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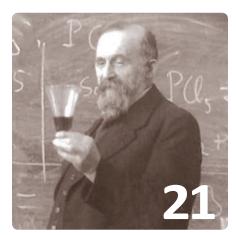




6 Science snapshots from Orlando

Did you miss the 2017 AOCS Annual Meeting and Industry Showcases? Five easy snapshots will bring you up to speed.







- **1 4** AOCS 2017 award recipients
- Georges Chavanne and the first biodiesel

 The first patent for modern biodiesel was issued 80 years ago, yet a peek into history reveals that much of current biodiesel research is uncannily similar to the technical investigations of that first groundbreaking patent.
- A rapid fingerprinting method for sterols in oils and sterol-rich foods

 The main phytosterols in intact oils and sterols-rich foods can be determined in
 a single analysis that eliminates several sample preparation steps and achieves
 results in less than a minute.
- Sheep milk: an unexplored food matrix to develop functional foods
 The chemical composition and nutritional benefits of sheep milk are compared with those of cow and goat milk.

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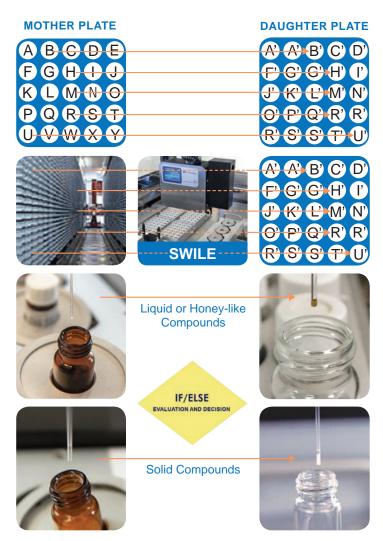
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- The 2017 AOCS Annual Meeting and Industry Showcases, held in Orlando, Florida, USA, on April 30— May 3, offered many interesting and informative oral presentations.
- Those who attended could choose from 492 talks given during 68 technical sessions.
- Although complete coverage is not possible, this article highlights several talks that exemplify the high-quality science presented at the meeting.

Orlando, Florida, offers warm temperatures, beautiful scenery, and Mickey Mouse. On April 30 through May 3, 2017, the city also played host to the 2017 AOCS Annual Meeting and Industry Showcases. The 1,389 attendees found the science discussed at the meeting only slightly less thrilling than the rides at Orlando's famous theme parks. This article provides snapshots of some of the interesting and informative oral presentations given during the meeting.

"ARE ALL FATTY ACIDS CREATED EQUAL?"

Presented by David W. L. Ma, University of Guelph, Canada Health and Nutrition Division

The ratio of n-6 polyunsaturated fatty acids (PUFAs) to n-3 PUFAs in the human diet has risen dramatically over the past century with increasing consumption of vegetable oils. A common perception is that n-6 PUFAs, such as linoleic acid and arachidonic acid, promote inflammation and therefore contribute to conditions such as cardiovascular disease, arthritis, and cancer. On the other hand, n-3 PUFAs, such as α -linolenic acid (ALA), docosahexaenoic acid (DHA), and eicosapentaenoic acid (EPA), are widely considered anti-inflammatory and beneficial to human health. The problem with this simplistic view, says David Ma, is that the structures and biological properties of fatty acids vary widely, even within the n-6 and n-3 families. Moreover, some studies have indicated beneficial effects for n-6 PUFAs in heart health and brain development.



However, studying the biological effects of a particular fatty acid in vivo has been challenging because each fatty acid can be metabolized within the body to other fatty acids with their own distinct effects. The enzyme Δ -6 desaturase catalyzes the first step in the metabolic conversion of both an n-6 PUFA (linoleic acid) and an n-3 PUFA (ALA) into longer-chain fatty acids, such as arachidonic acid, EPA, and DHA. Researchers have generated a transgenic mouse (D6KO) that lacks this enzyme; thus, the mice are unable to metabolize either linoleic acid or ALA (Stroud, C. K., et al., http://doi. org/10.1194/jlr.M900039-JLR200, 2009).

Ma and his colleagues used the D6KO mouse to study whether the pro- or anti-inflammatory effects of linoleic acid and ALA are dependent upon their conversion to other fatty acids. They fed wild-type or D6KO mice one of four diets (rich in linoleic acid, arachidonic acid, ALA, or EPA/DHA) for nine weeks, then sacrificed the mice and examined inflammatory markers within their tissues (Monk, J. M., et al., http://doi. org/10.1016/j.jnutbio.2016.01.004, 2016). The researchers found that the conversion of linoleic acid to arachidonic acid was required for the immune system to activate pro-inflammatory cytokines. In other words, arachidonic acid, but not linoleic acid, was associated with inflammation. On the other hand, ALA showed anti-inflammatory effects independent of its conversion to EPA and DHA.

Ma and his colleagues also used the D6KO mouse model to study the effects of four high-fat diets on the development of fatty liver disease (Monteiro, J., et al., http://doi.

org/10.1139/cjpp-2012-0308, 2013). The first diet—the negative control—contained lard (negligible amounts of n-3 and n-6 PUFAs). The other diets contained canola oil (low ALA), flaxseed oil (high ALA), or menhaden oil (positive control; rich in EPA/DHA). The researchers found that the flaxseed-oilrich diet reduced steatosis (abnormal lipid accumulation) and inflammation in the liver compared with the lard diet, independent of the conversion of ALA to EPA/DHA. However, EPA and DHA had even greater effects in preventing steatosis and inflammation in mice fed a high-fat diet.

During his presentation, Ma also shared some unpublished data from studies examining whether plant- (ALA) or fish- (EPA/DHA) based n-3 PUFAs are more effective for breast cancer prevention in mice. These studies used a transgenic mouse model of breast cancer (MMTV-neu), either by itself or crossed with the D6KO model (MMTV-neu x D6KO). The team found that flaxseed oil reduced tumor size and number in a dose-dependent manner, with a high-ALA diet having similar anti-tumor effects as an EPA/DHA-containing diet.

"THE EFFECTS OF OILSEED PROCESSING ON BIOACTIVE COMPOUNDS IN EDIBLE **CANOLA OIL: A CASE STUDY INVOLVING AUSTRALIAN PROCESSING PLANTS"**

Presented by Clare L. Flakelar, Charles Sturt University, Wagga Wagga, Australia **Processing Division Student Award Winner**

Crude canola oil contains 94–98% triacylglycerols, less than 2.5% phospholipids, 0.4–1.2% free fatty acids, and other minor components. These minor components include both undesirable compounds, such as chlorophyll and trace metals, and beneficial ones, such as phytosterols, tocopherols, and carotenoids. A challenge during canola oil processing is to remove the undesirable compounds while retaining the beneficial ones. Graduate student Clare Flakelar wondered whether recent changes in edible oil processing techniques have affected the retention of minor bioactive compounds in canola oil. So she and her colleagues measured the concentrations of phytosterols, tocopherols, and carotenoids in canola oil samples obtained from various stages of processing in five commercial plants in Australia.

These minor components of canola oil are thought to be beneficial to human health. Phytosterols, present at 7,000-10,000 mg/kg of crude canola oil, have been shown to lower LDL cholesterol and may reduce cardiovascular disease risk. Tocopherols, also known as vitamin E, are present at 700– 1,200 mg/kg in crude canola oil. As natural antioxidants, tocopherols may reduce the risk of cancer, cardiovascular disease, and other ailments. Finally, carotenoids, such as lutein and β-carotene, occur at levels of 50–150 mg/kg in crude canola oil. Several carotenoids have vitamin A activity and contribute to eye and skin health.

Previous studies have shown that edible oil processing in particular, the neutralization, bleaching, and deodorization steps—degrades or removes bioactive compounds. But

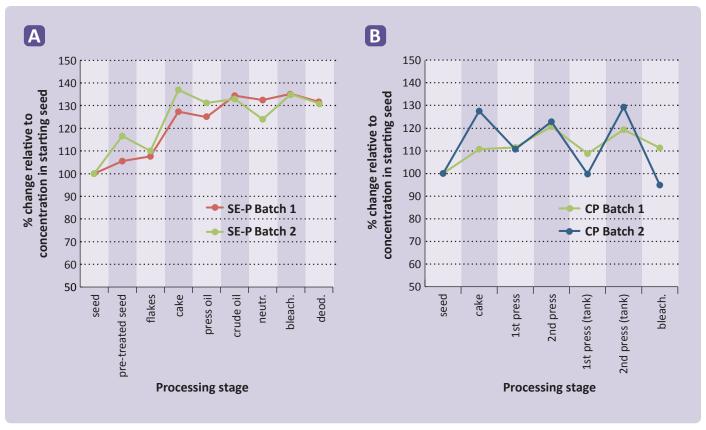


FIG. 1. Changes in tocopherol content with processing stage in canola oil that was (A) solvent extracted and physically refined (SE-P), or (B) cold pressed followed by the bleaching stage only (CP). Credit: Clare Flakelar

with a recent shift toward milder processing conditions, the retention of bioactive compounds may be enhanced compared with traditional refining methods. To investigate this possibility, Flakelar and her colleagues examined canola oil samples from five Australian commercial plants that use different refining techniques: 1) solvent extraction followed by physical refining (SE-P), 2) expeller press extraction followed by chemical refining (E-C), 3) expeller extraction followed by physical refining (E-P), 4) cold pressing followed by the bleaching stage only (CP), and 5) cold pressing followed by physical refining (CP-P).

The researchers developed a rapid method to simultaneously quantify phytosterols, tocopherols, and carotenoids in canola oil using normal-phase high-performance liquid chromatography (Flakelar, C. L., et al., http://doi.org/10.1016/j.foodchem.2016.07.059, 2017). Using this method, they measured the levels of three phytosterols (β -sitosterol, campesterol, brassicasterol), two tocopherols (α - and γ -tocopherol), and two carotenoids (β -carotene and lutein) in the canola oil samples.

The enrichment of phytosterols in the oil samples fluctuated with each step in the refining process, but the retention of the compounds was high in the finished oils for all processes. The SE-P samples showed higher levels of phytosterols in the finished oil than the other samples. The cold-pressed oils showed the lowest amounts of phytosterols in the finished oils, perhaps because the compounds were not efficiently extracted from the seeds by the cold pressing. The choice of

physical or chemical refining did not appear to substantially affect phytosterol content in the expeller-pressed oils (E-P and E-C), which had phytosterol levels in between those of the solvent-extracted and cold-pressed oils.

Similar results were obtained for the tocopherols (Fig. 1), with the SE-P oils showing the greatest enrichment in tocopherols relative to the starting seed, and the CP oils the lowest. In all samples, the proportion of $\alpha\text{-tocopherol}$ slightly decreased, and the proportion of $\gamma\text{-tocopherol}$ slightly increased, in the finished oils compared with the starting seeds. Under all processing conditions, the level of carotenoids dropped to undetectable after the bleaching step, which was expected because bleaching intentionally removes pigments such as carotenoids. Investigations into new processing methods that retain carotenoids, or extract and purify them as valuable byproducts, are warranted, says Flakelar.

This study indicates a high retention of phytosterols and tocopherols in finished oils for all processes, in contrast to previous studies that illustrated dramatic losses during refining. This result could indicate an improved efficiency of refining processes. The study also highlighted differences in the retention of minor components for different pressing techniques. For the CP samples, minor components were increased after a second pressing, but they still remained below the levels of the solvent-extracted samples. These results may guide future efforts to improve the retention of bioactive minor components in edible oils.

"SILICONE SURFACTANTS IN **OIL-BASED SYSTEMS**"

Presented by Tony O'Lenick, Siltech, LLC, USA **Surfactants and Detergents Division** JSD 20th Volume Celebration Honoring Milton Rosen

With his book Surfactants and Interfacial Phenomena, now in its fourth edition, Professor Milton Rosen provided a roadmap not only for the characterization and use of conventional surfactants, but also for entirely new classes of surfactant systems. Many of these concepts can be applied to the study of silicone/hydrocarbon surfactants in oil-based systems, says Tony O'Lenick. These non-traditional surfactants can impart a "slippery" feel and silicone-like spread to products ranging from sunscreen to motor oil, and are often more efficient at reducing the surface tension of oils than traditional surfactants.

When mixed together, silicone fluid and oil are immiscible, similar to water and oil. Therefore, surfactants that contain silicone and an alkyl tail should lower the surface tension of oil similar to conventional surfactants that contain a polar head group and a nonpolar tail. Applying Dr. Rosen's principles, it should be possible to characterize parameters of silicone surfactants such as surface tension lowering, critical micelle concentration (CMC), solubility, emulsification, wetting, and foaming.

Alkyl dimethicones, or silicone waxes, make up one class of silicone surfactants. These molecules consist of an alkyl chain attached to a siloxane backbone. If the length of the alkyl chain is 18 carbons or greater, the silicone wax is solid at room temperature. Silicone waxes with shorter alklyl chains, such as cetyl dimethicone, with 16 carbons in the chain, are liquid at room temperature. Cetyl dimethicone works as a surfactant in soybean oil, lowering the surface tension, improving the spread, and imparting a "silicone-like" feel. Like traditional surfactants, the CMC of cetyl dimethicone can be calculated by plotting the surface tension of the oil versus the weight percent of added surfactant. However, not all silicone surfactants have a readily identifiable CMC. Another measure of effective concentration is the "fixed surface tension." This value refers to the concentration of silicone surfactant added to reduce the surface tension of the oil to 25 dynes/cm.

Another parameter that can be defined for silicone surfactants is the critical gel concentration (CGC), or the minimum concentration needed to cause a system to gel.

C₂₆ dimethicone is a silicone wax with a 26-carbon alkyl chain, making it a solid at room temperature. At its CGC, C₂₆ dimethicone transforms liquid soybean oil into a gel. When added to olive oil at concentrations of 20–80%, C₂₆ dimethicone forms gels of varying firmness and opacity.

Silicone polymer surfactants can act as foaming agents in a triglyceride solution (C8-10). When an air bubble is introduced into the solution, the silicone polymer surfactant reduces the surface tension around the bubble, allowing it to expand rapidly. In addition, silicone polymers entangle with themselves in the liquid border between air bubbles, slowing foam drainage.

O'Lenick and his colleagues have also studied multi-domain, or gemini, silicone surfactants. Multi-domain alkyl dimethicones contain two different alkyl chains on the same siloxane backbone: a short, liquid carbon chain; and a longer, solid carbon chain (Fig. 2A). These multi-domain silicone surfactants can self-assemble into organized networks that have different properties from mixtures of the corresponding single-domain polymers (Fig. 2B). The multi-domain polymers can form gels that are two-phase liquid crystal systems. These gels have lower melting points, are more translucent, and flow more under pressure compared with a gel made from blending the two single-domain polymers. Thus, the multi-domain silicone surfactants can drastically alter rheology, aesthetics, and other properties of gels.

