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## Microreactors - An innovative tool for development of transesterification reaction continuous processes

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## WEDNESDAY

### MORNING

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#### PRO 4: General Processing

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Chair(s): J. Mulholland, N. Hunt Moore & Assoc Inc., USA; and T. Gum, Agribusiness & Water Tech Inc., USA

**New Opportunities in the Enzymatic Refining and Modification of Fish Oils.** W.D. Cowan<sup>1</sup>, H.S. Yee<sup>2</sup>, M.L. Damstrup<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 condensation of Fatty Acid Ethyl Esters is commonly applied in the production of highly concentrated fish oil products for nutraceutical and other applications. However, the whole production process utilizes a number of chemical reactions which result in yield loss and/or damage to the sensitive unsaturated fatty acids. This paper examines new findings on how enzymatic degumming and esterification can be applied to improve overall process efficiency and increase the sustainability of the fish oil process. Alternative reactor configurations are also considered to maximize enzyme working life.

**Reducing Energy Consumption in Biodiesel Purification.** M. Hastie, M.A. Dubé, A.Y. Tremblay, University of Ottawa, Ottawa, Ontario, Canada

The biodiesel purification process typically generates a wastewater stream containing residual catalyst, glycerol, methanol, and soaps. This stream must be treated prior to recycling or discharge, resulting in high purification costs and energy usage. In many biodiesel plants, evaporation is the preferred means of water purification for the purpose of recycling. The proposed Renewable Fuel Standard (RFS2) includes a Life Cycle Analysis (LCA) when determining if a fuel qualifies as an advanced biofuel. The LCA includes the energy consumed in regenerating the wastewater. The direct reuse of this water can result in considerable energy savings. Biodiesel produced from waste frying oil and RBD canola oil was washed with water with increasing sodium content to simulate the conditions encountered in a counter-current biodiesel purification system. Partition coefficients of the contaminants of interest, including sodium, glycerol and soaps, between the biodiesel and water phases were determined using ICP Spectroscopy and GC. The energy consumed in removing the impurities from the biodiesel was studied. The impact of regenerating the wastewater stream by evaporation, ultrafiltration or ion exchange on energy consumption was determined. Various counter-current washing schemes were studied to decrease water consumption in the purification process.

**Micoreactors - An Innovative Tool for Development of Transesterification Reaction Continuous Processes.** R. Richard<sup>1,2</sup>, S. Thiebaud-Roux<sup>1,2</sup>, L. Prat<sup>3</sup>, <sup>1</sup>Université de Toulouse; INPT; LCA (Laboratoire de Chimie Agro-Industrielle); ENSIACET, F-31030 Toulouse, France, <sup>2</sup>INRA; LCA (Laboratoire de Chimie Agro-Industrielle), F-31030 Toulouse, France, <sup>3</sup>Université

de Toulouse; INPT; CNRS; Laboratoire de Génie Chimique; UMR 5503, F-31030 Toulouse, France

To substitute fossil fuels, biodiesel can be produced from vegetable oils, animal fats, and waste cooking oils by transesterification with ethanol. This reaction, generally conducted in batch reactors, leads to high conversion of triglycerides into ethyl esters with diglycerides and monoglycerides as reaction intermediates and glycerol as by-product. Function of the scheme and the thermokinetic properties of the system, continuous processes may withdraw existing obstacles of batch processes such as the large number of steps, secondary reactions, stable equilibria and difficulties to separate the products. This system is complex due to phase equilibria and important coupling of phenomena (reaction, mixing, heat and mass transfers). Hence, to properly design a continuous process, numerous data are required. In this work, we transferred the batch reaction into a continuous microstructured device, which induces a better control of heat and mass transfers. Furthermore, it enables us to perform the reaction with small amounts of reactants to screen the operating conditions. The results show that reaction and separation can be carried out consecutively or simultaneously in microreactors and products can be obtained with higher purity. Continuous processes would reduce ethyl esters production costs.

**Proof-of-concept of Two-stage Countercurrent Enzyme-assisted Aqueous Extraction Processing of Soybeans.** Juliana Maria Leite Nobrega de Moura, Devin Maurer, Stephanie Jung, Lawrence A. Johnson, Iowa State University, Ames, Iowa, USA

Proof-of-concept of integrated continuous, two-stage, countercurrent, enzyme-assisted aqueous extraction processing of soybeans was demonstrated at pilot-plant scale (75 kg soybeans) over an eleven-day period with recycling of the enzyme used in the cream demulsification into the extraction step. Oil, protein, and solids extraction yields of  $98.0 \pm 0.5$ ,  $96.5 \pm 0.4$ , and  $86.8 \pm 0.5\%$ , respectively, were achieved by the integrated process. The use of a continuous three-phase centrifuge to separate solids achieved a concentrated cream fraction thereby reducing the amount of enzyme used for demulsifying the cream and recycling to extraction stages. Reduced enzyme use reduced the degree of hydrolysis (DH) when moving from laboratory to pilot-plant scale ( $7.2 \pm 1.2$  and  $16.4 \pm 2.0$  vs.  $8.8 \pm 2.2$  and  $10.7 \pm 3.0$ , for 1st and 2nd extraction stages, respectively). Enzymatic cream demulsification yield of 91.6% and free oil recovery of 93.0% were achieved when integrating extraction and cream demulsification. Approximately 6% of cream oil was lost during centrifugation and decantation procedures. About 79% overall free oil recovery relative to the initial amount of oil present in the extruded flakes was achieved, the remainder being unrecovered oil in the skim fraction (19%) and unextracted oil in the insolubles (2%).

**Biodiesel and Value-added Glycerol Carbonate from Supercritical Dimethyl Carbonate.** Zul Ilham, Shiro Saka, Department of Socio-environmental Energy Science, Graduate School of Energy Science, Kyoto University, Kyoto, Japan

Biodiesel has been successfully produced from triglycerides and dimethyl carbonate, utilizing non-catalytic supercritical dimethyl carbonate process in one-step and two-step methods. In this study, it was demonstrated that, the supercritical dimethyl carbonate process successfully converted triglycerides to fatty acid methyl esters (FAME) with glycerol carbonate and citramalic acid as by-products, while free fatty acids were converted to FAME with glyoxal.