

# 2009 Annual Meeting Abstracts

## Processing

### MONDAY

#### MORNING

##### **PRO 1: History of Processing**

Chair(s): M. Snow, Bunge North America Inc., USA; and J. Willits, Desmet Ballestra North America Inc., USA

**The Historical Retrospective of the Past 100 Hundred Years in Processing.** James M. (Mickey) Lay, Bunge North America, USA

This presentation will provide a historical review of the evolution of a processing industry. The author and presenter (James M. "Mickey" Lay) has been involved in processing the majority of his (37) year career with Bunge North America. The background preparation for the presentation will include many conversations and interviews with processing "Champions" of the past who have dedicated a lifetime to improving the safety and efficiency of our industry.

**The French Oil Mill Machinery Company History.** D. French, The French Oil Mill Machinery Company, Piqua, OH, USA

Daniel P. French, Chairman and President, will give a brief view of the 109-year history of The French Oil Mill Machinery Company, founded 25 May 1900, by Alfred Willard French. Dan French is the third generation of the French family to serve as President. He will show pictures of the early machines invented and patented by his grandfather, discuss some of his grandfather's and father's business philosophies which have helped the company survive and prosper, and bring the audience up to date with the company's current worldwide market position as "The Technology and Quality Leader Since 1900".

**The History of Meal Desolventizing Toasting Drying Cooling Technology.** T. Kemper, Desmet Ballestra North America, Marietta, GA, USA

This is a processing review presentation for the 100th anniversary of AOCS. Oilseed meals exiting the solvent extractor require further treatment prior to storage. The unit processes include thermal heating to evaporate and remove solvent, increasing moisture to deactivate anti-nutritional factors, removing excess moisture, and removing excess temperature. While the unit processes have remained the same for nearly 70 years, the equipment has evolved. This presentation will review the historical timeline of the evolution of meal desolventizing, toasting, drying and cooling equipment from the beginning of the 20th century through today.

**The History of Electrical and Automation in Crushing and Refining.** Doug Post, Interstates Companies, Sioux Center, IA, USA

Changes in electrical equipment and computerized control have constantly redefined state-of-the-art in the oil processing industry. Significant changes include computerization of controls, increased awareness and enforcement of safety standards not only in facilities, and the enhancement of software that changed oil processing and facility design. This session presents an overview of progress during the past century.

**A History of Edible Oil Deodorization.** S. Loft, Stan Loft Consulting, Sedona, AZ, USA

What exactly is deodorization?, or physical refining?, or deacidification?, or steam distillation? All these definitions are used interchangeably to define the process of stripping or removing undesirable constituents from most edible oils. Deodorization is considered the final processing step in producing a finished "Edible" oil. This review begins in the late 1800's when the European community desired to produce a bland tasting margarine. The process began with wood

fired kettles (e.g. David Wesson) and wanders 100 years through a gambit of techniques to increase vaporization efficiency while reducing thermal heat requirements. This paper will present various styles of Edible Oil Deodorizers that have evolved through the decades including current technology.

**The History of Extractors.** Allen Ost, Crown Iron Works, USA

An historical journey through the evolution in the design of solvent extractors from the earliest solvent extractors to the present day. The basic features, designs and capacities of these early extractors will be discussed, along with factors which influenced the configuration and design of modern day solvent extractors. Finally, the trends and challenges for the future will be discussed.

**AFTERNOON**

**PRO 2: Processing Hot Topics**

Chair(s): R. Narayanan, Ag Processing Inc., USA; and T. Gum, Agribusiness & Water Tech Inc., USA

**Designing, Building, and Operating for Safety.** M. Vander Velde, Interstates, Sioux Center, IA, USA

Recent events remind us that we must continue to improve the safety of the processing facilities we design, build, and operate. This session will investigate opportunities processors teamed with electrical, instrumentation, and control system providers have to improve plant safety. We intend to provide practical ways to incorporate enhanced safety into various phases of your plant's life cycle.

**Talent Jungle.** R. Weinberg, Human Resource Consultant, Omaha, NE, USA

Changes in HR field with retirements lay offs and restructuring presents a challenge for all managers. The session presents a strategy to deal with these changes.

**ISEO Activities and Regulatory Issues Facing the Industry.** R. Collette, Institute of Shortening and Edible Oils, Washington, DC, 20006, USA

The reformulation of food to reduce or remove Trans Fat has had a far reaching impact on the Oils and Fats Industry. The session presents an overview of the factors driving this trend. In addition, regulatory activities and other issues affecting the industry will be examined from a Washington perspective.

**Panel Discussion.**

**TUESDAY**

**AFTERNOON**

**IOP 3.1 / PRO 3: Alternative Feedstocks for Biofuels**

Chair(s): B. Moser, USDA, ARS, NCAUR, USA; and N. Dunford, Oklahoma State University, USA

**Biodiesel Prepared from Field Pennycress (*Thlaspi arvense* L.) Oil.** B.R. Moser, G. Knothe, S.F. Vaughn, T.A. Isbell, USDA, ARS, NCAUR, Peoria, IL, USA

Field pennycress (*Thlaspi arvense* L., FP) is a winter annual species of the mustard family (Brassicaceae) which is widely distributed throughout temperate North America that can serve as a winter rotational crop for conventional crops, thus not displacing farm land or negatively impacting the food supply. FP seed contains 36% oil by weight, which is high in erucic acid, with linoleic, linolenic, oleic, and gondoic acids also present in significant quantities.

Biodiesel prepared from FPO (FPME and FPEE) displays excellent cold flow properties, relatively poor oxidative stability, and relatively high kinematic viscosity versus soybean oil methyl and ethyl esters (SME and SEE). Comparison to ASTM D6751 reveals that FPME and FPEE are satisfactory with respect to all parameters measured here. Viscosity, methyl linolenate content, and oxidative stability do not meet the stated requirements in EN 14214, but blending or treatment with antioxidants are likely to ameliorate these deficiencies. Blends of FPME and FPEE in petrodiesel compared favorably to an analogous set of SME and SEE blends and were satisfactory after comparison to ASTM D975 and ASTM D7467. In conclusion, FPO has excellent potential as an alternative biodiesel feedstock.

**Potential of Jatropha and Syringa Oils as Biodiesel Feedstocks as Compared to Canola Oil.** N. Dunford<sup>1</sup>, A. Su<sup>1</sup>, J. Morris<sup>2</sup>, <sup>1</sup>Oklahoma State University, Stillwater, OK, USA, <sup>2</sup>Syringa Institute, Centurion, South Africa

Soybean oil has been the preferred feedstock for biodiesel production in USA because of its availability and lower cost. However many European countries limit the use of biodiesel made from highly unsaturated oils because of its lower oxidative stability. Canola oil is known for its higher stability and has a relatively low cloud point. Hence, it is a good feedstock for biodiesel production. The higher oil content of seeds (about 40%, w/w) also makes canola more attractive than soybean (about 20%, w/w oil content) as biofuel feedstock.