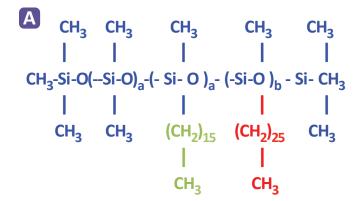




FIG. 2. A. Chemical structure of a multi-domain alkyl dimethicone surfactant. Shown in green is the short, liquid carbon chain; in red is the longer, solid carbon chain. B. The multi-domain surfactant self-assembles into organized networks (phase-contrast microscope image and gel at left) that are different from those of a polymer blend (right). Credit: Tony O'Lenick

O'Lenick concluded his presentation with the following statement: "Professor Rosen, those of us who were not given the opportunity to be your student; those of us not given the opportunity to listen to your lectures; you have touched our lives and research by your leadership, your roadmap, and most importantly your mentorship. And for that we thank you."

"SYNTHETIC BIOLOGY TO ENGINEER NOVEL OILS WITH ENHANCED PROPERTIES"

Presented by Timothy Durrett, Kansas State University, USA Biotechnology Division

Acetyl-triacylglycerols (acetyl-TAGs) are TAGs that contain an acetyl group in place of the *sn-3* fatty acyl group. The unusual structure of acetyl-TAGs confers unique properties that could be useful in biofuels, lubricants, and food emulsifiers. The seeds of the burning bush plant (*Euonymus alatus*) contain about 50% oil by weight, of which more than 95% is acetyl-TAGs (Fig. 3). By introducing a gene from *E. alatus* to the oilseed crop *Camelina sativa*, Timothy Durrett and colleagues produced a transgenic Camelina line that accumulates high levels of acetyl-TAGs in its seeds.

Compared with other vegetable oils, acetyl-TAGs have unique properties. For example, the viscosity of acetyl-TAG oil is about 40% lower than that of soybean oil, which could allow the direct injection of acetyl-TAGs into some types of diesel engine. In contrast, other vegetable oils must be transesterified prior to their use as biodiesel. Acetyl-TAGs also have improved cold-temperature properties compared with regular vegetable oils. Unlike soybean oil, acetyl-TAGs remain liquid at -20°C. In addition to possible use as a biofuel, acetyl-TAGs may find applications as lubricants, plasticizers, and food additives (emulsifiers and coatings). Because acetyl-TAGs have an acetyl group in place of a long fatty acid chain, they have about 6.3% fewer calories than regular TAGs.

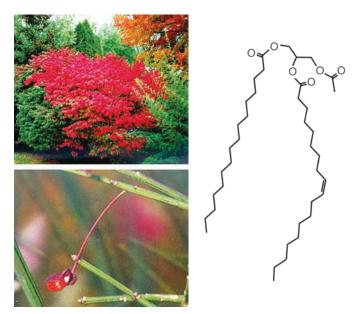


FIG. 3. (Left) The burning bush plant (Euonymus alatus) and seed. (Right) Chemical structure of an acetyl-TAG. Credit: Timothy Durrett

Using a deep transcript profiling approach, Durrett and his colleagues identified the gene in the burning bush plant that is responsible for the high levels of acetyl-TAGs in the plant's seeds. The gene encodes an enzyme, *Euonymus alatus* diacylglycerol acetyltransferase (*EaDAcT*), that uses acetyl-CoA to acetylate diacylglycerol. Next, the researchers introduced the gene encoding *EaDAcT* to *Camelina sativa*, an oilseed crop that requires minimal irrigation and fertilizer, and has a short life cycle. Unlike the burning bush plant, Camelina could cost-effectively produce large quantities of acetyl-TAG oil.

The researchers found that the transgenic Camelina seeds produced oil with about 50% acetyl-TAGs. They were able to further boost acetyl-TAG production in Camelina by suppressing DGAT1, the enzyme responsible for regular TAG synthesis, using RNAi. Seeds that expressed both *Ea*DAcT and DGAT1-RNAi accumulated up to 85% acetyl-TAGs in their oil (Liu, *et al.*, http://doi.org/10.1111/pbi.12325, 2015). Durrett and his colleagues also sequenced the transcriptomes of other acetyl-TAG-producing plants and identified some new enzymes with even higher acetyltransferase activities than *Ea*DAcT. When the researchers introduced a gene encoding one of these enzymes to Camelina, in combination with DGA1-RNAi, the resulting seed oil contained 90% acetyl-TAGs. Importantly, the genetically modified seeds appeared to germinate and grow normally.

To expand the functional repertoire of acetyl-TAG oils, Durrett and his colleagues are now engineering acetyl-TAGs with unusual fatty acids. For example, acetyl-TAGs with medium-chain fatty acids (MCFAs) at the *sn-1/sn-2* positions show further reductions in viscosity compared with the long-chain versions, and acetyl-TAGs with ricinoleic acid polymerize differently than their natural acetyl-TAG counterparts. However, the *in vivo* efficiency of MCFA incorporation into acetyl-TAGs is currently low, and Camelina seeds expressing acetyl-TAGs with ricinoleic acid fail to germinate, indicating that more work is needed in these areas.

"STRUCTURED TRIGLYCERIDES IN INFANT FORMULA: DEVELOPMENT OF FAT BLENDS WITH NUMEROUS BENEFITS"

Presented by Eric L. Lien, University of Illinois, USA Health and Nutrition Division

In the first three to four months after birth, an infant's body weight doubles, fueled by an energy intake that is about four times higher per kilogram per day than that of adults. Breast milk is rich in lipids, and the ability to digest fats is relatively well developed in young infants. Human milk provides superior nutrition for infants, but, some infants cannot be breastfed for various reasons. Therefore, suitable replacements for human milk are needed. In his presentation, Eric Lien summarized what has been learned about the lipid composition of human milk over the past century, how formulators have incorporated this knowledge, and new directions for further improving infant formula so that it more closely mimics breast milk.

Most infant formulas contain the cow's milk proteins whey and casein, a blend of vegetable oils as a fat source, lactose as

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a carbohydrate source, vitamins, and minerals. In 1965, British dietitian Elsie Widdowson evaluated a new infant formula that attempted to "humanize" the fat blend (Lancet 2, 1099–1105). The saturated fatty acid profile of the new formula was very similar to that of human milk. Widdowson studied the absorption of fat, protein, and minerals by infants consuming the formula at 5–7 days and 4–6 weeks of age. She found that the formula-fed infants absorbed less fat than the breastfed infants at 5-7 days, but by 4-6 weeks, fat absorption was comparable in the two groups. Surprisingly, calcium retention was drastically reduced (by about 9-fold) in the formula-fed infants compared with the breastfed infants at 5–7 days. The difference in calcium retention was much less pronounced at 4-6 weeks.

In 1968, Tomarelli and colleagues recognized that although the saturated fatty acid profiles of the new formula and human milk were similar, the positioning of palmitic acid (16:0) within triglycerides was quite different, and that this positioning had important metabolic consequences (J. Nutr. 95, 583-590). In human milk, about 70% of the palmitic acid is in the sn-2 position of triglycerides. In contrast, in vegetable oils, which supply the fat blends in infant formulas, most of the palmitic acid is in the sn-1 or sn-3 position. During digestion, pancreatic lipase clips off fatty acids from the sn-1 and sn-3 positions of the triglyceride, releasing two free fatty acids and one sn-2 monoglyceride into the intestinal lumen. Palmitic acid as an sn-2 monoglyceride is much more efficiently absorbed



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than free palmitic acid, which forms complexes with minerals such as calcium and is excreted in the feces as fatty acid soaps. This finding explained both the reduced fat absorption and the reduced calcium retention observed in formula-fed infants at 5–7 days compared with breastfed infants.

Much later, in 1995, Quinlan *et al.* examined fatty acid soaps in the stools of breastfed and formula-fed infants (*J. Pediatr. Gastroenterol. Nutr. 20*, 81–90). The researchers found that soap fatty acids comprised about 6% of the wet weight of stools from formula-fed infants, compared with only about 0.5% of stools from breastfed infants. In addition, the formula-fed infants excreted about four times more calcium in their stools than breastfed infants. The formula-fed infants also had harder stools than the breastfed infants.

In the late 1990s, structured triglycerides with palmitic acid at the *sn*-2 position were synthesized from vegetable oils using an enzymatic process. About 50% of the palmitic acid in these triglycerides is in the *sn*-2 position. In 1999, Kennedy *et al.* assessed the new lipid blend by comparing three groups of healthy newborn infants: control formula-fed, high-*sn*-2 formula-fed, and breastfed (*Am. J. Clin. Nutr. 70*, 920–927). After 12 weeks of feeding, stool characteristics and bone mineral density (as an indicator of calcium retention) were determined. The researchers found that the level of palmitic acid soaps in the stools of the high-*sn*-2 formula-fed infants was in between that of the control formula-fed and breastfed infants. The bone mineral density and stool consistency of the high-*sn*-2 formula-fed infants also more closely resembled those of the breastfed infants.

In the final part of his talk, Lien discussed some new approaches to improving the composition of infant formula, such as adding the prebiotic oligofructose to high-sn-2 formula. Oligofructose serves as a readily digestible carbohydrate source for some potentially beneficial colonic bacteria, such as bifidobacteria. These beneficial bacteria tend to be higher in the colon and feces of infants fed breast milk than those fed standard (non-high-sn-2) formula. In 2014, Lien and his coworkers compared the stool consistency and composition of healthy newborn infants fed either control formula, high-sn-2 formula with or without oligofructose, or human milk (Yao, et al., http://doi.org/10.1097/MPG.0000000000000443).

The researchers found that the addition of oligofructose to the high-sn-2 formula led to softer stools in the infants, but the amounts of fatty acid soaps and calcium in the infants' stools were not significantly different from the group consuming high-sn-2 formula without oligofructose. Unexpectedly, the researchers uncovered an apparent prebiotic effect of the sn-2 formula itself: Infants fed high-sn-2 formula with or without oligofructose showed comparable levels of stool bifidobacteria as those fed breast milk. In contrast, the control formula-fed infants had significantly lower levels of bifidobacteria in their stools. The growth of bifidobacteria may be promoted by sn-2-palmitate, or inhibited by palmitate-calcium soaps, the researchers suggest.

Laura Cassiday is an associate editor of Inform at AOCS. She can be contacted at laura.cassiday@aocs.org.









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AOCS 2017 award recipients

AOCS celebrates ingenuity and collaboration by honoring those individuals and teams who have taken the industry to the next level, who have advanced the quality and depth of the profession, and who have leveraged their knowledge for the benefit of the society.

These individuals from around the world were recognized during the 108th AOCS Annual Meeting and Industry Showcases held April 30–May 3. **Award nominations for 2018 are due November 1, 2017. To learn more, visit aocs.org/awards.**

SOCIETY AWARDS

A. R. BALDWIN DISTINGUISHED SERVICE AWARD

Recognizes: An active or previously active member of the Society making outstanding contributions and service to the Society over a substantial period of time.



A quick look at his membership record shows one reason why AOCS Fellow **STEVEN E. HILL**, T. Marzetti's, is the recipient of the 2017 A. R. Baldwin Distinguished Service Award. Actually, it is difficult to engineer a quick look, given the lengthy listing of his involvement with AOCS through myriad committees and service on various iterations of the

Governing Board.

Dr. Hill joined AOCS in 1987 as a student and received the Honored Student Award in 1991. He remembered those early days in his acceptance speech as the 2014–2015 AOCS President: "My first annual meeting was in 1989. I was 25 years old [and] in my third year of graduate school. ... As a student member, I was welcomed into AOCS and the area of science that I was studying; this experience at my first meeting was repeated many times."

Clearly, Dr. Hill's initial experience has informed his involvement in the Society. He has given unstintingly of his time to mentor students, to organize and teach short courses, to develop annual meeting programming, and to participate in and organize Division events. Further, he provided leadership for Sections and the AOCS Governing Board as well as the AOCS Foundation. In addition, he has served on many award committees, including as a Trustee of the Stephen S. Chang Award, as well as on the Books & Special Publications Committee, the Inform Editorial Advisory Committee, the Membership Development Committee, the Audit Committee, the Business Management Committee, and the Nominating and Election Committee.

It is no exaggeration to say that AOCS owes its very existence to his work, with others, during the financial downturn experienced by the Society in 2001. Dr. Hill was among the Governing Board and staff members who developed a new business model for the Society, which led to a budget surplus and financial stability immediately following implementation of the plan.

Dr. Hill's decades-long investment of time, talent, and energy in AOCS constitutes the very definition of the A.R. Baldwin Distinguished Service Award. In words taken from a letter of nomination, "Steven is not just a stalwart of the AOCS, he is our ambassador and champion of the AOCS mission."

FELLOW AWARD

Recognizes: Achievements in science or extraordinary service to the Society.



DILIP K. NAKHASI, Senior Director, Research, Development and Innovation, for Stratas Foods, LLC, has a distinguished record of scientific achievement in industry as well as service to AOCS.

As a Director of Innovation for Bunge North America, Inc., he and his team developed and introduced PhytoBake. This functional shortening received a 2010

IFT Innovation Award for its delivery of nutritional benefits through the use of phytosterol esters. His team also developed and introduced Delta P/RB, which is a structured lipid that employs medium-chain triglycerides to provide nutritional benefits to children with gastroenterological problems. Yet another development led to Saturate Sparing Technology, which created a shortening system using the nonlipid component to reduce saturated fat content.

Nakhasi has been named on 10 US and numerous international patents and has published in journals ranging from *Nutrition, Metabolism & Cardiovascular Diseases* to the *Journal of Food Lipids*.

He has been an AOCS member for 22 years, serving with distinction in a variety of capacities, including as both chair and vice-chair of the Edible Applications Technology Division. Nakhasi has also organized and presented at a number of short courses and served as a member of the Annual Meeting Administrative Committee. Further, he served as chair of the Program Committee from 2010-2016. In recognition of his service, he received the AOCS Award of Merit in 2016.



NISSIM GARTI, Professor of Applied Chemistry, The Hebrew University of Jerusalem, is considered by many to be the leading international expert on the theory and practice of fat crystallization, emulsion, microemulsion, and encapsulation technologies.

Garti is the author or co-author of more than 400 peer-reviewed articles;

the author, editor, or contributor to more than 70 books and special publications; and has been awarded more than 100 patents. He has conducted fundamental research that has potential for application in many different fields, from the delivery of pharmaceuticals and nutraceuticals to the stabilization of triglyceride polymorphs.

In addition to his academic pursuits, Garti has founded a number of startup companies in Israel, including LDS, NutraLease, Adumim Chemicals, and Memphile Technnologies. He received the AOCS Corporate Achievement Award in 2011 for his research on the development of novel nano-sized self-assembled lipid carriers as delivery vehicles for improved solubilization and bioavailability. He also received the Supelco/ Nicholas Pelick-AOCS Research Award in 2013 and the AOCS Stephen S. Chang Award in 2009, as well as a long list of awards from other organizations.

