycerides and free fatty acids) were removed using short path distillation. Being of animal origin, this TAG-DAG product was designed for use in meat-based products. The TAG-DAG mixture was thus applied as a stabilizing “functionalized fat” in meat emulsions, which are the basis of products such as sausages, pates, etc. Such products contain a considerable amount of fat, and by replacing the fat with the TAG-DAG mixture, the functionality of the product can be altered. In this work, the TAG-DAG mixture was characterized in terms of thermal properties (DSC)

and emulsification properties. Additionally, the fat fraction of a pork meat emulsion was replaced with the TAG-DAG mixture, and the emulsion was evaluated in terms of fat and jelly separation, total expressible fluid, and texture. The results showed that the TAG-DAG mixture, produced from by-products, could find applications in the area of meat emulsion production as a type of functionalized pork fat, which potentially could reduce the need for stabilizers in meat emulsions.

**13. Valorization of Animal By-products Using Short Path Distillation** Mia Fiilsøe Falkeborg\*, Bjørn Alexander S. Hansen, Maria Barmar Larsen, and Eleonora Miquel Becker, *Danish Technological Institute, Denmark*

Large amounts of lipid-based by-products are generated in the food industry each year. Being food grade, these by-products can be sold as high-value ingredients by use of fractionation or purification processes such as short path distillation (SPD). This work focused on valorization of oil and fat by-products from the food industry, specifically, rendered fat from pork and beef. Being of animal origin, the products should be tailored for use in animal-based food products. In this work, partial glycerides were formed through enzymatic glycerolysis of rendered pork fat. The target product was a mixture of tri- and diglycerides (TAG and DAG), which could be applied as “functionalized fat” in e.g. meat emulsion products (sausages, pates, etc.). To purify the TAG-DAG mixture after glycerolysis, the monoglycerides (MAG), free fatty acids (FFA) and leftover glycerol were removed using SPD. To achieve a high product yield with a low amount of by-products, the glycerolysis reaction and SPD process were optimized. The outcome was response surfaces showing the influence of multiple process parameters. The process showed good potential for obtaining a high-value product from a by-product, and business plan calculations showed the commercial potential of the process.

**14. New Developments to Increase the Production Rate of Chemically Refined Oils** Li-Chih Hu\*, Nathan Dias, and David Gittins, *Imerys Filtration Minerals Inc., USA*

Oil for or from oleochemical processes (including biodiesel production) may come from a range of sources with variable qualities, and contain an appreciable level of metals, phospholipids, soaps, catalysts, and suspension solids. Dry-washing and filtration enables the refined oil to meet the quality and stability requirements for the next process step or customer. This poster describes a single-step oil treatment which is excellent in both adsorbent (metals, phospholipids and soap removal) and filter aid (suspension solids and high viscosity component removal, high throughput and long filtration cycle) performance.

**15. Adsorbent Filter Aids for the Treatment of Edible Oils and Biodiesel Feedstocks** Nathan Dias\*, Li-Chih Hu, and David Gittins, *Imerys Filtration Minerals Inc., USA*

Physically refined vegetable oil contains soluble metals and phospholipids. These impurities need to be removed to meet the quality requirements downstream. This poster describes an adsorbent filter aid specifically designed for this purpose. In addition to its excellent ability to remove metals and phospholipids, this material delivers a prolonged filtration cycle at high throughput, and is not oxidation-catalytic which preserves the nutritional components in the refined oil.