Wheat, corn and sorghum have been typical crops grown in the southern Great Plains for many decades. The introduction of winter canola provides a tremendous opportunity as a rotation crop to control weed, insect and disease problems caused by continuous wheat planting. Winter canola is also a good crop as feedstock for biofuel industry. There is great interest in growing canola among farmers in the Great Plains. It is estimated that canola acreages will continue to increase. Currently there are several initiatives involving canola production, oilseed processing and biodiesel production in the region. On-farm or small capacity canola seed crushing and biodiesel production is also expanding in the Great Plains.

Recent price increases in food products including edible oils such as canola and soybean oil has been one of the driving forces for an intensified search for nonfood biofuel feedstocks. *Jatropha Curcas* has a number of agronomic characteristics which make it a good dedicated biofuel feedstock. *Jatropha* can grow in arid, semiarid and wasteland, requires minimal water and fertilizer and can survive on marginal soils. The oil content of the *Jatropha* seeds is also high, about 40%.

*Syringa* tree (*Melia azedarach* L.), also known as Indian or Persian lilac, is an invasive plant in South Africa. The mission of Syringa Bioscience (Pty) Ltd based in South Africa has been finding ways to utilize syringa trees for value-added product development. Since syringa berries contain significant amount of triacylglycerides, conversion of syringa berry oil to biodiesel has been explored.

In this study chemical and physical characteristics of canola, *jatropha* and syringa berry oil were determined. Oil samples were converted to biodiesel by using the traditional transesterification method. Sodium methoxide in methanol was used as catalyst. Material balances for conversion of oils to biodiesel were calculated. Viscosity, flash point, storage stability, cloud point, free and total glycerol content, differential scanning calorimeter cooling curves, mineral content and calorific value of biodiesel samples were measured. The quality of biodiesel made from *jatropha* and syringa oil was compared to that of biodiesel from canola oil.

**Cuphea Oil as a Potential Source of Biodiesel with Improved Properties.** G. Knothe, S. Cermak, R. Evangelista, USDA, ARS, NCAUR, USA

Biodiesel is usually produced from common vegetable oils such as soybean, rapeseed (canola), and palm as well as other feedstocks such as animal fats and used cooking oils. To enhance feedstock supply, other vegetable oils such as *jatropha* are of increasing interest. However, most of these feedstocks provide fatty acid profiles varying within the range of C16 and C18 fatty acids. To improve fuel properties such as cold flow and oxidative stability, either additives must be used and/or the fatty ester profile modified. The latter approach entails either changing the composition of biodiesel derived from "conventional" feedstocks or utilizing feedstocks with inherently different fatty acid profiles. In this work, a feedstock with a less common fatty acid profile, a variety of cuphea oil (PSR 23; a cross of *Cuphea viscosissima* x *C. lanceolata*) containing about 65% decanoic acid in its fatty acid profile was studied as biodiesel feedstock. Fuel properties such as cetane number, viscosity, oxidative stability and cold flow were investigated. Especially cold flow appears advantageous with the cloud point of cuphea methyl esters determined around -9 to -10°C. Cuphea, although facing technical issues regarding its wide-scale commercialization, is therefore of interest as

biodiesel feedstock and may serve as model for other oils with similar fatty acid profiles.

**Preparation of Jojoba Oil Ester Derivatives for Biodiesel Evaluation.** Shailesh N. Shah<sup>1</sup>, Brajendra K. Sharma<sup>1,2</sup>, Bryan R. Moser<sup>1</sup>, Sevim Z. Erhan<sup>1</sup>, <sup>1</sup>Food and Industrial Oil Research, National Center for Agricultural Utilization Research, Agricultural Research Service, U. S. Department of Agriculture, 1815 N. University St., Peoria, IL 61604, USA, <sup>2</sup>Department of Chemical Engineering, Pennsylvania State University, University Park, PA 16802, USA

The jojoba plant (*Simmondsia chinensis* L.) produces seeds that contain around 50 to 60 weight percent of inedible long-chain wax esters that are suitable as a potential feedstock for biodiesel production. Jojoba oil methyl esters (JME) were prepared in an effort to evaluate important fuel properties of jojoba-based biodiesel, such as kinematic viscosity, cloud point, pour point, cold filter plugging point, acid value, oxidative stability, and lubricity. A comparison was made with soybean oil methyl esters (SME) and relevant biodiesel fuel standards such as ASTM D6751 and EN 14214. JME was characterized using FTIR and <sup>1</sup>H and <sup>13</sup>C NMR. JME displayed superior low temperature properties and inferior kinematic viscosity versus SME. Blends (B5 and B20) of JME in ultra low sulfur diesel fuel (ULSD) were also evaluated for the aforementioned fuel properties and compared to an analogous set of blends of SME in ULSD and relevant petrodiesel fuel standards such as ASTM D975 and D7467. JME blends in ULSD displayed improved low temperature properties in comparison to neat ULSD and blends of SME in ULSD. In summary, jojoba oil has potential as an alternative, non-food feedstock for biodiesel production.

**Review of Pretreatment Procedures for Animal Fats as Feedstock for Biodiesel Production.** James Willits, Desmet Ballestra North America, Marietta, GA, USA

In today's economic market, it is critical to be able to utilize the most cost effective feedstock for a biodiesel production plant. In almost all cases animal fats require pre-treatment before entering the biodiesel process. This presentation will cover a range of animal fats with discussion of their quality ranges and the corresponding process systems required to deal those that will adversely effect the finished biodiesel quality.

**Agro-Industrial Residues as Low-Price Feedstock for Diesel-like Fuel Production by Thermal Cracking.** A.L. Santos, D.U. Martins, O.K. Iha, R.A.M. Ribeiro, R.L. Quirino, J.C. Rubim, P.A.Z. Suarez, LMC-IQ-UnB, Brasilia, DF, Brazil

Pyrolysis of industrial fatty wastes (soybean soapstock, beef tallow, poultry industry waste) was carried out in absence of catalysts. In all cases, an organic mixture of hydrocarbons and oxygenated compounds was obtained. These mixtures were distilled and diesel like fractions were isolated and characterized by GC-FID, GC-MS and FTIR, showing the formation of olefins, paraffins, and some oxygenated compounds such as carboxylic acids and esters. The main physical-chemical properties of those isolated diesel-like fuels (density, viscosity, distillation curve, carbon residue, copper corrosion test, cetane index, cold filter plugging point, acid index and heating value) were determined using ASTM standard methods and matched Brazilian specification for diesel fuel. CNPq, FINEP, MCT, MDA, FBB, FINATEC, CAPES

**Alternative Crops for Biodiesel Production in Brazil: Potential in Semi-Arid Regions.** S.M.P. Meneghetti, UFAL, Maceió, Alagoas, Brazil

Since the biodiesel program has been started in Brazil, the investigation of alternative sources of triacylglycerides from species adapted at semi-arid lands became a very important task for Brazilian researchers. Thus, we initiated studies with various alternative crops with the aim to evaluate some properties and chemical composition of the oil, as well as any potential application in biodiesel production.