As a long-time AOCS member, Garti has been active at AOCS meetings, organizing and presenting at annual meeting sessions as well as conferences devoted to the physical properties of lipids. He also served as an associate editor of the Journal of the American Oil Chemists' Society from 2008–2012.

YOUNG SCIENTIST RESEARCH AWARD

Recognizes: A young scientist that has made a significant and substantial research contributions in one of the areas represented by an AOCS Division.



LAURA NYSTRÖM, Associate Professor of Food Biochemistry, ETH Zürich, Switzerland, graduated from the University of Helsinki (UH, Finland) in 2002, finishing her doctoral studies there in 2008 in food chemistry, and continuing as a postdoctoral researcher in the Cereal Technology group at UH. After working from 2009-2016 as a tenure track assis-

tant professor of food biochemistry at ETH Zürich, Switzerland,

she was promoted to her current position as associate professor of Food Biochemistry at the same university.

Nyström has worked as a visiting scientist at the United States Department of Agriculture/Agricultural Research Service Eastern Regional Research Center (Wyndmoor, Pennsylvania, USA); the University of Nebraska-Lincoln (USA); and the University of Copenhagen (Denmark). Her research focuses on dietary fibers in cereal grains and associated minor phytochemicals, radical mediated degradation of polysaccharides, lipid oxidation and antioxidants, and enzymatic lipid modification. The two main thrusts of her program center on the stability and molecular interactions of dietary fibers, and the identity and bioactivities of sterol conjugates.

"A key facet of our research strategy," she writes, "is to integrate cutting-edge technologies that provide a greater detail and depth of understanding of the topics. Our long-term goal is to identify and optimize tailored food ingredients for optimized nutrition and technological functionality."

Nyström has published more than 40 original publications, two book chapters, and has participated in over 50 international conferences. She received the Euro Fed Lipid Young Lipid Scientist Award in 2012, the Young Scientist Research Award of the AACC International in 2015, and a Starting Grant from the European Research Council in 2015. She has been an AOCS member since 2011.

SCIENTIFIC AWARDS

MILLIPORESIGMA/NICHOLAS PELICK -**AOCS RESEARCH AWARD**

Award: Plague, \$10,000 honorarium, and \$1,500 travel stipend.

Sponsored by: MilliporeSigma and Nicholas Pelick, a longtime member and Past President of AOCS.

Recognizes: Outstanding original research in fats, oils, lipid chemistry, or biochemistry.



FEREIDOON SHAHIDI, University Research Professor in Biochemistry, Memorial University of Newfoundland, Canada, is an internationally recognized scientist in the area of nutraceuticals and functional foods, particularly in food lipids and natural antioxidants. His research has concerned both basic and applied areas of lipid science and technology. Further, he

has been listed among the most highly cited scientists in the areas of food, nutrition, and agricultural sciences.

Shahidi's first contribution to the understanding of fats and oils was in formulating nitrite-free meat-curing systems. He found that the pigment responsible for the color of cured meats was a mononitrosyl ferrohemochrome rather than a dinitrosyl compound as was originally thought. He further confirmed that this pigment had its own antioxidant potential and was able, together with other cure adjuncts, to render similar

stability to treated meats as those observed for nitrite-cured products.

For almost 30 years, he has concentrated on the role of omega-3 fatty acids and marine oils in combatting degenerative diseases. His recent findings have revealed that chemically binding highly unsaturated fatty acids found in marine and algal oils with epigallocatechin gallate (the main catechin in green tea) can fully arrest colon cancer in a mouse model and to reverse tumor growth in human lung cancer.

Shahidi has published more than 750 research articles in peer-reviewed journals, as well as book chapters, and has edited or written 64 books, including serving as editor-in-chief of all six volumes of the 6th edition of *Bailey's Industrial Oil and Fat Products* (2005) and is now preparing the 7th Edition of this set in 7 volumes. He is an active member of a number of professional societies, including AOCS, the American Chemical Society, the Institute of Food Technologists, the Royal Society of Chemistry, the International Union of Food Science and Technology, and the International Society for Nutraceuticals and Functional Foods—which he founded.

His involvement in AOCS is lengthy and wide-ranging. Named as an AOCS Fellow in 2008, Shahidi has also served as chair of both the Lipid Oxidation and Quality and Protein and Co-products Divisions. He received the AOCS Stephen S. Chang and Alton E. Bailey Awards in 2014 and has been an AOCS member for 25 years.

STEPHEN S. CHANG AWARD

Award: Jade sculpture and \$1,500 honorarium. Endowed by: The late Stephen S. Chang and his wife, Lucy D. Chang.

Recognizes: A scientist or technologist who has made decisive accomplishments in research for the improvement or development of products related to lipids.



MOGHIS U. AHMAD, Vice President, Jina Pharmaceuticals Inc., Illinois, USA, has conducted both basic and applied research, and has discovered a variety of new lipid products for chemical, pharmaceutical and biotechnological applications. He has contributed to the field of lipid chemistry in numerous ways, including through the search for new industrial

oils, the chemical and enzymatic synthesis of lipid products, the synthesis of dietary cis and trans fatty acids, process research, and large-scale synthesis of lipid products for industrial applications.

He is a founding member of Jina Pharmaceuticals Inc., which was established in 2006. Under his direction, Jina developed the Nanoaqualip® Technology to administer poorly soluble therapeutic drugs for various treatments including cancer. Using this novel technology, several lipid-based formulations were developed in complete aqueous systems free from toxic organic solvents. In addition, he has contributed to the synthesis and applications of a new class of lipid molecules for the development of nanosomal or liposomal drug-deliv-

ery systems, such as carbohydrate—lipid conjugates for drug targeting.

Dr. Ahmad's innovative career in the pharmaceutical industry was initiated with the development of synthetic cardiolipin (a complex phospholipid) through novel synthetic procedures, followed by the application of synthetic cardiolipin in liposomal drug delivery. He was the first to develop a novel cationic cardiolipin and analogues that have proven to be less toxic than commercially available cationic lipids. This research led to the first cationic cardiolipin-based transfection reagents, marketed by NeoPharm, Inc.

His research is detailed at length in 60 research publications in peer-reviewed journals and book chapters, and more than 30 patents and patent applications. His leadership in AOCS—which he joined in 1970—includes serving as an officer in the AOCS Phospholipids Division and editing several AOCS Press books. Titles include *Lipids in Nanotechnology* and *Polar Lipids: Biology Chemistry, and Technology*. Ahmad is currently editing the upcoming book *Fatty Acids: Chemistry, Synthesis, and Applications*. He is an elected officer of the Lecithin and Phospholipid Society and is currently vice president of that society. Ahmad is an elected fellow of both the Royal Society of Chemistry (2011) and AOCS (2014), and the recipient of the AOCS Alton E. Bailey Award (2016).

DIVISION AWARDS

ANALYTICAL DIVISION: HERBERT J. DUTTON AWARD

\$1,000 honorarium, \$1,000 travel stipend, and a plaque

The award is presented for significant contribution to the analysis of fats and oils or to improvement in the understanding of the processes used in the fats and oils industries. The award is named for Dr. Dutton, a long-time research leader at the US Department of Agriculture facility in Peoria, Illinois, USA.

N. A. MICHAEL ESKIN, University of Manitoba, Canada

Student Award
KATRIN MATHEIS, Technical University of Munich, Germany
SARAH MAYFIELD, University of Arkansas, USA







Eskin

Matheis

Mayfield

BIOTECHNOLOGY DIVISION

Student Award

First place: JINGBO LI, Aarhus University, Denmark
Second place: HEE JIN KIM, Korea University, Republic of Korea
Third place: SARAH WILLETT, University of Georgia, USA













Kim Willett Reeves Ramel Lichtenstein

EDIBLE APPLICATIONS TECHNOLOGY DIVISION: TIMOTHY L. MOUNTS AWARD

\$750 honorarium and a plaque

The award is for either basic or applied research accomplishments relating to the science, technology, or application of edible oils in food products. It memorializes the former AOCS president, who was a distinguished research scientist with the US Department of Agriculture. The award is sponsored by Bunge North America.



Toro-Vazquez

JORGE TORO-VAZQUEZ, Universidad Autónoma de San Luis Potosí, Mexico

Outstanding Achievement Award ROBERT REEVES, Retired, USA

Student Award PERE RAMEL, University of Guelph, Canada

HEALTH AND NUTRITION DIVISION: RALPH HOLMAN LIFETIME ACHIEVEMENT AWARD

\$500 honorarium, \$1,000 travel stipend, and an orchid print

The award recognizes outstanding performance and meritorious contributions to the health and nutrition interest area. The award is named after Ralph Holman in recognition of his lifetime service to the study of essential fatty acids.

ALICE LICHTENSTEIN, Tufts University, USA













Taha

Rogers

Len

Acosta

Hill

Goel

Boakye



Boyer

SURFACTANTS AND DETERGENTS DISTINGUISHED SERVICE AWARD

\$2,000 honorarium and a plaque

The award recognizes a significant advance in, or application of, the principles of surfactant chemistry by a chemist working in the industry. The award is sponsored by Milton Rosen in honor of his father, Samuel, who worked as an industrial chemist on the formulation of printing inks for more than four decades.

EDGAR ACOSTA, University of Toronto, Canada

NEW INVESTIGATOR RESEARCH AWARD

AMEER Y. TAHA, University of California, USA

Student Award

AMANDA N. ROGERS, Chapman University, USA

SAMUEL ROSEN MEMORIAL AWARD

RANDAL HILL, Flotek Chemistry, USA

Student Award

SACHIN GOEL, University of Toronto, Canada

INDUSTRIAL OIL PRODUCTS DIVISION: ACI/NBB GLYCERINE INNOVATION AWARD

CHRISTOPHE LEN, Université de Technologie de Compiegne, France

Student Award

PRINCE BOAKYE, Delaware State University, USA

POSTER AWARDS

ANALYTICAL DIVISION

Best Overall: Pierluigi Delmonte, US Food and Drug

Administration, USA

First Place Student: Ayse Ece Turan, Ryerson University,

Canada

Second Place Student: Tao Zhang, Jiangnan University, China

DISTINGUISHED SERVICE AWARD

MICHAEL BOYER, AWT, USA

PROCESSING DIVISION:

HEALTH AND NUTRITION DIVISION

Best Overall: Payam Vahmani, Agriculture and Agri-Food Canada, Canada

First Place Student: Nuanyi Liang, University of Alberta, Canada *Second Place Student:* Merritt Drewery, Louisiana State University, USA

Third Place Student: Adriana V. Gaitán, Louisiana State University, USA

Student Award

HENOK D. BELAYNEH, University of Nebraska-Lincoln, USA **CLARE L. FLAKELAR**, Charles Sturt University, Australia **JINGBO LI**, Aarhus University, Denmark

Belayneh





Li

INDUSTRIAL OIL PRODUCTS DIVISION

First Place: Malick Samateh, The City College of New York, USA *Second Place:* Chazley J. Hulett, Montana State University Northern. USA

LIPID OXIDATION AND QUALITY DIVISION

First Place: Thanh P. Vu, University of Massachusetts Amherst, USA

PROTEIN AND CO-PRODUCTS DIVISION

First Place: Nandika Bandara, University of Alberta, Canada

Second Place: Chinonye M. Udechukwu, Dalhousie University,

Canada

Third Place: Hongyi Wu, University of Manitoba, Canada

SURFACTANTS AND DETERGENTS DIVISION

First Place: Tomone Sasayama, Tohoku University, Japan

STUDENT RECOGNITION

MANUCHEHR (MANNY) EIJADI AWARD \$1,000 scholarship and a certificate

The Eijadi Award recognizes outstanding merit and performance by an AOCS Honored Student. The award, established by Mr. Eijadi, is intended to help the recipient finance his or her studies.



Bokhari

SYED AWAIS ALI SHAH BOKHARI, Universiti Teknologi PETRONAS, Malaysia

AOCS FOUNDATION HONORED STUDENT AWARDS

Travel stipend and a certificate

The award recognizes graduate students at any institution of higher learning who are conducting research in any area of science dealing with fats and lipids and who are interested in the areas of science and technology. Supported by contributions from members as well as companies in the industry.

SYED AWAIS ALI SHAH BOKHARI, Universiti Teknologi PETRONAS, Malaysia

SUBIN RAJ CHERI KUNNUMAL RAJENDRAN, Dalhousie University, Canada

JINGBO LI, Aarhus University, Denmark
PERE RAMEL, University of Guelph, Canada
RYAN WEST, Ryerson University, Canada
ZIPEI ZHANG, University of Massachusetts Amherst, USA



Bokhari



Rajendran



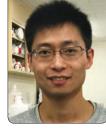
Li



Ramel



West



Zhang

PETER AND CLARE KALUSTIAN AWARD

\$1,000 scholarship and a certificate

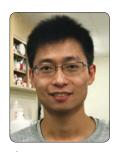
The Kalustian Award recognizes outstanding merit and performance by an AOCS Honored Student. The award is supported by the Kalustian estate.

ZIPEI ZHANG, University of Massachusetts Amherst, USA

RALPH H. POTTS MEMORIAL FELLOWSHIP AWARD

\$2,000 scholarship, travel stipend, and a plaque

The Ralph H. Potts Award is presented annually to a graduate student working in the chemistry of fats and oils and their derivatives. The award is sponsored by AkzoNobel to memorialize Ralph Potts, a pioneer in research on industrial uses of fatty acids.



Zhang



Goel

SACHIN GOEL, University of Toronto, Canada Lecture: S&D 4

THOMAS H. SMOUSE FELLOWSHIP AWARD

\$10,000 scholarship, \$5,000 research funding, and bookends

The Archer Daniels Midland Foundation, the AOCS, the AOCS Foundation, and the family and friends of Dr. Smouse have established and assisted in funding a fellowship program designed to encourage and support outstanding graduate



Nwachukwu

research in a field of study consistent with the areas of interest to the AOCS.

IFEANYI NWACHUKWU, University of Manitoba, Canada

BEST PAPER AWARDS

ACI DISTINGUISHED PAPER

Elucidation of Softening Mechanism in Rinse-Cycle Fabric Softeners. Part 2: Uneven Adsorption—The Key Phenomenon to the Effect of Fabric Softeners (*JSD 19*: 756–773).

Takako Igarashi, Koichi Nakamura, Masato Hoshi, Teruyuki Hara, Hironori Kojima, Masatsugu Itou, Reiko Ikeda, and Yoshimasa Okamoto

ADM AWARD FOR BEST PAPER IN PROTEIN AND CO-PRODUCTS CHEMISTRY/NUTRITION

Conversion of Canola Meal into a High Protein Feed Additive via Solid State Fungal Incubation Process (*JAOCS 93*: 499–507).

Jason R. Croat, Mark Berhow, Bishnu Karki, Kasiviswanathan Muthukumarappan, and William R. Gibbons

ADM AWARD FOR BEST PAPER IN PROTEIN AND CO-PRODUCTS ENGINEERING/TECHNOLOGY

Optimization of Enzymatic Process Condition for Protein Enrichment, Sugar Recovery and Digestibility Improvement of Soy Flour (*JAOCS 93*: 1063–1073)

Abdullah A. Loman and Lu-Kwang Ju

EDWIN N. FRANKEL AWARD FOR BEST PAPER IN LIPID OXIDATION AND QUALITY

Kinetic Analysis of Co-oxidation of Biomembrane Lipids Induced by Water-Soluble Radicals (*JAOCS 93*: 803–811). Atsushi Takahashi, Naomi Shibasaki-Kitakawa, Takao Noda, Yuko Sukegawa, Yuya Kimura, and Toshikuni Yonemoto

PHOSPHOLIPID BEST PAPER AWARD

Chitosan/Lecithin Liposomal Nanovesicles as an Oral Insulin Delivery System (*Pharmaceutical Development and Technology 22*: 390–398)

Mayyas Al-Remawi, Amani Elsayed, Ibrahim Maghrabi, Mohammad Hamaidi, and Nisrein Jaber

ADDITIONAL AWARDS

ALTON E. BAILEY AWARD

\$750 honorarium and a plaque

The award recognizes outstanding research and exceptional service in the field of lipids and associated products. The medal commemorates Alton E. Bailey's great contributions to the field of fats and oils as a researcher, as an author of several standard books in the field, and as a leader in the work of the Society. Archer Daniels Midland Company Inc. sponsor the award.



Marangoni

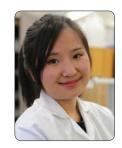
ALEJANDRO MARANGONI, University of Guelph, Canada

HANS KAUNITZ AWARD

\$1,000 honorarium, \$500 travel allowance, and a certificate

The award recognizes the outstanding performance and merit of a graduate student within the United States.

RUOJIE ZHANG, University of Massachusetts Amherst, USA



Zhang



Georges Chavanne Gerhard Knothe and the first biodiesel

- August 31, 2017, marks the 80th anniversary of what was probably the first report/patent on what is considered to be biodiesel today.
- This patent for a "procedure for the manufacture of a heavy fuel," was issued in 1937 to Georges Chavanne, a professor of chemistry at the University of Brussels. Although this kind of fuel would not be investigated again until after the energy crises of the 1970s, the reasons for and the results of the technical investigations from the 1930s that led to Chavanne's patent are very similar to those of current biodiesel research.
- This is the story of the collective effort that inspired the first biodiesel patent, the underlying chemistry of the process, and why the biodiesel Chavanne made still meets the definition of biodiesel today.

In the summer of 1938, a bus ran the commercial bus line between Brussels and Leuven (Louvain) in Belgium powered by a fuel that had probably never been tested before. This fuel consisted of the ethyl esters of palm oil, thus derived from a feedstock that certainly is not native to Belgium. How did this development come about?

On April 1, 1935, a royal decree created a Commission on Fuels ("Commission des Carburants") within the Belgian Department of Colonies [1]. The objective was to systematically study the production and utilization of fuels of local origin in the Belgian Congo, which was Belgium's major African colony. The background, similar to that of other work from these times aimed at using vegetable oils as fuels [2], was to provide a degree of energy independence to the African colony of a European country.

THE "SUPER 8" OF THE COMMISSION ON FUELS

As of early 1940, this commission was chaired by Camille Camus, a Director General in the Ministry of Colonies. The seven other members included the chief of staff (*chef de service*) of the Ministry of Colonies, an engineer for military construction, and The Director General for agriculture in the Ministry of Colonies, Marcel van den Abeele (1898–1980), who wrote the introduction of the detailed 1942 four-chapter publication on this commissioned work from which significant parts of this article are taken (the authors of the chapters are not directly indicated) [1].

Four members of the commission held academic positions, two with backgrounds in organic chemistry, one chemical engineer, and a mechanical engineer. These four academics were Edmond Connerade, a professor of chemistry at the Faculté polytechnique in Mons whose publications include work on hydrogenation as well as fuel-related uses of coal;

Albert Coppens (1885–1966), a professor of mechanical engineering at the University of Leuven (Louvain); Eugene Mertens (1889-1970; later Eugène Mertens de Wilmar), also a professor at the University of Leuven but of chemical engineering and who worked on products from coal and utilization of products from the Congo; and Georges Chavanne (1875–1941), a professor of chemistry at the University of Brussels.

According to the 1942 report by van den Abeele, it was Chavanne who recommended a classical reaction for producing a fuel, namely the alcoholysis of a vegetable oil with a low molecular alcohol, such as methanol or ethanol. The commission then selected the ethanolysis of palm oil. These ethyl esters of palm oil meet (1875-1941) works at the the current definition of biodiesel in the standard ASTM D6751 as the "mono-alkyl esters of longchain fatty acids derived from vegetable oils or animal fats." That Chavanne likely played a major role

in this project is also shown by the first document to arise from it, namely Belgian patent 422877 granted on August 31, 1937, solely in Chavanne's name for a "procedure for the manufacture of a heavy fuel" [3]. It was not until about 1980, as a result of the energy crises of the 1970s, that this kind of fuel would be investigated again.

CHAVANNE'S LIFE AND CAREER

Georges Chavanne [4–7] was born on October 9, 1875, in Hôpitaux-Neufs, a small community in the department of Doubs of the Franche-Comté region in southeastern France close to the Swiss border. He attended school in Bourg-en-Bresse (department of Ain) and Besançon (the capital of Doubs). After his military service, he attended the prestigious *École* normale supérieure from 1896-1899. He enrolled at the Sorbonne University in Paris in 1900, being a student of Louis-J. Simon, with whom he shared numerous publications into the 1920s. Among his teachers were two Nobel laureates—Ferdinand-Frédéric-Henri Moissan (1852–1907; Nobel Prize in Chemistry in 1906 for the isolation of fluorine) and Jean-

Baptiste Perrin (Nobel Prize in Physics 1926 for

study of Brownian motion)—which undoubt-

edly gave him an excellent scientific pedi-

gree. Chavanne received his doctorate in 1904. One year later, the chair for general chemistry at the Free University of Brussels became available. In 1906, upon recommendation of his teacher Henri Moissan and the famous Marcellin Berthelot (1827–1907), Chavanne became a professor at the Free University of Brussels, advancing to a full professorship in 1910.



Georges Chavanne

University of Brussels.

(Despy-Meyer, A., et al., 2001)

The first Solvay Conference on Chemistry was held in Brussels, April 1922. Chavanne is in the back row, far left. Other luminaries in this photo include several Nobel laureates: Jean-Baptiste Perrin (middle row, fifth from left; Physics 1926) and, all seated in the front row, Francis William Aston (second from left; Chemistry 1922), William Bragg (third from left; Physics 1915), Svante Arrhenius (second from right; Chemistry 1903), and Frederick Soddy (far right; Chemistry 1921). Ernest Solvay, the Belgian industrialist and namesake of the conference, is also in the front row, hand resting on a cane. Photo from H. Deelstra, B. Mahieu: Ernest Solvay, les Sociétés chimiques et les chimistes de Belgique à son époque (1863-1922); http://chimienouvelle.be/CN115web/CN%20115%20Deelstra.pdf

During his army enlistment in World War I, he rose from sergeant to second lieutenant but was recalled from the front in April 1915 and reassigned to a gunpowder factory in Angoulême, then to a war laboratory at the *École normale* supérieure. After returning to the University of Brussels, where he resumed his professorial duties, he became involved in administration, serving as the dean of the faculty of sciences from 1919 to 1921 and representing the faculty of sciences in the administrative council from 1922 to 1924. Chavanne participated in the prestigious Solvay conferences on chemistry from 1922 to 1934. A photograph of the participants in the first Solvay Conference on Chemistry in Brussels includes Chavanne.

In May 1940, the university closed due to the events of World War II. Chavanne, a widower in ill health, returned to France with his mother, changing residence several times. In July 1941, he returned to Brussels, passing away within the month on July 29, 1941.

The research of Chavanne spanned a wide range in organic chemistry. His research dealt with subjects such as oxidation, combustion, and properties of hydrocarbons, including studies on the analysis and properties of gasoline. It may be surmised that it was this fuel-related expertise that ultimately led to his service on the Commission des Carburants. One publication, "The Slow Combustion of Hydrocarbons," is apparently a review of this subject (Chem. Abstr. 25: 31101,1931). His paper, "Action of oxygen on 1,4-dimethylcyclohexane," evidently his

sole publication in an American journal (Journal of the American Chemical Society), mentions grants received from the American Petroleum Institute to investigate "the oxidation of different cyclohexane and cyclopentane hydrocarbons with side chains." His work on the Commission on Fuels and producing ethyl esters of palm oil was among the last of his career as no other publications on other topics appear to be available.

DETAILS THAT PARALLEL WITH TODAY

The contributions of Chavanne and apparently four co-workers (last names given as Gillet, Frédéric, van den Heuvel, and Chakhovsky) to the project are detailed in the first of the four technical sections of the extensive report [1]. The discussion in this circumspect section indicates that they not only studied the transesterification reaction, but also analyzed the properties of the resulting fuel, including viscosity, density, heat of combustion, cold flow, and effect of the fuel on the metal of the injectors. It may be noted that the reaction was acid-catalyzed (sulfuric acid) in contrast to modern practices that employ more rapid base catalysis, preferably using alkoxide (methoxide when using methanol as alcohol). Another difference is that today, methanol is widely used to give the methyl esters of the oil or fat, the major reason being that methanol is less expensive than ethanol. It may be noted that a publication under Chavanne's name that appeared posthumously (1943) in a general chemical journal presents some of the information from this chapter [8].

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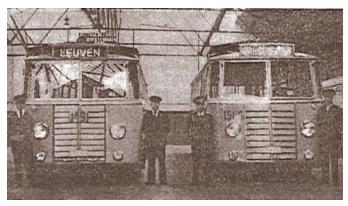


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The second chapter of the report [1] is concerned with the production of palm oil ethyl esters on a semi-industrial scale, which was conducted under the direction of Eugéne Mertens.

The third section discusses the use of the palm oil ethyl esters in the laboratory engine test, as well as a practical test over 20,000 km on the route between Brussels and Louvain (Leuven that was performed under the direction of Albert Coppens (two co-workers with last names given as Defrenne and Lebeau). The results of the likely first cetane test of a biodiesel fuel are presented, agreeing with more recent results that palm oil esters have cetane numbers. The cold start behavior, which is still a significant issue today, is also discussed. Overall, the performance of the biofuel was satisfactory compared to conventional diesel fuel, although corrosion of engine parts due to remaining acidity is mentioned. The fourth section describes the production of palm oil under the auspices of the General Directorate of Agriculture in the Ministry of Colonies.



Buses used in the first practical test of biodiesel drove the route from Brussels to Leuven (Louvain) in 1937. (van den Abeele, M., et al., 1942).

Predating this work using biodiesel as a neat fuel, is a United States patent granted to John W. Orelup and O. Ivan Lee in 1928 [9] which discusses the use of alkyl esters of fatty acids as an ingredient of gasoline (apparently not diesel fuel) to prevent carbon (deposit) build-up in an engine. Approximately at the same time of the Belgian project, John Walton suggests in a publication dating from 1938 [10] that "to get the utmost value from vegetable oils as fuel it is academically necessary to split off the glycerides and to rum on the residual fatty acid," although no practical tests had been carried out.

In conclusion, the work conducted on this project from the 1930s presages much of the recent work on biodiesel, from the background to the technical investigations, with the results largely agreeing with this more present research.

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A rapid fingerprinting Gabriel D. Fernandes and Rosana M. Alberici method for

sterols in oils and sterol-rich foods

Sterols are triterpenicalcohols which occur naturally in plants, animals, and fungi. The most characteristic sterol in animal fats and oils is cholesterol. Plant sterols are called phytosterols, primarily sitosterol and campesterol, with minor but significant contributions from stigmasterol, brassicasterol, and avenasterol. The phytosterol profile is a particular characteristic of each plant, but it can also be affected by refining procedures and storage conditions.

- Health-related labeling claims have increased the need to develop methods for measuring sterols in oils and other sterol-rich foods.
- A new method using direct analysis in real time (DART-MS) was recently developed to enable the rapid determination of the main phytosterols in intact oils and sterol-rich foods.
- The method eliminates the need for several sample preparation steps, and achieves results in a singleanalysis that lasts less than 1 minute.

Phytosterols and phytostanols (the hydrogenated form of phytosterols) are recognized as having functional characteristics related to the prevention of cardiovascular disease. By means of a competing pathway, phytosterols have the ability to lower cholesterol absorption in humans, consequently reducing serum cholesterol and low-density lipoprotein (LDL) cholesterol levels. For these reasons, the US Food and Drug Administration (FDA) allows food and supplement manufacturers to make health claims about the relationship between phytosterols and a reduced risk of coronary heart disease, provided that the products contain specified amounts of the five major phytosterols that have shown beneficial effects (campesterol, campestanol, stigmasterol, β-sitosterol, and sitostanol). Consequently, the ability to analyze sterols in oils and other sterol-rich foods is critical to assessing the validity of such claims.

Previous methods for phytosterol analysis were limited by a lack of validation for stanol quantification, limited range or accuracy, or unsuitability for the analysis of dietary supplements. AOCS approved Official Method Ce 12-16, which was approved in 2016, can be used to determine total free sterols/stanols and total steryl/ stanol esters, as well as to quantify each of the five major phytosterols that are the subject of the FDA's health claim [1]. While the AOCS Method is very accurate, it requires sample preparation with reagents before the phytosterols can be separated and detected using gas-chromatography and flame ionization detection.

Several direct MS protocols reduce the time of analysis by eliminating the chromatographic step. The introduction of ambient desorption/ionization techniques [2] enabled further simplifi-



cation through direct analysis in real time-mass spectrometry (DART-MS) [3, 4]. DART-MS allows samples to be analyzed in an open-air ambient in just a few seconds, with minimal or no sample preparation. Our research group recently developed a new method, which we described in Food Chemistry [5], based on transmission mode DART (TM-DART-MS) to characterize sterols and related compounds in vegetable oils, commercial blended vegetable oils, sterols enriched margarines, butter, and animal oils.