**Safflower as an Alternative Feedstock for Biodiesel Production in Turkey.** Asli Isler, Filiz Karaosmanoglu, Istanbul Technical University, Maslak, Istanbul, Turkey

Biofuels are one of the biobased industrial products of the biorefinery technology. Production and use of biofuels have a lot of advantages on local, state and national levels. Biofuels are classified into four classes as first, second, third and

fourth generation biofuels. Biodiesel as one of the most common first generation biofuels uses as feedstock vegetable oils, animal fats and wastes. In the aspect of higher production costs and biofuel versus food-feed new oil seed resources are gaining importance, today. Although Turkey has appropriate lands for oil seeds plant production, production does not meet the consumption and oil seeds and vegetable oils are imported in large quantities. So production of oil seeds must be supported along well with the agriculture policies and appropriate energy agriculture plans. Safflower, which can be used for the production of biodiesel as a feedstock is one of the perfect candidates for Turkey. Since 1980's use of new seeds was one of the research subjects in Turkey. In this study, safflower is introduced with its history, properties and use in the world and in Turkey, safflower-biodiesel relationship is investigated according to the real data from 2007-2008 harvest season safflower agriculture in Turkey and safflower is presented as an alternative feedstock candidate for biodiesel production.

**Low Cost Photobioreactors for Algal Biofuel Production and Carbon Capture.** J. Butler, Solix Biofuels, Inc., Fort Collins, Colorado, USA

Photobioreactors facilitate the cultivation of specific strains of algae and allow optimal growth conditions to be maintained. To date, however, the high capital and operating costs of photobioreactors have prohibited their use for "low-valued" products such as biofuels. Recent efforts at Solix Biofuels have focused on the development of photobioreactor technology with high productivity, low capital cost, low operating cost, and low energy utilization. Efforts are now being expanded to develop the downstream production processes necessary to process the algae biomass into fuel and commercially viable co-products. The Solix system is applicable to a wide range of algae species. This presentation will provide details on the Solix photobioreactor technology, control and operational strategy, and downstream processes. Current and projected performance metrics are presented for the system operating with the eustigmatophyte *Nannochloropsis oculata*.

## WEDNESDAY

### MORNING

#### **PRO 4 / EXH 3: Processing Exhibitor Session**

Chair(s): T. Neuman, Westfalia Separator Inc., USA; and J. Mulholland, N. Hunt Moore & Associates, USA

**Using Centrifugal Force and High Vacuum to Distill Heat Sensitive Materials.** D. Casilio, R. Kromer, Myers Vacuum, Kitanning, PA, USA

A high vacuum centrifugal still is a physical process that has been designed to optimize separation of heat sensitive, high molecular weight material. This paper will detail the features of applying a very thin, continuously moving film on a heated rotor under high vacuum. The residence time is measured in tenths of seconds. This allows the user to make very fine cuts with the least amount of decomposition from heat. This short path still has the advantage of a condenser equal in size to the heated surface. A comparison to wiped and falling film principles will be shown and some applications will be presented.

**Controlling Process through Color Change.** T. Schwalbach, Optek Inc., Germantown, WI, USA

In line process control can be precisely done by monitoring the real-time changes of color intensity during the process. In measuring Chlorophyll to 300ppb, the Optek sensor will use the AOCS scale for Chlorophyll to measure low level green in oil. To measure the amount of clay residue in edible oils after the bleaching process, the Optek sensor will use the AOCS scale for red to monitor the success or identify interruption of the filtration process. The Optek process control concept is based on the Lambert-Beer's law of light absorption. The powerpoint presentation will address how in-line process control using light absorption will provide precision and repeatability.

**Purification of Glycerin from Biodiesel Plants.** P. Alasti, Artisan Industries Inc., Waltham, MA, USA

With the expected growth of biodiesel in the United States, as emphasized by Barak Obama during his Presidential

campaign, we can expect a glut of crude glycerin in the coming years, as more biodiesel plants come on stream. Refining the glycerin to various purities will be instrumental in insuring profitability regardless of feedstock and energy costs. We will present Artisan's refining process and compare it with two alternate processes currently available in the market.

**Purifine® PLC: Industrial Application in Degumming and Refining.** Tim Hitchman, Verenum Corporation, San Diego, CA, USA

Purifine® PLC is a unique enzyme product for application in the degumming step of edible and industrial oils processing. Use of Purifine® PLC results in increased oil yield, enhanced processing efficiency and other benefits. The product works by breaking down phospholipid impurities that are normally removed in the heavy phase during degumming. As a result neutral oil normally entrained in the gum is released and available for recovery. Furthermore, Purifine® PLC is unique in that it converts the phospholipid impurities into diacylglycerol (DAG), which is equivalent to neutral oil and provides additional oil yield. In total, neutral oil + DAG benefits are proportional to the phosphorus content of the oil, reaching 2% yield gains in the case of crude soybean oil with 1000 ppm phosphorus. Industrial scale implementation requires a minimal modification of most existing plant layouts. Use of the Purifine® PLC product results in a low phosphorus degummed oil that is easily refined for edible or biodiesel end uses.

**Waste Water Evaporation and Recycling in Fats and Oils Processing.** Sorin Dinu<sup>1</sup>, William Younggreen<sup>1</sup>, John Piazza<sup>2</sup>, <sup>1</sup>Alfa Laval Copenhagen A/S, Soborg, Denmark, <sup>2</sup>Alfa Laval Inc., Richmond, Virginia, USA

This paper discusses the minimization of water consumption in fats and oils processing by use of evaporation systems. Multiple-effect evaporation systems allow for concentration of contaminants in waste water streams such as the heavy phase from the wash stage centrifuge in a neutralization line or the effluent from vacuum systems. The water recovered from this concentration is clean enough to be reused in the process. System setup as well as process economics are presented.

**Increased Efficiency for Grains and Oilseeds Drying.** Farah Salaria, Solex Thermal Science, Calgary, AB, Canada

The drying of oilseeds extends the harvest season and prevents spoilage thus reducing field losses. Oilseeds have different limits on temperatures that they can be heated up to and varying ideal moisture content for storage. The drying process is governed by typical factors like ambient conditions outside, relative humidity, temperature, grain texture, sensitivity to heat, moisture content and toughness of the kernel, etc. Conventional methods involve high volume of hot air which is blown through the bed of oilseeds/grains. Use of hot air as the heating media as well as for moisture removal limits the efficiency of this technique. Large volumes of air must be heated for heat transfer and as the air picks up moisture from the oilseeds the temperature of air drops and the air reaches saturation. Much of this energy is lost up the stack. Distribution of air through grain beds and silos is often not ideal leading to non-uniform drying. Use of indirect heat through hot water or steam, in many cases obtained from a waste heat source, can radically reduce the volume of air, consumption of energy and emissions. This is achieved through plate heat exchanger designed for cross air flow for removal of moisture. The indirect heating reduces demand for a large volume of hot air and keeps the air temperature constantly high which enables the air to carry increased amounts of moisture before reaching saturation.