HOW TM-DART-MS WORKS

DART was developed in 2005, and is now marketed commercially by JEOL and IonSense (Fig. 1). It is an atmospheric pressure ion source which instantaneously ionizes gases, liquids, and solids in the open air under ambient conditions. DART ionization combines the techniques of thermal desorption and Penning ionization. Basically, the gas (N₂, Ne, or He) enters the ion source, and an electrical potential (+1 to +5 kV), generating a glow discharge containing excited-state species (metastable species). The gas stream can then be heated from room temperature to 450°C, depending on the surface or chemical being analyzed. The excited-state species can interact directly with the sample to desorb and ionize the analyte. The ions formed are directed to the mass spectrometer inlet by both the gas flow and a slight vacuum in the spectrometer inlet. DART produces relatively simple mass spectra, dominated by

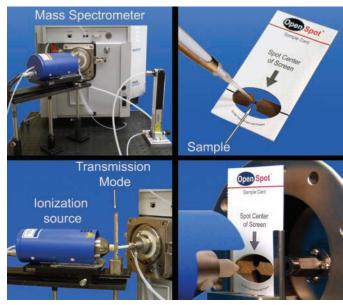


FIG. 1.TM-DART-MS system as described (Prof. Facundo Fernandez's Lab, School of Chemistry and Biochemistry, Georgia Institute of Technology, Atlanta, USA). 2 µL of the sample, previously diluted in acetone (1:1 v/v), are deposited directly on an open spot sample card (IonSense, Saugus, MA, USA) that is placed into the holder of a commercial DART-SVP 100 (IonSense, Saugus, MA, USA) coupled to a hybrid QTOF mass spectrometer (micrO-TOF-QI, Bruker, Bremen, Germany)

protonated molecules [M+H]⁺ in positive-ion mode, or deprotonated molecules [M-H]⁻ in negative-ion mode.

PHYTOSTEROLS IN VEGETABLE OILS

A TM-DART-MS typical profile from vegetable oil is characterized by four distinct areas corresponding to: free fatty acids (FFA) + monoacylglycerols (MAG); sterols + squalene + triterpenic alcohols; diacylglycerols (DAG) and triacylglycerols (TAG) (Fig. 2).

Since TAG are the main components in oils and fats, the MS is characterized by TAG ions as well as fragment ions—DAG and MAG—that appeared as the result of the in-source fragmentation during DART ionization. Phytosterols are observed in the region between *m/z* 350 and 450 (Table 1).

Besides, its precursor, squalene, can also be detected in olive oil. Olive and canola oils can be easily differentiated based on their sterol MS profiles (Fig. 3), although β -sitosterol is the most abundant phytosterol-related ion present in these samples. These fingerprint profiles can be used as an identity parameter for vegetable oils. In addition to providing characterization, sterols TM-DART(+)-MS analysis could, therefore, serve as a traceability parameter in identifying and detecting adulteration.

PHYTOSTEROLS AND PHYTOSTANOLS FROM STEROL-RICH FOODS

As an example of sterol-enriched margarines, we have analyzed two commercial spreads: Benecol®, which is enriched with stanol fatty acid (FA) esters, and Promise Active®, which is enriched with sterol FA esters. The sterol MS profiles showed that is possible to identify separately the free sterols/stanols, instead of the sterol/ stanol ester as a unique molecule (Fig. 3). Here, we had another occurrence of fragmentation on the ionization source. From Benecol*, the main stanols ions were sitostanol and campestanol, consequently related to sitostanyl FA esters and campes-

FIG. 2.TM-DART-MS typical profile from vegetable oil (in this example, an olive oil sample). P=palmitic acid, S=stearic acid, L=linolenic acid, O=oleic acid

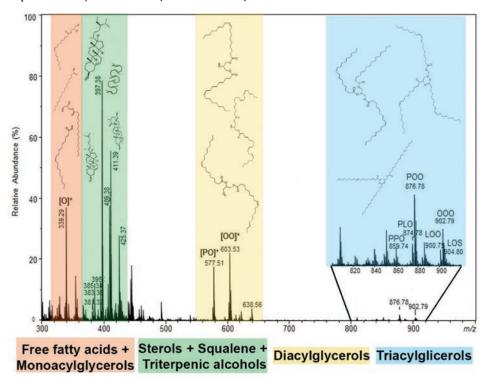


FIG. 3. TM-DART(+)-MS of olive oil, canola oil, Benecol® spread, Promise Active®, fish oil, Heart Choice® Plant Sterols (sterols esters such as β -sitosterol (300 mg), campesterol (163 mg) and stigmasterol (117 mg)/softgel)

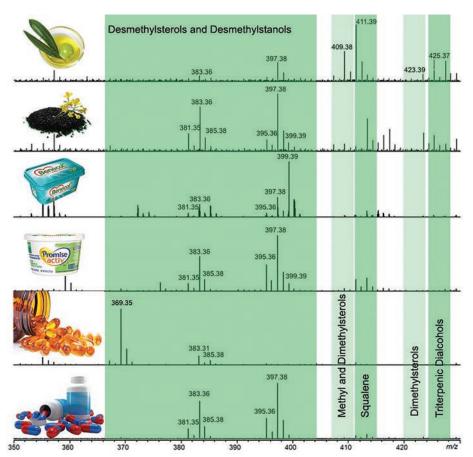


TABLE 1. Ion assignment to phytosterols-specific species found in vegetable oils by TM-DART-MS

Compound	[M–H ₂ O+H] ⁺ <i>m/z</i>	Molecular formula	
Cholesterol	369.35	C ₂₇ H ₄₆ O	
Brassicasterol	381.35	C ₂₈ H ₄₆ O	
Campesterol	383.36	C ₂₈ H ₄₈ O	
Brassicastanol	383.30		
Campestanol	385.38	C ₂₈ H ₅₀ O	
Hydroxi-cholesterol	385.34	C ₂₇ H ₄₆ O ₂	
Stigmasterol			
Δ5-Avenasterol	395.36	C ₂₉ H ₄₈ O	
Δ7-Avenasterol	393.30		
Grammisterol			
β-Sitosterol	397.38	C ₂₉ H ₅₀ O	
Stigmastanol	397.36		
3,5-Stigmastadiene	397.38*	C ₂₉ H ₄₈	
Sitostanol	399.39	C ₂₉ H ₅₂ O	
Cycloleucalenol			
Obtusifoliol		C ₃₀ H ₅₀ O	
Citrostadienol			
Cycloartenol	409.38		
Butyrospermol			
Amyrins			
2,3-Epoxisqualene			
24-Methylene cycloartanol	423.39	C ₃₁ H ₅₂ O	
Squalene	411.39*	C ₃₀ H ₅₀	
Erythrodiol* Uvaol*	425.37 / 443.48* / 407.36**	C ₃₀ H ₅₀ O ₂	

^{*[}M+H]*** [M-2H2O+H]+

tanyl FA esters fragment ions. Promise Active® showed ions attributed to sitoesteryl FA esters and campesteryl FA esters.

CHOLESTEROL FROM ANIMAL FATS AND OIL

Determination of cholesterol in animal fats and oils is an important topic in the food industry, since high amounts of cholesterol in foods are closely related to cardiovascular disease risks. Cholesterol has also been commonly used to detect mixtures of animal oils in vegetable oils. TM-DART(+)-MS was also applied to the detection of cholesterol in a cod fish liver oil sample (Fig. 3).

In conclusion, TM-DART-MS has been found to offer a novel, simple, and high-throughput platform that can be used to characterize sterols and related compounds in fats and oils. The methodology is much less time-consuming and avoids laborious sample preparation and/or derivatization steps employed by alternative methodologies.

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Sheep milk: an unexplored food matrix to develop C. F. Balthazar and A. G. Cruz functional foods

The main constituents of milk are water, fat, protein, lactose, vitamins, and minerals, but the chemical composition of any type of fresh milk varies over time depending on such factors as lactation stage, animal age, breed, number of milking sessions per day, the time of year, environmental temperature, lactation efficiency, animal nutrition, genetic factors (not only at the species level, but also with respect to breed), hormones, and/or udder disease. For example, climatic variation and seasonal changes may affect the physiology of milk-producing animals and, consequently, the quality and availability of the nutrients found in their milk.

- Due to sheep milk's high content of solids, it is primarily used to make cheese.
- Although highly nutritious, sheep milk's potential with respect to functional foods remains largely unexplored.
- This article compares the chemical composition and nutritional benefits of sheep milk with those of cow and goat milk.

But although the fatty acid composition and minor compounds in milk are influenced by physiological and environmental factors, the milk from each species has its own characteristic composition. For instance, sheep milk has a distinctively high solids content, which makes it particularly suitable for cheese making. One gallon of sheep milk produces up to 2.5 times as much cheese as a gallon of cow or goat milk. Popular varieties of cheese made with sheep milk include feta, ricotta, Pecorino Romano, Roquefort, and Manchego.

Sheep milk is also richer in proteins, lipids, minerals (calcium, phosphorous, potassium, and magnesium), and essential vitamins (A, B, and E) than cow's milk. In fact, it has three times more protein than goat or cow's milk (Table 1), and the proteins in sheep milk have high biological value, which contributes to better digestibility.

TABLE 1. Proximate composition of cow, goat, and sheep milk

Parameter	Cow milk	Goat milk	Sheep milk
Moisture (g/100 g)	87.9 ± 0.5	87.6 ± 0.7	82.9 ± 1.4
Fat (g/100 g)	3.3 ± 0.2	3.8 ± 0.1	5.9 ± 0.3
Ash (g/100 g)	0.7 ± 0.0	0.8 ± 0.1	0.9 ± 0.1
Lactose (g/100 g)	4.7 ± 0.4	4.1 ± 0.4	4.8 ± 0.4
Protein (g/100 g)	3.4 ± 0.1	3.7 ± 0.1	5.5 ± 1.1
Casein (g/100 g)	3.0 ± 0.1	2.4 ± 0.1	4.7 ± 0.5
αs1–casein (%)²	39.7	5.6	6.7
αs2–casein (%)²	10.3	19.2	22.8
β–casein (%)²	32.7	54.8	61.6
κ–casein (%)²	11.6	20.4	8.9

Source: Balthazar et al. (2017).

Structural conformation, micelles content, and casein subunits in sheep milk resemble those of goat milk, which are considered to be less sensitizing than cow's milk.

Studies indicate that sheep milk can serve as a substitute for people who are allergic to bovine milk due to its higher content of primary milk components and minerals.

HEALTH AND NUTRITION

However, it has been found that IgE antibodies from people with allergies recognize $\alpha S1\text{-casein},$ $\alpha S2\text{-casein},$ and $\beta\text{-casein}$ from sheep and goat milk, but rarely recognize the caseins in cow's milk. Also, the genetic polymorphisms of milk proteins play an important role in inducing different degrees of allergic reaction.

Caseins represent 80% of the protein in ruminant milk, while caseins make up a much smaller percentage of the proteins in human milk. The mineralization and hydration characteristics of ruminant milk caseins vary, depending on the species. For example, compared to cow's milk, sheep and goat milk casein micelles have higher mineralization and lower hydration, which makes them more stable when heated.

The molecular and sequential conformation of amino acids in ruminant milk is also species-specific and impacts digestibility, nutritional quality, and protein thermostability. The high percentage of heat-resistant caseins and heat-sensitive whey proteins in sheep milk are responsible for the unique texture and viscosity of yogurts made with this milk. Technologically, these proteins have unique properties that allow the milk to be converted easily into yogurt and cheeses without having to add other solids, such as thickeners or milk powder, or use membrane technology to concentrate the milk solids. Studies with sheep casein variants can identify economically important traits that can be used to improve sheep breeds for the production of specific milk proteins.

The characteristics of casein in sheep milk are particularly interesting due to high numbers of polymorphisms related to cheese manufacturing. Sheep milk is primarily used to make cheeses, which are in increasing demand. Therefore, there is a lack of knowledge about the role milk's natural proteolytic and lipolytic enzymes have on cheese formation and ripening.

The taste and aroma of sheep milk are smooth and sweet; moreover, its creamy texture is due to the small fat globules. This peculiarity in the size of sheep milk fat globules facilitates its digestion.

Lipases play an important role in milk production within the mammary gland. Lipases in milk catalyze and hydrolyze the triglycerides, producing free fatty acids (FFA). The activity of lipases in sheep milk is about one-tenth of the lipase activity in bovine milk. In sheep, the triglycerides containing medium-chain fatty acids have a higher rate of hydrolysis than those containing longchain fatty acids. FFA levels in sheep milk cheeses result from the lipolytic process that occurs during maturation. Also, in comparison with cow's milk, sheep milk has higher concentrations of polyunsaturated fatty acids, including isomers of conjugated linoleic acid (cis-9, trans-11, trans-10, and cis-12) which are responsible for anti-carcinogenic and lipolytic actions.

Sheep milk has an intense, homogeneous white color due to the absence of β-carotene pigment—a precursor of retinol (vitamin A) that causes cow's milk yellowish color. In contrast, sheep milk contains the converted form, vitamin A, which does not have a yellowish color.

Dairy products derived from sheep milk, such as Greek yogurt and cheeses, have been described as having angiotensin-converting enzyme (ACE) inhibitor peptides, most of which are derived from the β -casein subunit. Also, some κ -caseinderived peptides resulting from the hydrolysis of sheep milk by pepsin, trypsin, and chymotrypsin, are known to exert antioxidant activity. Several peptides with ACE inhibitory action and antibacterial action have been identified in traditional cheeses derived from sheep milk.

Although sheep milk is a rich source of nutrients, its main use is for cheese production, during which the milk's high content of total dry solids contributes to high yields. However, sheep milk remains largely unexplored from a functional food point of view. Since milk and its derivatives are recognized to be excellent carriers of prebiotics and probiotics, sheep milk has great potential as a probiotic bacteria carrier and a valuable alternative to the dairy industry.

Celso Fasura Balthazar is a doctoral student at Universidade Federal Fluminense, Brazil. At the moment, he is at Università degli Studi di Foggia (Italy), as an external PhD student. His research focuses on enhancing the functional appeal of dairy products with prebiotics and probiotics, with an emphasis on developing functional dairy products based on sheep's milk. He can be contacted at celsofasura@id.uff.br. Adriano Gomes da Cruz is a professor in Instituto Federal do Rio de Janeiro, Brazil. His research involves dairy products and novel technologies applied in food science.

Further reading

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New technology produces seed oils from plant cells

Olio is an Inform column that highlights research, issues, trends, and technologies of interest to the oils and fats community.

Laura Cassiday

In April 2017, at the In-Cosmetics Global exhibition in London, a UK-based company called Olixol (www.olixol.com) officially launched their borage and jojoba oils for personal care products. These oils were not extracted from the seeds of plants grown the conventional way. Instead, they were produced in large tanks of cultured plant cells. According to some experts, Olixol has the potential to transform the personal care industry by providing a reliable source of high-value seed oils, while avoiding the fluctuations in availability and price typical for such oils.

With Olixol's patented technology, oil-producing cells are identified and isolated from the seeds of interest. Then, the cells are propagated in a proprietary medium and fed sugar in the presence of light and air. The cells produce oil, which separates naturally and is removed from the tank. The process allows for the continuous production of seed oils, without the agricultural land or the laborious cultivation, harvesting, crushing, and extraction required for oilseeds.

I recently had the opportunity to speak with Olixol's CEO, Tim Merrell, about the company's technology, current offerings, and future prospects for personal care and beyond.

Can this technology be scaled up to produce large quantities of edible oils, such as soybean oil?

Most companies that do bio-manufacturing have challenges scaling up. We've actually done the reverse. We're scaling down for these specialty oils for personal care and nutrition. For the first five or six years of the company's existence, we were focusing very much on more industrial-type oils, so at that time we were operating in bioreactors, vessels that were 25,000 L in capacity, and we were producing castor oil.

The technology that we're using for personal care is what we call a single-stage technology, so we're feeding dextrose, a monosaccharide, heterotrophically into the cell culture. Whereas for our lower-value, higher-volume oils, such as castor oil, we actually use a two-stage process where we are taking waste CO_2 from a corn ethanol plant, using one set of tanks to turn the CO_2 to C_3 glyceral-dehyde, an intermediate carbohydrate. Then we're feeding that C_3 into the second stage to produce the oil.

So it all comes down to economics. For some of the smaller-batch, high-value oils for personal care, dextrose is the easier way because we have flexibility of where we produce. We don't have to co-locate next to a large source of waste carbon. It just gives us a lot more flexibility. But we definitely see benefits for larger-volume oils. We've moved away from biofuels over the last few years because the price of mineral oil is so low that there's less incentive to move toward the biofuels space again.

Are you interested in eventually applying this technology to some of the widely consumed edible oils, such as soybean, canola, or palm oil?

Absolutely. Again, for us there's sort of a breakpoint where oils could either be produced viably using just dextrose, or whether we have to look into the two-stage conversion of CO_2 to C_3 and then C_3 to oil. For the two-stage process, this is something that we'd probably do with partners because it's a fairly capital-intensive business in order to scale it up. But yes, in the future we could definitely see the technology being used for the more widely consumed edible oils. We think this could be a really viable way to produce them.

How does the cost of your process, including the sugar, compare to the cost of oilseed cultivation, harvesting, and oil extraction?

We are cost-competitive, but we're not looking to be a discount in the market in any way. In personal care, we believe that we add sufficient value and that we are a drop-in replace-

ment. The purity of the oils we produce is very high. They're not exposed to high temperature or solvents. They're very fresh—we produce them constantly throughout the year. Within an hour or so of being produced, the oils are filtered to remove any moisture, and they're nitrogen blanketed. The sustainability story is fairly strong, even factoring in the amount of land used to produce the feedstock for dextrose. We're still using significantly less land than agri-cropping, and also a lot less water.

Do you only have to isolate cells from the plant embryos once, or does this need to be done periodically?

No, it's typically done just the one time. However, it's done many times in our evaluation of an oil. So we take seeds from various sources, and then at a bench-top reactor level, we isolate and dissect the seed, placing the oil-producing cells into our proprietary media. Then we grow small amounts of oil in the laboratory, where we can analyze it not just from a fatty acid point of view, but more in depth, for all of the components, such as tocopherols, phytosterols, etc. Then we can pick the cell line that we believe delivers the best quality oil that the market is looking for.

Once that's been done, it's an ongoing process, and we keep the cell line going. The dextrose is fed in. The oil that is produced within the cells is naturally separated. It's going through the lipid layer of the cell wall, and then it just rises to the top, and we use volumetric displacement to pull the oil off. So the cells remain undamaged and unstressed, and typically a reactor needs to be stripped down and repopulated with cells only once every two years.

For edible oils, how would refining fit in with your

It's an interesting question, the whole refining process, whether it's needed or not. If you're talking about canola or palm oil, which are two oils we've produced in large volumes, the market would expect these to be refined oils. Typically, the refining process is only required because the extraction process, either the heat or the solvent extraction, has introduced impurities from the seed itself rather than from the oil, and those need to be removed. So sure, there might be some degumming involved, for example, but we think that we can deliver a very stable raw oil, and if the end user chooses to refine that or not, that would be up to them.

Do you currently offer only jojoba and borage oils, or are there others for sale?

At this time, those are the only ones that we're selling publicly in the open market. We have engaged with most of the major personal care and cosmetic businesses, and we're now working on various specific projects for them. These are typically oils that are either supply constrained or nature constrained, just simply because there is not enough seed available. For instance, sometimes people manage to gather enough seeds of a wild plant to establish a functional or bioactive benefit, but these plants could never be grown in a commercial plantation setting.

We're also very mindful of the Nagoya protocol, which seeks to prevent the bio-piracy of resources that are specific to certain geographic areas. So in our UK production facility, we are limited somewhat in the number of oils that we can produce. Our next commercial development is a production facility in Brazil in 2018, which will enable us to produce many Amazonian oils that are very exciting. Many of these have not been available outside of the Brazilian market because the Brazilian market is an enormous personal care market, and most or all of the production has been taken up by domestic demand. So we're really excited about that, and by producing in Brazil, we will be Nagoya-compliant, also.

Are your oils sold just in Europe?
We currently have a distribution network in France, Italy, Germany, Austria, Switzerland, Spain, and the UK. We haven't yet appointed a distributor in the US, although we are in talks with a couple of people there. We would also definitely consider setting up production in the US to address US domestic demand. It's a technology that travels very well.

Is there anything else you would like to say about Olixol?

We're really excited by the opportunity that personal care offers. I think that the formulators for cosmetic companies have put up with less-than-ideal-quality oils for a long time, and this is a chance for us to provide to them a very consistent and pure product, and to be able to deliver on a regular basis, rather than have the supply spikes and the price volatility that has been part and parcel for the exotic oil market. So hopefully we can address the key needs that they have, and push forward and offer a broader range of oils year by year.

Olio is produced by Inform's associate editor, Laura Cassiday. She can be contacted at laura.cassiday@aocs.org.

AOCS MEETING WATCH

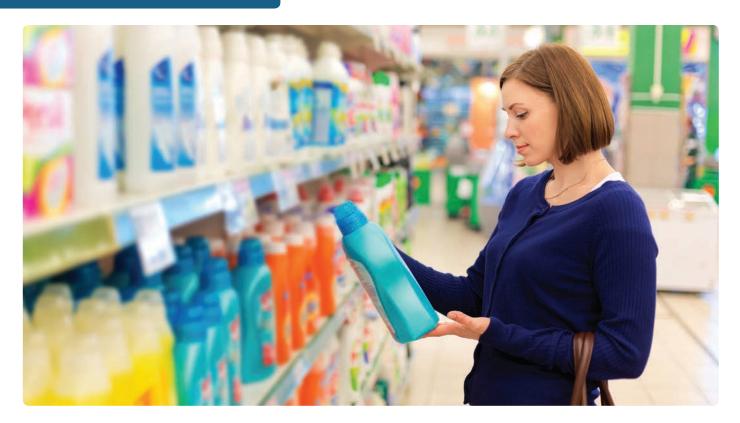
September 11–14, 2017. 17th AOCS Latin American Congress and Exhibition on Fats, Oils, and Lipids, Grand Fiesta Americana Coral Beach Hotel, Cancun, Mexico. http://lacongress.aocs.org

May 6–9, 2018. AOCS Annual Meeting & Expo, Minneapolis Convention Center, Minneapolis, Minnesota, USA.

September 6, 2018. JOCS AOCS Joint Symposium, Kobe Gakuin University, Arise Campus, Kobe, Japan

October 28-31, 2018. Fabric and Home Care World Conference, Boca Raton Resort & Club, Boca Raton, Florida, USA.

For in-depth details on these and other upcoming meetings, visit http://aocs.org/meetings or contact the AOCS Meetings Department (email: meetings@aocs.org; phone: +1 217-693-4821; fax: +1 217-693-4865).



EU sector-specific classification of specialty chemicals

Regulatory Review is a regular column featuring updates on regulatory matters concerning oils- and fats-related industries.

Radu-Adrian Gropeanu and Daniela Schroth

Registration dossiers submitted to Echa contain proposals for the classification and labelling of a substance in accordance with the CLP Regulation.

Under certain circumstances, the classification of a substance is added to Annex VI of CLP (list of harmonized classification and labelling of certain hazardous substances). Once on this list, the classification of a substance is then obligatory for all manufacturers and suppliers who must comply with REACH.

Harmonization of classifications is mostly aimed at substances that are carcinogenic, mutagenic, toxic for reproduction, or respiratory sensitising; however, they may also be determined for other hazard categories.

But what about substances not regulated under REACH? The registration of polymers, for example, is not required in the EU, unlike in other countries, such as in China, the United States and Japan.

How can we be sure that these are also adequately tested and classified so that there are no risks to human health and the environment, while keeping down costs to industry?

Addressing this, members of associations such as the European Committee of Organic Surfactants and their Intermediates (CESIO) and the European Center of Silicates (CEES) are developing classification strategies for their respective chemicals.

CESIO CLASSIFICATION OF SURFACTANTS

Surfactants are substances that lower the surface tension between two liquids, or a liquid and a solid. They can be used as detergents, emulsifiers, dispersants, or foaming agents. Surfactants can be classified as polymeric or non-polymeric, or

by their chemical nature, such as anionics, cationics, nonionics, or zwitterionics.

Production of surfactants was estimated at more than 3 million metric tons in Western Europe in 2015, with the ethoxylates and the anionics being produced in much higher volumes.

CESIO was founded in the 1970s by several chemical companies to improve knowledge on the safe use of surfactants. Each of the surfactant families considered—grouped by the chemical nature of the hydrophilic head—was assigned to a company. These "lead" companies for each substance class performed toxicological tests on mammals. The results were then published with the recommendations for classification.

Since CESIO's first list of recommendations, released in 1984, new issues, covering more classes of surfactants, in addition to more end-points such as ecotoxicological properties, have been added. The latest list, released in March 2017, contains the CLP and the GHS-UN classifications for the majority of surfactants available on the market. For some substance families however, it was not possible to create GHS-UN recommendations; in these cases, the individual companies have the responsibility for the GHS-UN classification.

READ-ACROSS

The properties of polymers vary significantly with the molecular weight. Therefore, one chemical nature (described by a unique CAS number) may have several different classifications depending on the degree of polymerisation (for example the fatty alcohol to ethylene oxide ratio for the ethoxylated fatty alcohols).

An important step forward was to set-up the read-across principle in order to limit animal testing. Based on this, it was no longer necessary to test each member of a polymeric surfactant family. The classification decision was made by testing some of the homologues, correlating the data to the chemical structure and clustering the surfactants in groups of substances sharing the same classification. This principle was eventually accepted by the EU and introduced in the detergents Regulation.

Some polymer classes, previously listed on CESIO's harmonized classifications list have been removed. For example, the mixed ethoxylated and propoxylated fatty alcohols have been removed due to their structural complexity which brings high differences in properties and therefore makes clustering very difficult. Nowadays these polymers are separately assessed.

CEES CLASSIFICATION OF WATER SOLUBLE SILICATES

In 2010, CEES members were responsible for more than 90% of the total Western European soluble silicates production. Soluble silicates have a wide variety of industrial, professional and consumer uses, such as in the detergent industry or pulp and paper manufacture. In contrast to the polymers of CESIO, the CEES members had to register their products under REACH. To achieve this, the members collected all published and internal data about chemical, physical, toxicological, and

ecotoxicological properties of soluble silicates. A consortium was founded outside of CEES, which, provided with this data, has the job of registering the materials covered by the work of the association.

Toxicological tests have revealed that the health hazards, especially skin and eye irritation, are strongly influenced by the molar ratio (MR)—which defines the number of moles of silica per mole of alkali metal oxide in soluble silicates. Tests also found that the form of the product had an effect, with less severe results at high MRs and a non-solid form of the product.

Ecotoxicological tests also revealed that these products are, with a high probability, not harmful to aquatic organisms.

ANIMAL TESTING

One of the main goals of REACH is the avoidance of unnecessary animal testing. The set-up of consortia for the registration of chemicals has been welcomed in order to fulfil this requirement. Through the exchange of data and, in the case of CESIO, the assignment of leads for each chemical family, the companies have much lower overall costs (testing and registration) in comparison to the cost to each company if they were to register every substance.

Nowadays, the classification recommendations of the associations for the non-polymeric surfactants, as well as the water soluble silicates, are identical to those in the REACH registration dossiers. CESIO members agreed on harmonized classifications, based on lead company testing and the read-across principle, for substances exempt from registration under REACH, namely polymers. In fact, the majority of the substances from the latest CESIO recommendation list are polymers which were tested and classified based on the same high REACH standards used for the registration of non-polymeric substances. Moreover, in the last decade, the association has made efforts to assess the hazards of the surfactants following GHS-UN classification rules.

OUTLOOK

Membership of associations offers manufacturers the opportunity to exchange information on their products and to avoid unnecessary tests and, consequently, save time and money. The provided classifications, based on the GHS rules, allow a worldwide consistent classification of the products brought on the market by the global producers, which also makes the work easier for the potential downstream users.

In the context of polymers, the consortia formation is also very important, as registration under REACH looks more and more likely. Here, associations offer access to existing data as well as the experience gained over decades for classification and registration of certain polymer groups, so that new studies do not need to be initiated.

Radu-Adrian Gropeanu and Daniela Schroth are product safety managers at Dr Knoell Consult.

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Argentina: marketing trends and food labeling



Leslie Kleiner

Food labeling usually mirrors marketing trends and/or consumer demands and expectations. To understand the trends driving food labels in Argentina, I interviewed Cristina Zapata, a specialist in international commerce and professor of graduate studies at the University of Buenos Aires.

Which food marketing trends are Argentinean consumers following?

Nutrition is a very important aspect of food, but food is also a social event permeated by cultural beliefs. Therefore, to understand the market in Argentina, it is important to discuss the relationship Argentineans have with food. In this respect, most Argentinean men value large and tasty meals with high quality ingredients. In contrast, Argentinean women tend to focus more on their personal appearance than on nutrition, even when this focus may lead to eating disorders. These trends are applicable to high-and middle-class populations, in which it is common to see families comprised of very thin mothers and overweight fathers and children. The rest of the population focuses on a more traditional approach of meals made at home. However, those below the poverty level rely on social programs for access to a daily food plate.

Given this context, food and beverage companies make use of a lexicon that targets various beliefs and market sectors. For example, an idea that has now been engrained is that the word "LIGHT" or "CERO" (zero in English) is synonymous with the word "diet." Because of this, there is a parallel campaign from official regulatory agencies indicating that the word "light" should not be associated with weight loss, calorie reduction, or diet. However, most people who read labels focus on the main package claim (e.g., "LIGHT") and disregard ingredient lists and full nutritional panels. Generally speaking, nutritional panels are read for a description of caloric value only. Examples of commercially available products with the "LIGHT" claim on the package include potato chips, salad dressings, mayonnaise, yogurt, cream cheese, and canned fruit, among others.

Besides the "LIGHT" label, how is the "FAT FREE" label positioned in the market?

In the dairy industry, the term "DESCREMADO" is a euphemism for the term "FAT FREE." Products bearing the "DESCREMADO" label are slightly more costly than those made with whole milk.