**HF Screw Press Evolution: Bigger and Better.** Harald C. Boeck, Harburg-Freudenberger Maschinenbau GmbH, Hamburg, Germany

**Latest Developments in Ice Condensing in Oil Refining: An Economical and Environmental Must.** Marc Kellens, Desmet Ballestra, Belgium

The ice condensing technology has been introduced in the edible oil refining industry since the early 90's. Its initial

purpose was to reduce the environmental impact of a refinery. Despite a proven reliability and good industrial track record, the technology today is still being mainly considered for those cases where local permits or environmental restrictions discourage the use of classical barometric vacuum systems. The economical savings were often found not enough to value the higher investment cost, as the capital return was in the range of 4 to 6 years. New technological developments and higher energy prices have made the ice condensing technology more efficient and hence more competitive on both capital as well as operational cost. Capital investment return is now less than 2 years. Ice condensing systems maintain low pressures, typically below 2 mbar, in deodorisation processes by the condensation and freezing of sparge steam from the deodorisation process on internally cooled tubes. These units substantially reduce steam consumption (both motive and sparge steam) as compared to classical vacuum systems. The Sublimax dry ice condensing vacuum system has been further developed and improved over the years, with a minimum refrigerant content and even lower energy consumption, using more energy efficient chillers. The current vertical ice condensing technology reduces pressure drops to a minimum, allowing an operating pressure in the deodoriser vessel even below 1 mbar. This opens new perspectives for the mild refining of heat sensitive oils like for example fish oil and even cocoa butter. Examples of some technological innovations and resulting applications, are given to demonstrate the potential of the new generation vacuum ice condensing.

**Pretreatment for Biodiesel.** T. Neuman, Westfalia Separator Inc., USA.

## AFTERNOON

### **PRO 5: General Processing**

Chair(s): G. Hatfield, Bunge Canada, Canada; and V. Jain, University of Arkansas, USA

**Influence of Fatty Acid Composition on Lipase Efficiency in Interesterification.** W.D. Cowan<sup>1</sup>, H.S. Yee<sup>2</sup>, J. Hemann<sup>3</sup>, H.C. Holm<sup>3</sup>, <sup>1</sup>Novozymes UK, Chesham, Bucks, UK, <sup>2</sup>Novozymes MY, Kuala Lumpur, Malaysia, <sup>3</sup>Novozymes A/S, Bagsvaerd, Denmark

Enzymatic interesterification of lipids is now well established as a process for fat modification but plant scale experience shows that not all fats behave in the same manner. The fatty acid composition has been shown to be an important factor in how lipases function. This paper examines the influence of fatty acid type on lipase effect and indicates how choice of lipase can be matched to fat composition to improve interesterification efficiency. Four different lipases and several fats of different composition are included in this study.

**A New Fluidized Bed Desolventizer for Gentle Rapeseed Meal Processing.** Frank Pudel<sup>1</sup>, Karl-Heinz Leidt<sup>1</sup>, Klaus Weigel<sup>2</sup>, Reinhard Zettl<sup>2</sup>, Lothar Mörl<sup>3</sup>, <sup>1</sup>Pilot Pflanzenöltechnologie Magdeburg e.V., Magdeburg, Germany, <sup>2</sup>Dr. Weigel Anlagenbau GmbH, Magdeburg, Germany, <sup>3</sup>Otto-von-Guericke-Universität, Magdeburg, Germany

In a conventional desolventiser/toaster the meal is treated under conditions which lead to the denaturation of the contained proteins. Such proteins have lost their techno-functional potential and are not suited for applications out of animal feeding. A more gentle meal processing is possible by using the fluidized bed technology. Generally there are two different options to remove the solvent from the meal: a) by nitrogen or b) by superheated hexane. The presentation will describe the developed pilot scale fluidized bed desolventiser and will give results from test trials with rapeseed meal concerning to the obtainable hexane content and protein quality.

**Viscosity of Fish Oil in Equilibrium with High Pressure Carbon Dioxide.** B. Seifried, F. Temelli, University of Alberta, Edmonton, Alberta, Canada

Viscosity affects a wide variety of processes through its impact on mass, heat and momentum transfer. Optimal design of processing equipment involving high pressure carbon dioxide (CO<sub>2</sub>) requires viscosity data. Therefore, the objective of this study is to determine the viscosity of fish oil in equilibrium with CO<sub>2</sub>. The viscosity of fish oil (FO) in

equilibrium with CO<sub>2</sub> was determined in triplicate at 40, 55, and 70°C and pressures ranging from 0.1 to 5.65 MPa using a rotational rheometer equipped with a high pressure cell at shear rates of 200 to 600 s<sup>-1</sup>. Viscosity of FO determined at a shear rate of 300 s<sup>-1</sup> decreased with pressure due to dissolution of CO<sub>2</sub>. However, at higher temperatures the solubility of CO<sub>2</sub> in FO is lowered leading to a less pronounced viscosity decrease with pressure. The highest viscosity drop of 80% (25.6 to 5.1 mPa.s) with a pressure increase from 0.1 to 5.65 MPa was observed at 40°C, whereas it was only 57% (11.4 to 4.9 mPa.s) at 70°C. Rheological measurements showed dilatant behavior for the fish oil saturated with CO<sub>2</sub> at elevated pressures, with a flow behavior index increasing with pressure from 1.0 to 1.3. Novel process developments and design of equipment, such as spray nozzles, filters and packed columns will benefit from a better understanding of the rheological behavior.

**Removal of PCBs and Dioxins from Fish Oils via Combined Adsorption and Stripping.** R. Verhe<sup>1</sup>, B. De Meulenaer<sup>1</sup>, W. De Greyt<sup>2</sup>, J. Maes<sup>2</sup>, J. Vila Ayala<sup>2</sup>, <sup>1</sup>Ghent University, Faculty of Bioscience Engineering, Department of Organic Chemistry, Ghent, Belgium, <sup>2</sup>Desmet-Ballestra, Zaventem, Belgium

European regulation on the maximum dioxins and furans in fish oils for animal feeding (6 pg WHO-TEQ / g oil) or human consumption in refined fish oils as ingredient in "healthy" foods and nutraceuticals (2 pg WHO-TEQ / g oil) are currently existing. In addition European regulation regarding PCB's is expected very soon. Fats intended for human consumption need to be purified from dioxins, furans and PCB's. The aim of this study is the screening of different polar and apolar adsorbents on the efficiency to remove dioxins and/or PCB's and the influence of adsorption on fat oxidation parameters and nutritional parameters. Screening various types of adsorbents establishes that filter aid, silica powder and bleaching earth (0.1-0.5% (w/w) at 70°C, 40 min) have no adsorption for dioxins and PCB's. However activated carbon shows excellent results: removal of 99 % dioxin, 90% non-ortho PCB's and 20-55% mono-ortho PCB's. Optimized conditions are: 20-40 min, 50-90°C, 0.25-0.75 % (w/w). The influence on the oxidative stability and nutritional characteristics was studied by the determination of primary oxidation products, secondary oxidation products, conjugated diens/triens, polar fraction, dimers and polymers, fatty acid composition and concentration of vitamins A and E. A combination of active carbon treatment and deodorization (stripping 180-220°C, steam 1-4%, 1-4 mbar, 60 min) removes the majority of dioxins and PCB's. Active carbon does not fully remove PCB's meanwhile batch deodorization does not fully remove dioxins. For both techniques the ideal conditions were selected according to the best results in terms of removal of contaminants and preservation of the nutritional properties.

**Catalyst Removal from Photoirradiated Soy Oil to Obtain CLA-rich Soy Oil for Food Applications.** V. Jain, A. Proctor, University of Arkansas, Fayetteville, AR, USA

Studies with conjugated linoleic acid (CLA) have shown it effective in reducing human body fat and as an anti-carcinogenic, anti-atherosclerotic, anti-diabetic agent in animal models. CLA-rich soy oil has been recently produced by simple photo-irradiation of soy oil linoleic acid using iodine as a homogenous catalyst. The objectives of this research were to a) identify new iodo-compounds after irradiation and b) determine the most effective means to remove iodine from the oil. <sup>1</sup>H NMR and GC-MS were used to identify any iodo-compounds following photo-irradiation and solvent partition, adsorption and deodorization were examined as potential iodine removal methods. Water, hexane, and acetone were used for partitioning and clays, activated carbons, silica, magnesium silicate (Magnesol), and ion exchange resin cartridges were used in adsorption studies with deodorization and thin film evaporation. <sup>1</sup>H NMR and GC-MS did not identify any iodo-compounds. In addition, water was not an effective partition solvent indicating there is no HI formed in the oil and thereby suggesting iodine only as molecular iodine. Magnesol adsorption was found to be the most effective means of removing iodine. The absence of organic iodo-compounds and efficient removal by adsorption offers the opportunity for CLA rich oil to be used commercially.

**Effect of Minor Oil Constituents on Soy Oil CLA Yields and Oxidative Stability.** Tanushree Tokle, Vishal Jain, Andy Proctor, University of Arkansas, USA

We showed that the degree of oil refining affected conjugated linoleic acid (CLA) yields from soy oil linoleic acid (LA), with CLA yields being proportional to the degree of oil refining. Therefore, the objective was to determine the



effect of minor soy oil components on CLA yield and oxidative stability. Soy oil with tocopherol levels from 1100-2600ppm, lutein from 0.8-50.8ppm, free fatty acids (FFA) from 200-3200ppm and phospholipids from 600-3600ppm, were each irradiated in a pilot scale photoisomerization unit for 12hrs at 45oC with 0.35% iodine as the catalyst. CLA was determined by FAME-GC and oxidative stability by weight change at 60oC. Phospholipid, FFA and lutein levels up to 1100ppm, 1200ppm and 30.8ppm, respectively, produced CLA yields similar to levels in the control RBD soy oil. Tocopherol levels up to 1400ppm increased CLA yields. However, increasing levels beyond 1400ppm reduced CLA yields. Phospholipids caused the most rapid decline in CLA yields with increasing concentration, while lutein had little effect. Phospholipids, fatty acids and tocopherols all increased oxidation induction time to varying degrees but lutein had no affect. With the exception of tocopherols, low levels of each component did not affect CLA yields until a threshold level was reached.

**Enzyme-assisted Aqueous Extraction of Oil and Protein from Canola (*Brassica napus* L.) Seeds.** Sajid Latif<sup>1</sup>, Levente L. Diosady<sup>2</sup>, Farooq Anwar<sup>1</sup>, <sup>1</sup>Department of Chemistry & Biochemistry, University of Agriculture, Faisalabad-38040, Pakistan, <sup>2</sup>Department of Chemical Engineering and Applied Chemistry, University of Toronto, Toronto, Ontario M5S 3E5, Canada

Four enzymes, Protex 7L, Multifect Pectinase FE, Multifect CX 13L, and Natuzyme, were tested for their effectiveness in releasing oil and protein during aqueous extraction. Enzyme-extracted oil content of canola seeds (22.2 to 26.0%) was found to be significantly ( $P < 0.05$ ) higher than that of the control (without enzyme) (16.48%). An appreciable amount of protein (3.5-5.9%) originally present in the seed was extracted into the aqueous and creamy phases (ACP's) during aqueous extraction of oil. The physico-chemical properties of oils extracted from canola seed by conventional solvent extraction, and aqueous extraction, with or without enzyme addition were compared. Significant ( $P < 0.05$ ) differences were observed in free fatty acid, specific extinctions at 232 and 270 nm, peroxide value, color (1-in. cell) and concentration of tocopherols ( $\alpha$ ,  $\gamma$ , and  $\delta$ ). However, no significant variation ( $P < 0.05$ ) was observed in iodine value, refractive index, density, saponification value, unsaponifiable matter and fatty acid composition. A better oil quality was obtained with aqueous extraction (with and without enzyme) then with solvent extraction. While the enzymes enhanced the oil extraction, the oil yield was still significantly ( $P < 0.05$ ) lower than that obtained by solvent (hexane) extraction.

**Enzymatic Degumming - Recent Developments.** F. Galhardo, Bunge, 725 N Kinzie Av Bradley IL 60915, USA

The use of PLA and PLC phospholipases in oil degumming result in degradation of phospholipids respectively into their lyso and phosphoro forms, reducing oil losses through emulsion. This session will review data from plant scale applications

**Valorization of Used Frying Oil in the Field of Biolubricants by Fractionation.** X. Pages, M. Gaud, C. Alfos, B. Gadenne, ITERG, France

The European collective research program IBIO LAB: Improvement of Biolubricant Manufacturing and Development thanks to the Obtaining of EcoLabels in a wide range of industrial Sectors, is based on the use of different feedstocks; Used Frying Oils (UFOS) due to their low cost are a raw material of interest; ITERG's technology department has studied both on laboratory and industrial scales the different ways of processing UFOS. refining trials and fractionation trials have be done following dry fractionation process and allow to obtain interesting fractions: their characteristics (fatty acid composition, viscosity; melting point, oxidation stability) show the possibility of formulation of low cost greases.