Recently, the dairy company "La Serenisima" introduced for its Finlandia Balance cheese a campaign utilizing the "BAJO EN GRASA" (low in fat) label. This label differentiates the product from others that are fat-free. However, this message was not readily accepted by consumers, since they don't easily differentiate between the concepts: LIGHT, DESCREMADO, and BAJO EN GRASA (light, fat-free, and low in fat, respectively).

Furthermore, medical doctors, nutritionists, aestheticians, and career models populate the media with opinions and advice on health, nutrition, and diet, which further contributes to the confusion.

How are "ORGANIC" and "FAIR TRADE" labels perceived by consumers?

Organic Food and beverages have low demand since their production is limited and mostly reserved for exports. A limitation of organic products is that many local producers do not have registered brands; they end-up selling the product at a

good price (American dollars) but either in bulk and/or without branding.

The concept of Fair Trade is not yet active in the market. As part of a social campaign, our previous government (President Cristina Fernández de Kirchner), started the promotion of "fair trade" products. However, the commercial success of these products was quite limited. Currently, the federal government under the new administration (President Mauricio Macri) is fomenting public markets for small-scale produce

growers to sell directly to consumers, thereby avoiding middle-man operations. This is more of a social action rather than fair trade promotion, but it seems to point in that direction.

Latin America Update is produced by Leslie Kleiner, R&D Project Coordinator in Confectionery Applications at Roquette America, Geneva, Illinois, USA, and a contributing editor of *Inform*. She can be reached at LESLIE.KLEINER@roquette.com.

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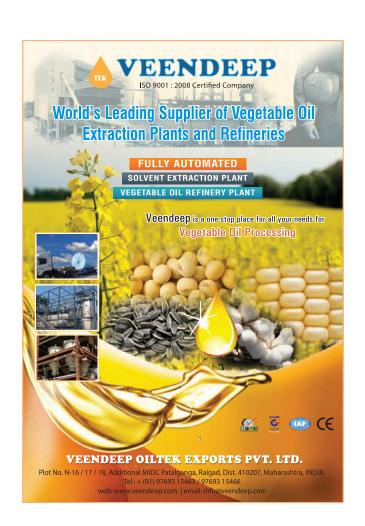
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PATENTS

Methods and compositions to reduce serum levels of triacylglycerides in human beings using a fungal lipase

Schuler, C., et al., Bio-Cat, Inc., US9555083, January 31, 2017

The invention relates to methods and compositions for reducing serum levels of triacylglycerides in human subjects. In particular, the invention relates to the oral administration of an effective amount of a fungal lipase formulation, to a human subject having borderline-high or high serum levels of triacylglycerides, for a time period sufficient to reduce serum triacylglyceride levels.

Antimicrobial compositions containing free fatty acids

Folan, M.A., inventor, US9555116, January 31, 2017

The invention concerns antimicrobial compositions comprising free fatty acids emulsified with membrane lipids or hydrolyzed derivatives thereof, and pharmaceutical formulations comprising same. The compositions can be used in the treatment of prophylaxis of microbial infections. They can also regulate the rate of blood clotting rendering them suitable for incorporation in catheter locking solutions and for use in wound care.

Porous structure for forming antifingerprint coating, method of forming anti-fingerprint coating, substrate comprising the anti-fingerprint coating formed by the method, and product comprising the substrate

Lee, E.J., et al., LG Chem, Ltd., US9556341, January 31, 2017

Provided are a porous structure for forming anti-fingerprint coating capable of providing a self-cleaning function to a surface of a substrate, a method of forming anti-fingerprint coating using the same, an anti-fingerprint coated substrate prepared by the same method, and a product including the same. When the porous structure including a lipolytic enzyme is formed on the surface of the substrate, contaminants decomposed by an enzyme are absorbed into a pore, and thus anti-fingerprint coating may be more effectively performed to remove detectable contamination from a surface of the substrate. As a result, contamination by fingerprints

on the surface of a display device, the appearance of an electronic device, or building materials can be effectively reduced.

Blown and stripped plant-based oils

Hora, M.J., et al., Cargill, Inc., US9556398, January 31, 2017

A method for producing a high-viscosity, low volatiles blown stripped plant-based oil is provided. The method may include the steps of: (i) obtaining a plant-based oil; (ii) heating the oil to at least 90C; (iii) passing air through the heated oil to produce a blown oil having a viscosity of at least 200 cSt at 40C; (iv) stripping the blown oil from step (iii) to reduce an acid value of the blown oil to from 5 mg KOH/g to about 9 mg KOH/g; (v) adding a polyol to the stripped oil from (iv), and (vi) stripping the oil from step (v) to reduce the acid value of the oil to less than 5.0 mg KOH/g or less.

Method for producing EPA-enriched oil and DHA-enriched oil

Furihata, K., et al., Nippon Suisan Kaisha, Ltd., US9556401, January 31, 2017

Alcoholysis of oils and fats containing EPA and DHA is performed by a lipase having substrate specificity for fatty acids having 18 carbons or less and in the presence of a reaction additive such as magnesium oxide; then the glyceride fraction is separated; alcoholysis of the glyceride fraction is performed by a lipase having substrate specificity for fatty acids having 20 carbons or less and in the presence of a reaction additive such as magnesium oxide; and EPA-enriched oil and DHA-enriched oil are simultaneously obtained.

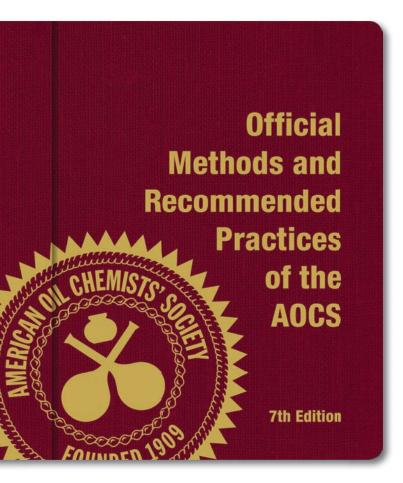
Hydroformylation of triglycerides in a self-emulsifying medium

Hapiot, F., et al., Centre National De La Recherche Scientifique (CNRS) and Universite D'Artois, US9556402, January 31, 2017

The invention relates to a method for the hydroformnylation of triglycerides by homogeneous catalysis in the presence of at least one substituted cyclodextrin, said method comprising a step a) of combining, under agitation, at least one catalyst, waiter, at least one unsaturated triglyceride and said substituted cyclodextrin, in the presence of gaseous hydrogen and carbon monoxide, said step being carried out in reactive conditions allowing the formation of an emulsion during the agitation and a decanting of the products once the agitation has stopped.

Patent information is compiled by Scott Bloomer, a registered US patent agent with Archer Daniels Midland Co., Decatur, Illinois, USA. Contact him at scott.bloomer@adm.com.





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- Cd 30-15 (Official Method) Analysis of 2- and 3-MCPD Fatty Acid Esters and Glycidyl Fatty Acid Esters in Oil-Based Emulsions
- ► Ce 12-16 (Official Method) Sterols and Stanols in Foods and Dietary Supplements Containing Added Phytosterols
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EXTRACTS & DISTILLATES

Dietary lipids differentially modulate the initiation of experimental breast carcinogenesis through their influence on hepatic xenobiotic metabolism and DNA damage in the mammary gland

Miguel Ángel Manzanares, et al., J. Nutritional Biochem. 43: 68-77, 2017, http://dx.doi.org/10.1016/j.jnutbio.2017.01.016.

Breast cancer is the most common malignancy among women worldwide. In addition to reproductive factors, environmental factors such as nutrition and xenobiotic exposure have a role in the etiology of this malignancy. A stimulating and a potentially protective effect on experimental breast cancer has been previously described for high corn oil and high extra-virgin olive oil diets, respectively. This work investigates the effect of these lipids on the metabolism of 7,12-dimethylbenz(a)anthracene (DMBA), a polycyclic aromatic hydrocarbon that can initiate carcinogenesis and its consequences in an experimental rat breast cancer model. The PUFA n-6-enriched diet increased expression of Phase I enzymes prior to DMBA administration and raised the activity of CYP1s in the hours immediately after induction, while reducing the activity of Phase II enzymes, mainly NQO1. The levels of reactive metabolites measured in plasma by GC-MS and DMBA-DNA adducts in the mammary gland of the animals fed the high corn oil diet were also higher than in the other groups. On the other hand, the high extra-virgin olive oil diet and the control low-fat diet exhibited better coordinated Phase I and Phase II activity, with a lower production of reactive metabolites and less DNA damage in the mammary gland. The concordance between these effects and the different efficacy of the carcinogenesis process due to the dietary treatment suggest that lipids may differently modify mammary gland susceptibility or resistance to cancer initiation over the exposure to environmental carcinogens.

A lipidomic study on the regulation of inflammation and oxidative stress targeted by marine ω-3 PUFA and polyphenols in high-fat high-sucrose diets

Gabriel Dasilva, et al., J. Nutritional Biochem. 43: 53-67, 2017, http://dx.doi.org/10.1016/j.jnutbio.2017.02.007.

The ability of polyphenols to ameliorate potential oxidative damage of ω -3 PUFAs when they are consumed together and then, to enhance their potentially individual effects on metabolic health is discussed through the modulation of fatty acids profiling and the production of lipid mediators. For that, the effects of the combined consumption of fish oils and grape seed procyanidins on the inflammatory response and redox unbalance triggered by high-fat high-sucrose (HFHS) diets were studied in an animal model of Wistar rats. A standard diet was used as control. Results suggested that fish oils produced a replacement of ω -6 by ω -3 PUFAs in membranes and tissues, and consequently they improved inflammatory and oxidative stress parameters: favored the activity of 12/15-lipoxygenases on ω -3 PUFAs, enhanced glutathione peroxidases activity, modulated proinflammatory lipid mediators synthesis through the cyclooxygenase (COX) pathways and down-regulated the synthesis *de novo* of ARA leaded by $\Delta 5$ desaturase. Although polyphenols exerted an antioxidative and antiinflammatory effect in the standard diet, they were less effective to reduce inflammation in the HFHS dietary model. Contrary to the effect observed in the standard diet, polyphenols up-regulated COX pathways toward ω-6 proinflammatory eicosanoids as PGE₂ and 11-HETE and decreased the detoxification of ω -3 hydroperoxides in the HFHS diet. As a result, additive effects between fish oils and polyphenols were found in the standard diet in terms of reducing inflammation and oxidative stress. However, in the HFHS diets, fish oils seem to be the one responsible for the positive effects found in the combined group.

Soybean polar lipids differently impact adipose tissue inflammation and the endotoxin transporters LBP and sCD14 in flaxseed vs. palm oil-rich diets

Manon Lecomte, et al., J. Nutritional Biochem. 43: 116-124, http://dx.doi.org/10.1016/j.jnutbio.2017.02.004.

Obesity and type 2 diabetes are nutritional pathologies, characterized by a subclinical inflammatory state. Endotoxins are now well recognized as an important factor implicated in the onset and maintain of this inflammatory state during fat digestion in high-fat diet. As a preventive strategy, lipid formulation could be optimized to limit these phenomena, notably regarding fatty acid profile and PL emulsifier content. Little is known about soybean polar lipid (SPL) consumption associated to oils rich in saturated FA vs. anti-inflammatory omega-3 FA such as α-linolenic acid on inflammation and metabolic endotoxemia. We then investigated in mice the effect of different synthetic diets enriched with two different oils, palm oil or flaxseed oil and containing or devoid of SPL on adipose tissue inflammation and endotoxin receptors. In both groups containing SPL, adipose tissue (WAT) increased compared with groups devoid of SPL and an induction of MCP-1 and LBP was observed in WAT. However, only the high-fat diet in which flaxseed oil was associated with SPL resulted in both higher WAT inflammation and higher circulating sCD14 in plasma. In conclusion, we have demonstrated that LPS transporters LBP and sCD14 and adi-



pose tissue inflammation can be modulated by SPL in high-fat diets differing in oil composition. Notably high-flaxseed oil diet exerts a beneficial metabolic impact, however blunted by PL addition. Our study suggests that nutritional strategies can be envisaged by optimizing dietary lipid sources in manufactured products, including fats/oils and polar lipid emulsifiers, in order to limit the inflammatory impact of palatable foods.

Significance of cooking oil to bioaccessibility of dichlorodiphenyltrichloroethanes (DDTs) and polybrominated diphenyl ethers (PBDEs) in raw and cooked fish: implications for human health risk

Mi, X.-B., et al., J. Agric. Food Chem. 65: 3268–3275, 2017, http://dx.doi.org/10.1021/acs.jafc.7b00505.

The present study examined the bioacessibility of DDTs and PBDEs in cooked fish (yellow grouper; *Epinephelus awoara*) with and without heating using the colon extended physiologically based extraction test. The bioaccessibility of DDTs and PBDEs increased from 60 and 26% in raw fish to 83 and 63%, respectively, after the addition of oil to raw fish. However, they decreased from 83 to 66% and from 63 to 40%, respectively, when oil-added fish were cooked. Human health risk assessment based on bioaccessible concentrations of DDTs and PBDEs in fish showed that the maximum allowable daily fish consumption rates decreased from 25, 59, and 86 g day⁻¹ to 22, 53, and 77 g day⁻¹ for children, youths, and adults, respectively, after fish were cooked with oil. These findings indicated that the significance of cooking oil to the bioaccessibility of DDTs and PBDEs in food should be considered in assessments of human health risk.

In-situ transesterification process for biodiesel production using spent coffee grounds from the instant coffee industry

Tuntiwiwattanapun, N., et al., Ind. Crops Prod. 102: 23–31, 2017 https://doi.org/10.1016/j.indcrop.2017.03.019.

Industrial spent coffee grounds (IND-SCG) are a potential non-edible biodiesel feedstock due to their abundant global supply and high oil content. In this study, an *in-situ* transesterification (in-situ TE) was developed and scaled up for IND-SCG biodiesel production. Several hurdles must be overcome, including the high acid value, and wide range in particle size of IND-SCG. Washing IND-SCG with methanol reduced its high acid value with negligible loss of oil. Size reduction (0.25–1.68 mm) and an increase of the reaction temperatures (30–60 °C) were found to improve the biodiesel yield significantly. The whole deacidified IND-SCG was processed at 50 °C; and a maximum biodiesel yield of 77% was achieved within 3 h. The process was successfully scaled up for processing 4 kg IND-SCG per batch with a yield comparable to the

30-g scale. The IND-SCG biodiesel met the ASTM biodiesel standard in terms of total glycerin, water content, kinematic viscosity and oxidative stability index (OSI), but its acid value exceeded the standard. A simple process modification using acidic water to neutralize alkaline catalyst during refining step, instead of strong acid, enabled the IND-SCG biodiesel to meet the standard for acid value. The oxidative stability index of the *in-situ* IND-SCG biodiesel was superior to that of the conventional process, probably due to the co-extraction of natural antioxidants.