## Processing Posters

Chair(s): S. Metin, Cargill, USA

### Critical Fluids for Lipid Processing.

F. Eller, S. Taylor, J. Teel, NCAUR, ARS, USDA, Peoria, IL, USA

A lipase interesterification of soybean oil triacylglycerides with ethyl ferulate to produce feruloylated acylglycerols has been described and patented. These feruloylated acylglycerols, termed SoyScreen™, are believed to have potential as natural sunscreens and as antioxidants for the cosmetic and food industry. Critical fluid technology has been used in several ways to improve this lipid processing method. Liquid carbon dioxide (L-CO<sub>2</sub>) has been used to purify the SoyScreen™ product from the reaction mixture. Both by-product fatty acid ethyl esters (FAMES) and unreacted ethyl ferulate are separated from the reaction mixture using a counter-current fractionation method. A method utilizing supercritical carbon dioxide in conjunction with a silica column was also used to purify the ethyl ferulate used in this reaction to provide a nearly snow-white product. After it was determined that partially deacylated sunflower oil had some advantages over soybean oil as a starting material, a continuous counter-current fractionation L-CO<sub>2</sub> method was developed to remove by-product fatty acid propyl esters from the propanolysis reaction mixture. Critical fluid technology is being used to enhance the synthesis of SoyScreen™ by cycling CO<sub>2</sub> through a bed of Novozyme 435 containing triacylglycerides and ethyl ferulate. In this process, CO<sub>2</sub> removes FAMES from the reaction mixture and precipitates them in a reduced pressure receiver before recycling the CO<sub>2</sub>.

### **Detect Changes in Protein Structure of New Co-Products of BioEthanol Production Using IR Spectroscopy: Comparison between Blend DDGS, Wheat DDGS vs. Corn DDGS, Wheat vs. wheat DDGS, and Corn vs. Corn DDGS.**

Peiqiang Yu, Daal Damiran, College of Agriculture and Bioresources, University of Saskatchewan, Saskatoon, SK, Canada

The objective of this study was to use IR spectroscopy to identify the differences in protein amides profile between wheat vs. wheat DDGS, corn vs. corn DDGS, between bio-ethanol plants, between wheat DDGS, corn DDGS and blend DDGS. The protein IR spectrum has two primary features, the protein amide I (ca. 1600-1700 cm<sup>-1</sup>) and amide II (ca. 1500-1560 cm<sup>-1</sup>) bands. The amide I band can be used to determine protein secondary structures. The amide I and II profile depends on the protein molecular structural chemical make-up. Results showed that the protein structural chemical makeup was revealed and identified. Amide I and II peak area changed ( $P < 0.05$ ) by bioethanol processing. Amide I-to-II ratio differed between wheat and wheat DDGS (4.61 vs. 3.08,  $P < 0.05$ ), corn and corn DDGS (4.56 vs. 2.21,  $P < 0.05$ ), No difference ( $P > 0.05$ ) between wheat and corn, between bio-ethanol plants but significances between wheat DDGS, blend DDGS and corn DDGS was detected. These results indicated that bio-ethanol processing change original protein structure (amide I and II profile) which may play a role to determine nutritive value.

### **Processing of *prunus virginiana* for Oil, Hull, and Meal Fractions.**

Sunmin Wang<sup>1</sup>, Amberly Faye<sup>2</sup>, Bonnie Li<sup>1</sup>, Johanna Clancy<sup>1</sup>, Martin Reaney<sup>1</sup>, <sup>1</sup>Department of Food and Bioproduct Sciences, University of Saskatchewan, 51 Campus Drive, Saskatoon, Saskatchewan, S7N 5A8, Canada, <sup>2</sup>Department of Agricultural and Bioresource Engineering, University of Saskatchewan, 57 Campus Drive, Saskatoon, Saskatchewan, S7N 5A9, Canada

Fresh chokecherries were collected from the University of Saskatchewan (SK, Canada) horticultural farm. They were subsequently water washed and fractionated into juice/pulp and pit fractions using a small commercial juicer. The clean chokecherry pits were dried and passed through a roller cracking mill. Following the pit cracking, a mixture of cracked hull and mostly intact kernels was obtained. The kernels were subsequently separated by air classification and screening. The kernels which made up 5% of fresh fruit weight contained between 42 and 48% oil. Chokecherry pits collected from approximately 1,000 trees were studied for pit weight, pit oil content, the amount of oil per single pit, and fatty acid profile of kernel oils. The results showed that kernel oil had less than 6 percent saturated fats with combined oleic and linoleic acid ranging from 89.6% to 91.6%. The oil also contained less than 7 percent eleostearic acid. Analysis of pit oil content by non-invasive proton NMR (miniSpec, Bruker) methods identified high oil chokecherry varieties.

### **Preparation and Characterization of Phytosterol Nanodispersions.**

Wai Fun Leong<sup>1</sup>, Chin Ping Tan<sup>1</sup>, Yaakob Bin Che Man<sup>1</sup>, Oi Ming Lai<sup>2</sup>, Kamariah Long<sup>3</sup>, Misni Misran<sup>4</sup>, <sup>1</sup>Faculty of Food Science and Technology, Universiti Putra Malaysia, Serdang, Malaysia, <sup>2</sup>Faculty of Biotechnology and

Biomolecular Sciences, University Putra Malaysia, Serdang, Malaysia, <sup>3</sup>Malaysian Agricultural Research & Development Institute, Kuala Lumpur, Malaysia, <sup>4</sup>Faculty of Science, University of Malaya, Kuala Lumpur, Malaysia

The purpose of this study was to prepare and characterize the phytosterol nanodispersion for food formulation. Phytosterol nanodispersions were prepared by mixing the emulsifier containing aqueous phase and the phytosterol containing organic phase using high-pressure homogenization. Organic solvent was then removed by evaporation under reduced pressure. The effect of four different types of organic solvent (hexane, isopropyl ethanol, ethanol and acetone), three organic to aqueous phase ratios and four homogenization pressures on volume-weighted mean diameter ( $D_{4,3}$ ), polydispersity index (PDI) and concentration of phytosterol in prepared nanodispersions were observed. The results showed that among the four different types of organic solvent used, hexane was able to produce the smallest particle size at  $D_{4,3}$  of approximately 50 nm. Nanodispersion prepared by higher homogenization pressure and higher organic to aqueous phase ratio resulted in larger phytosterol nanoparticles at significant level of  $P < 0.05$ . The losses of phytosterol after high pressure homogenization were ranging from 10–28%, and enhanced by an increase in homogenization pressure. Evaporation of organic solvent by evaporation contributed to minimal losses of phytosterol at 0.5–7%.

### **Thermal and Pressure Treatment of Emulsion Recovered from Aqueous Extraction Processing of Soybean Flour.**

Stephanie Jung, Abdullah Mahfuz, Iowa State University, Center for Crops Utilization Research, USA

High-pressure pasteurization (600 MPa, 5 min, 25°C) and sterilization (600 MPa, 5 min, 75°C) were compared as alternatives for heat preservation (85°C for 20 s and 121°C for 12 min) of the cream emulsion recovered from aqueous extraction of soybean flour. The effects of these treatments on the properties and stability of the emulsion were established by determining zeta potential, particle size, light microscopy, rheological properties, creaming stability and microbial load after treatment and during 42 days of refrigerated storage. The results suggest that aqueous extraction of soybean flour provides a cream emulsion which properties can be further enhanced by high-pressure pasteurization. A new way to obtain soy emulsion for food applications was therefore identified.

### **Rates of Heterogeneous Acid Catalyzed Methanolysis of Trilaurin, Trimyristin, Tripalmitin, Triolein, and Mixed Triglycerides.**

J.D. Cain<sup>1</sup>, E.G. Alley<sup>1</sup>, R.A. Hernandez<sup>1</sup>, W.T. French<sup>1</sup>, M.C. Paraschivescu<sup>1</sup>, W. Holmes<sup>2</sup>, <sup>1</sup>Mississippi State University, Dave C. Swalm School of Chemical Engineering, Mississippi State, MS 39762, USA, <sup>2</sup>Mississippi State Chemical Laboratory, Mississippi State, MS 39762, USA

Trilaurin, trimyristin, tripalmitin, triolein, and soybean oil were the triglycerides chosen for this study. Heterogeneity was assured by determining the solubility of each in methanol. A molar concentration of 110  $\mu\text{mol/mL}$  in methanol was chosen because this assured heterogeneity of all five materials. Transesterification of each triglyceride was performed in 0.10% sulfuric acid in methanol, stirred at 60°C. Transesterification of soybean oil was performed on 100 mg quantities following the same experimental procedure as for the individual triglycerides. At each time interval the entire sample was quenched with 2% aqueous sodium chloride/5% sodium bicarbonate and extracted with toluene. Appropriate dilutions were made and each sample solution was quantified using an internal standard and high temperature gas chromatography with flame ionization detection.

### **Extraction of Tocopherol-enriched Oil from Roselle Seed by Supercritical Fluid Extraction (SFE-CO<sub>2</sub>).**

Chin Ping Tan<sup>1</sup>, Kar Lin Nyam<sup>1</sup>, Yaakob B. Che Man<sup>1</sup>, Oi Ming Lai<sup>2</sup>, Kamariah Long<sup>3</sup>, <sup>1</sup>Faculty of Food Science & Technology, Universiti Putra Malaysia, Serdang, Selangor, Malaysia, <sup>2</sup>Faculty of Biotechnology and Biomolecular Sciences, Universiti Putra Malaysia, Serdang, Selangor, Malaysia, <sup>3</sup>Malaysian Agricultural Research & Development Institute, Kuala Lumpur, Malaysia

Natural tocopherol was extracted by supercritical fluid extraction (SFE-CO<sub>2</sub>) from roselle seed. Several SFE-CO<sub>2</sub> parameters, such as extracting pressure, extracting temperature, and flow rate of carbon dioxide were examined as the

independent variables of central composite design (CCD). Through the response surface methodology (RSM), the optimal processing conditions were determined and the quadratic response surfaces were drawn from the mathematical models. The results demonstrated that the quadratic terms of extracting pressure, quadratic terms of flow rate of carbon dioxide, pressure x temperature interaction, and temperature x flow rate of carbon dioxide interaction significantly affected the yield of the natural tocopherol extraction. The optimal conditions for roselle seed were extracting pressure 200 bar, extracting temperature 80 °C, and flow rate of carbon dioxide 20 ml/ min. These optimum conditions yielded a 89.75 mg/ 100 g tocopherol concentration. Close agreement between experimental and predicted values was obtained.

### **Development of a Recyclable Nanosheet Heterogeneous Catalyst for Continuous Biodiesel Synthesis Utilizing Waste Grease as Feedstock.**

Erin Jo Mercer, Fathi Halaweish, South Dakota State University, Brookings, SD, USA

Biodiesel (BD) is a renewable fuel similar to petro-diesel, allowing for distribution by available infrastructure and use in diesel engines. Though current production results in a benign product, the process is not environmentally favorable. Our objective is to develop a heterogeneous catalyst for biodiesel synthesis that reacts a broad range of feedstock, so as to best utilize local recycled resources, under catalytic conditions that are environmentally benign. Studies of titanium nanosheets, a strong acid reported effective in the esterification of acetic acid and ethanol, are underway for application in transesterification for BD synthesis. Catalyst synthesis is according to Hervieu et al., and optimal conditions for catalyst activation are being studied in our lab. Our initial work utilizing soybean oil feedstock shows up to 80% conversion to BD, achieved in one hour in an open system. Reacting used Burger King® grease has shown significant conversion in early trials as well. Optimization of time, temperature, pressure, and stir-rate is necessary in continuing development of recycled feedstock use. NMR and ASTM D 6584 analysis will be compared to current production methods. Ultimately, an efficient means of production could permit an existing BD industry to expand and a subsequent rise in bioenergy consumption.

### **Comparison of the Color and Microstructural Properties of Soy Proteins Dried With Spray-Drying, Freeze-drying and INERJET.**

Y Arcand<sup>1</sup>, J Boye<sup>1</sup>, M Benali<sup>2</sup>, J Moore Gagne<sup>3</sup>, P Dufresne<sup>3</sup>, D Montpetit<sup>1</sup>, M Marcotte<sup>1</sup>, <sup>1</sup>Agriculture & Agri-Food Canada, St Hyacinthe, Quebec, Canada, <sup>2</sup>Natural Resources Canada, Varennes, Quebec, Canada, <sup>3</sup>University of Sherbrooke, Sherbrooke, Quebec, Canada

The objective of this work was to study the effect of drying technique and conditions on color, thermal and microstructural properties of soy protein powders. Soy proteins were extracted using alkaline extraction followed by isoelectric precipitation and/or ultrafiltration. The samples were dried using one of three drying techniques (freeze drying, spray drying and jet spouted bed drying with inert particles). The effect of fat content (1.5, 5% oil) was also studied. Colour of the soy samples was measured against the CIELAB scale using a colorimeter. Thermal properties (enthalpy and transition temperatures (onset denaturation temperature, maximum denaturation temperature, width of peak at half-height) were measured with a differential scanning calorimeter. Microstructural differences in the dried powders were studied with a scanning electron microscope. Our results showed that while the drying technique and conditions generally had minimal effect on color and thermal stability of the dried protein powders, the microstructural properties of the powders were, distinctly different which could impact their hydration properties.

### **Functional Properties of Soy Protein Powders Dried on Inert Particles.**

J Boye<sup>1</sup>, Y Arcand<sup>1</sup>, M Benali<sup>2</sup>, J Moore Gagné<sup>3</sup>, P Dufresne<sup>3</sup>, D Montpetit<sup>1</sup>, M Marcotte<sup>1</sup>, <sup>1</sup>Agriculture and Agri-Food Canada, St Hyacinthe, Quebec, Canada, <sup>2</sup>Natural Resources Canada, Varennes, Quebec, Canada, <sup>3</sup>University of Sherbrooke, Sherbrooke, Quebec, Canada

Soy proteins are increasingly being used in food formulation due to their excellent nutritional and functional properties. Spray drying is the main technique currently used for the drying of soy protein concentrates and isolates. We evaluated a novel jet-spouted bed drying technique with Teflon beads used as inert particles (namely INERJET) to produce soy protein powders and compared the physico-chemical and functional properties (protein solubility, emulsifying and foaming properties, fat absorption and water hydration capacities) of the powders with soy proteins

dried using freeze-drying and spray-drying. Rheological and gelling properties of the soy proteins were also evaluated. Our results showed that while differences in the functional properties of the powders were observed as a function of drying conditions, the functional properties of the soy protein powders dried using the novel INERJET technique was generally comparable to those dried using spray drying. The jet-spouted bed drying technique is a novel drying method that could offer some distinct advantages to the food processing industry such as compactness, higher energy efficiency, and enhanced flowability of soy protein powder.

### **Scaling-up Enzyme-assisted Aqueous Extraction Processing of Soybeans.**

J.M.L.N. de Moura, N.M. de Almeida, L.A. Johnson, Center for Crops Utilization Research, Iowa State University, Ames, IA, USA

Enzyme-assisted aqueous extraction processing (EAEP) of flaked and extruded soybeans was scaled up to 2 kg and the effects of different extrusion and extraction conditions were evaluated. Standard single-stage EAEP at 1:10 solids-to-liquid ratio was used to evaluate the effects of screw speed and of extruding directly into water. Increasing extruder screw speed from 40 to 90 rpm improved oil extraction yield from 85 to 95%. Oil, protein and solids extraction yields of 97 and 95%, 86 and 84%, and 78 and 77% were obtained when extruding directly into water and not extruding into water, respectively. Scaling-up standard single-stage EAEP yielded 97, 86, and 78% of total oil, protein, and solids extraction, respectively, while scaling-up two-stage countercurrent EAEP at 1:6 solids-to-liquid ratio yielded 99, 94, and 83% of total oil, protein, and solids extraction, respectively. These yields were similar to those obtained at 0.08 kg scale, but higher oil contents were observed in the skim fractions produced at larger scale for both processes. Extraction conditions in the two-stage countercurrent EAEP were modified during scale-up tests. Increasing reaction time (15 min to 1 h) and slurry pH (8.0 to 9.0) in the first extraction increased oil yield in the (cream + free oil) fraction from 76 to 87% and the oil yield of the skim fraction decreased from 23 to 12%.

### **Optimized Flaking Conditions for Enzyme-Assisted Aqueous Extraction Processing of Soybeans.**

J.M.L.N. de Moura, N.M. de Almeida, L.A. Johnson, Center for Crops Utilization Research, Iowa State University, Ames, IA, USA

Soybean moisture (7.2-12.8%) and conditioning temperature (50.9 -79.1°C) were evaluated for their effects on total oil extraction and oil distribution among the fractions generated by the enzyme-assisted aqueous extraction processing. An experimental design (2<sup>2</sup>), with three center points and four axial points, was used. Oil extraction was performed by using two-stage countercurrent EAEP at 1:6 solids-to-liquid ratio, 0.5% protease (wt/g extruded flakes), pH 9.0, and 50°C for 1h. Total oil extraction improved when using soybean-moisture contents ranging from 8 to 12% but was not affected by conditioning temperature. Higher (79.1°C) and lower temperatures (50.9°C) improved free oil extraction and reduced emulsion production. Free oil extraction increased when increasing soybean moisture content from 10 to 12.8% (to a higher extent at temperature in the axial points) although total oil extraction was reduced at 12.8% moisture. Oil content in the skim fraction was not significantly affected by temperature and moisture, although undesirable high oil content was observed at 8% moisture and 55°C. High total oil extraction, associated with high free oil yield, low cream production, and low oil content in the skim was obtained at moisture and temperature combinations of 10% and 50.9°C and 12% and 75°C.

### **The Supercritical Fluid Extraction of Flax Lignans.**

L. Comin, F. Temelli, M.D.A. Saldana, University of Alberta, Edmonton, Alberta, Canada

Lignans, such as secoisolariciresinol diglucoside (SDG) found in flax, have been implicated in the prevention of hormonally related cancers as well as other prevalent diseases. These compounds are typically extracted from the plant matrix using organic solvents, which raise environmental and health concerns. Supercritical carbon dioxide (SCCO<sub>2</sub>) is a non-toxic, cheap solvent, which, when combined with polar modifiers, can be used to extract polar compounds. The objective of this study was to determine the optimal processing conditions for the extraction of SDG by SCCO<sub>2</sub>. Extraction of SDG from ground, defatted flax seeds was performed with SCCO<sub>2</sub> modified with ethanol (0, 10 and 20 mol%). Three temperatures (40, 50 and 60 oC) and 3 pressures (35, 40 and 45 MPa) were selected, and a Box-Behnken design was used. Extracts, taken over a 6 hr extraction period, were analyzed for SDG using HPLC. No significant effects of temperature, pressure and solvent modifier level on SDG solubility were found, and there was no

significant difference between the solubilities of SDG in SCCO<sub>2</sub> at the different conditions tested. However, the model indicated that a solubility of 0.898 µg SDG/ L solvent (ambient conditions) could be obtained at 45 MPa, 60 oC and 7.77 mol% ethanol. These results indicate that further research is needed to demonstrate efficient use of SCCO<sub>2</sub> for SDG extraction.

### **Glyceroxides of Potassium, Sodium and Lithium as Biodiesel Catalyst.**

Felicia, Hwee Yoong Gok, Jianheng Shen , Gabrielle Schutte, Ramaswami Samminaiken, Martin Reaney, University of Saskatchewan, Sskatoon, Sk Canada

The glycerol product of a sodium catalyzed biodiesel production reaction can solidify as the methanol is evaporated. A similar reaction is observed when glycerol is combined with aqueous sodium hydroxide. We have investigated the composition of the solid material and found that it may potentially be recycled as a catalyst for biodiesel production. A single crystal, that had formed spontaneously when glycerol and aqueous sodium hydroxide were mixed was subjected to x-ray diffraction analysis and a high resolution spectrum was obtained. It was demonstrated that the crystal was a glyceroxide that excluded both water and free glycerol. Furthermore, the same material may be recovered from waste glycerol remaining after biodiesel production. We observed, as have others, that crystalline sodium glyceroxide was an excellent catalyst for the production of biodiesel. Recovering a portion of the catalyst used in biodiesel production will be discussed.