An HPLC-CAD/fluorescence lipidomics platform using fluorescent fatty acids as metabolic tracers

Quinlivan, V.H., *et al.*, *J. Lipid Res.* 58: 1008–1020, 2017, https://doi.org/10.1194/jlr.D072918.

Fluorescent lipids are important tools for live imaging in cell culture and animal models, yet their metabolism has not been well-characterized. Here we describe a novel combined HPLC and LC-MS/MS method developed to characterize both total lipid profiles and the products of fluorescently labeled lipids. Using this approach, we found that lipids labeled with the fluorescent tags, 4,4-difluoro-5,7-dimethyl-4-bora-3a,4a-diaza-s-indacene (BODIPY FL), 4,4-difluoro-5-(2-thienyl)-4-bora-3a,4a-diaza-s-indacene [BODIPY(558/568)], and dipyrrometheneboron difluoride undecanoic acid (TopFluor) are all metabolized into varying arrays of polar and nonpolar fluorescent lipid products when they are fed to larval zebrafish. Quantitative metabolic labeling experiments performed in this system revealed significant effects of total dietary lipid composition on fluorescent lipid partitioning. We provide evidence that cholesterol metabolism in the intestine is important in determining the metabolic fates of dietary FAs. Using this method, we found that inhibitors of dietary cholesterol absorption and esterification both decreased incorporation of dietary fluorescent FAs into cholesterol esters (CEs), suggesting that CE synthesis in enterocytes is primarily responsive to the availability of dietary cholesterol. These results are the first to comprehensively characterize fluorescent FA metabolism and to demonstrate their utility as metabolic labeling reagents, effectively coupling quantitative biochemistry with live imaging studies.

Plasma trans-fatty-acid concentrations in fasting adults declined from NHANES 1999–2000 to 2009–2010

Vesper, H.W., et al., Am. J. Clin. Nutr. 105: 1063–1069, 2017, https://doi.org/10.3945/ajcn.116.141622.

The consumption of trans fatty acids (TFAs) is associated with an increased risk of cardiovascular disease, and reducing their consumption is a major public health objective. Food intake studies have provided estimates for TFA concentrations in the US population; however, there is a need for data on TFA blood concentrations in the population. The objective of this study was to determine plasma TFA concentrations in a nationally representative group of fasted adults in the US population in NHANES samples from

1999–2000 and 2009–2010. Four major TFAs [palmitelaidic acid (C16:1n-7t), trans vaccenic acid (C18:1n-7t), elaidic acid (C18:1n-9t), and linoelaidic acid (C18:2n-6t,9t)] were measured in plasma in 1613 subjects from NHANES 1999-2000 and 2462 subjects from NHANES 2009-2010 by gas chromatography-mass spectrometry. Geometric means and distribution percentiles were calculated for each TFA and their sum by age, sex, and race/ethnicity (non-Hispanic white, non-Hispanic black, Mexican American), and covariate-adjusted geometric means were computed by using a model that included these demographic and other dietary factors, as well as survey year and any significant interaction terms. These nationally representative data for the adult US population show that TFA concentrations were 54% lower in NHANES 2009-2010 than in NHANES 1999-2000. Covariate-adjusted geometric means for the sum of the 4 TFAs were 81.4 µmol/L (95% CI: 77.3, 85.6 μmol/L) and 37.8 μmol/L (95% CI: 36.4, 39.4 μmol/L) in NHANES 1999-2000 and 2009-2010, respectively. Even with the large decline in TFA concentrations, differences between demographic subgroups were comparable in the 2 surveys. The results indicate an overall reduction in TFA concentrations in the US population and provide a valuable baseline to evaluate the impact of the recent regulation categorizing TFAs as food additives.

Structure and physical properties of oleogels containing peanut oil and saturated fatty alcohols

Valoppi, F., et al., Eur. J. Lipid Sci. Technol. 119: 1–11, 2017, https://doi.org/10.1002/ejlt.201600252.

This study examined the capability of fatty alcohols with chain lengths from $C_{14}OH$ to $C_{22}OH$ to gel peanut oil. The gelation was achieved by crystallizing the samples at 5°C/min or 40°C/min. Results showed that minimum gelling concentration decreased as fatty alcohol chain length increased and it was higher for fast cooled samples than for the corresponding slow cooled ones. More than 7% of C₁₄OH was necessary to obtain a self-standing material highlighting its low capacity as oleogelator. Other oleogels were compared at 5% fatty alcohol concentration in peanut oil and oleogels containing C₁₆OH yielded the weakest system, with the lowest ability to retain oil. This was attributed to its higher solubility in oil as compared to other fatty alcohols as well as to the formation of larger crystal aggregates. As the fatty alcohol chain length increased, systems became stronger, displaying smaller crystal aggregates. For all cases, an increase in cooling rate lead to the formation of weaker gels with reduced capacity to entrap oil.

Enzymatic synthesis of steryl hydroxycinnamates and their antioxidant activity

Schär, A., et al., Eur. J. Lipid Sci. Technol. 119: 1600267, 2017, https://doi.org/10.1002/ejlt.201600267.

Steryl hydroxycinnamates are of increasing interest as they are antioxidant esters of phytosterols with potential cholesterol lowering properties. Apart from ferulates, also other plant steryl



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hydroxycinnamates have been identified in natural products. In this study hydroxycinnamic acid derivatives were ethylated enzymatically using Rhizomucor miehei lipase (yields from 16 to 97%), and transesterified by lipase from Candida rugosa to yield steryl hydroxycinnamates (yields from <LOQ to 55%). The influence of the structural differences between the hydroxycinnamic acid derivatives on the esterification yields was very different for the two lipases applied. Furthermore, the antioxidant activity of steryl and stearyl hydroxycinnamates was evaluated by DPPH radical scavenging activity and in two methyl linoleate systems. In bulk methyl linoleate, free sinapic acid showed the highest antioxidant activity over other sinapates, whereas in emulsified methyl linoleate, stearyl sinapate was highest. In conclusion, the enzymatic synthesis and antioxidant activity of steryl hydroxycinnamates is highly dependent on the acid structure.

Raman spectroscopy of fish oil capsules: polyunsaturated fatty acid quantitation plus detection of ethyl esters and oxidation

Killeen, D.P., et al., J. Agric. Food Chem. 65: 3551-3558, 2017, https://doi.org/10.1021/acs.jafc.7b00099.

Fish oils are the primary dietary source of ω -3 polyunsaturated fatty acids (PUFA), but these compounds are prone to oxidation, and commercial fish oil supplements sometimes contain less PUFA than claimed. These supplements are predominantly sold in softgel capsules. In this work, we show that Fourier transform (FT)–Raman spectra of fish oils (n = 5) and ω -3 PUFA concentrates (n = 6) can be acquired directly through intact softgel (gelatin) capsules. These spectra could be used to rapidly distinguish supplements containing ethyl esters from those containing triacylglyceride oils. Raman spectroscopy calibrated with partial least-squares regression against traditional fatty acid methyl ester analyses by gas chromatography-mass spectrometry could be used to rapidly and nondestructively quantitate PUFA and other fatty acid classes directly though capsules. We also show that FT-Raman spectroscopy can noninvasively detect oxidation with high sensitivity. Oils with peroxide values of as low as 10 mequiv kg⁻¹, which are on the cusp of falling outside of specification, could be readily distinguished from oils that were within specification (7 mequiv kg⁻¹).

Derivatization of castor oil-based estolide esters: preparation of epoxides and cyclic carbonates

Doll, K.M., et al., Ind. Crops Prod. 104: 269-277, 2017, https://doi.org/10.1016/j.indcrop.2017.04.061.

Estolides that are based on castor oil and oleic acid are versatile starting points for the production of industrial fluids with new properties. A variety of unsaturated estolides were derivatized by epoxidation with hydrogen peroxide. The epoxidized estolides were further modified using supercritical carbon dioxide and tetrabu-

tylammonium bromide to chemically incorporate carbon dioxide into the material yielding a 5-membered cyclic carbonate structure. These new epoxides and cyclic carbonates exhibited higher pour points, oxidation onset temperatures, and viscosities, compared to the corresponding unsaturated precursors. One derivative had a dynamic viscosity of ~9000 mPa s at 40°C, demonstrating potential for use in industrial applications.

Industrial Applications

Supercritical CO₂ extraction of rice bran oil—the technology, manufacture, and applications

Sookwong, P. and S. Mahatheeranont, J. Oleo Sci., online May 2017, http://doi.org/10.5650/jos.ess17019.

Rice bran is a good source of nutrients that have large amounts of phytochemicals and antioxidants. Conventional rice bran oil production requires many processes that may deteriorate and degrade these valuable substances. Supercritical CO₂ extraction is a green alternative method for producing rice bran oil. This work reviews production of rice bran oil by supercritical carbon dioxide (SC-CO₂) extraction. In addition, the usefulness and advantages of SC-CO₂ extracted rice bran oil for edible oil and health purpose is also described.

Continuous production of biodiesel from microalgae by extraction coupling with transesterification under supercritical conditions

Zhou, D., et al., Bioresour. Technol. 238: 609-615, 2017, https://doi.org/10.1016/j.biortech.2017.04.097

Raw material for biodiesel has been expanded from edible oil to non-edible oil. In this study, biodiesel continuous production for two kinds of microalgae Chrysophyta and Chlorella sp. was conducted. Coupling with the supercritical carbon dioxide extraction, the oil of microalgae was extracted firstly, and then sent to the downstream production of biodiesel. The residue after decompression can be reused as the material for pharmaceuticals and nutraceuticals. Results showed that the particle size of microalgae, temperature, pressure, molar ration of methanol to oil, flow of CO₂ and n-hexane all have effects on the yield of biodiesel. With the optimal operation conditions: 40 mesh algae, extraction temperature 60 °C, flow of n-hexane 0.4 ml/min, reaction temperature: 340°C, pressure: 18–20 MPa, CO₂ flow of 0.5 L/min, molar ration of methanol to oil 84:1, a yield of 56.31% was obtained for Chrysophyta, and 63.78% for Chlorella sp. due to the higher lipid content.

Performance of mechanical co-extraction of *Jatropha curcas*L. kernels with rapeseed, maize, or soybean with regard to oil recovery, press capacity, and product quality

Romuli, S., et al., Ind. Crops Prod. 104: 81–90, 2017, https://doi.org/10.1016/j.indcrop.2017.03.035.

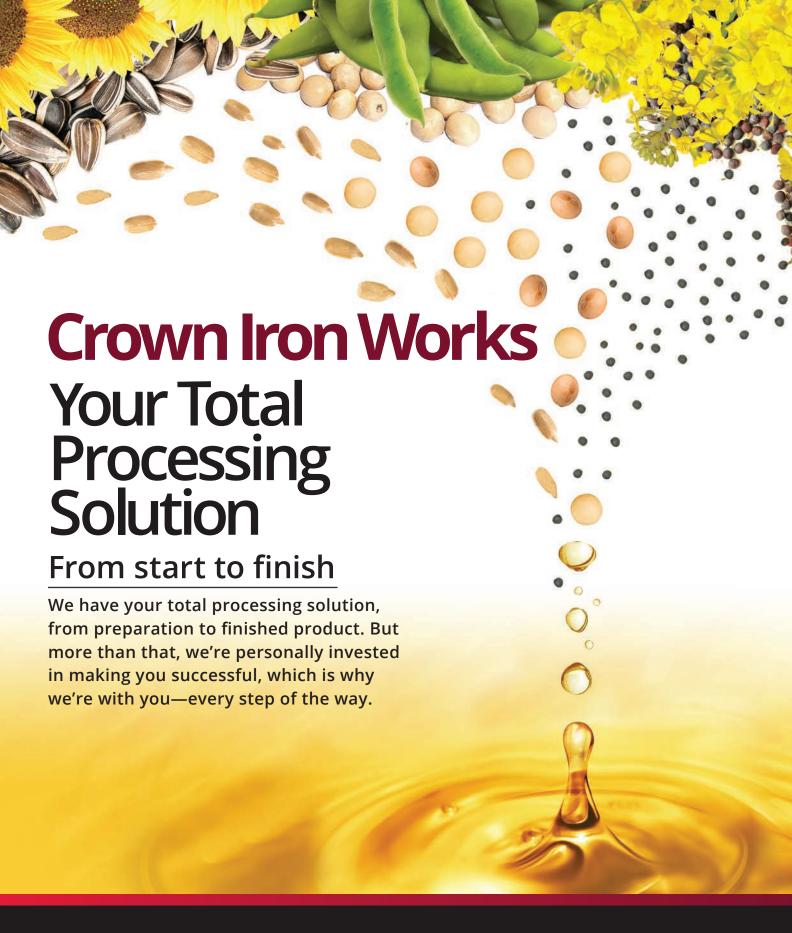
Jatropha curcas L. shelled seeds (kernels) have been gaining attention as protein source due to their high protein content. However, due to the soft texture, screw pressing the kernels resulted in a low performance, which was indicated by high residual oil content in the press cake. In order to overcome the low oil recovery from the extraction process, J. curcas kernels blended with rapeseed, maize, or soybean as additives were studied. The blending effects on the performance of mechanical oil extraction, along with the quality of oil and press cake were evaluated. To achieve the maximum oil recovery, blending ratio, nozzle diameter and screw speed were optimized using response surface methodology. Higher blending ratio showed significantly higher oil recovery (p < 0.05). Soybean blend exhibited highest oil recovery of 91.0%. Specific energy requirement and oil recovery were negatively correlated with throughput. Oil from maize blend generated the highest acid value and free fatty acid of 8.39 mg KOH/g and 4.19%, respectively. Carbon residue of the sedimented oil from all material blends fulfilled the standard threshold of German Institute for Standardization (DIN 51605). The highest press head temperature of 104.6 °C was observed from soybean blend. The ash content of the press cake from pure *J. curcas* kernels was lower than the other material blends. Press cake from soybean blend revealed low pepsin insoluble nitrogen and high crude protein content, pepsin plus trypsin digestibility and available lysine. Regarding the antinutritional factors, press cake from rapeseed blend showed highest phytate and trypsin inhibitors of 4.1% and 22.7 mg trypsin inhibited/100 mg sample, respectively. In terms of oil recovery and press cake quality, soybean appears to be the most suitable additive compared with rapeseed and maize.

Rice bran oil extraction using alcoholic solvents: physicochemical characterization of oil and protein fraction functionality

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Rice bran, an underutilized rice processing by-product, is a promising source for food and biodiesel oil production and can also be used to produce protein for use in human food products. The main objective of this study was to assess the feasibility of replacing hexane, which is traditionally used to extract vegetable oils, with safer solvents, i.e., ethanol and isopropanol, in rice bran oil (RBO) extraction. Thus, the effects of the solvent type on the physicochemical characteristics of the oil and defatted bran products were studied. The results showed that the presence of water in the alcoholic solvents negatively affected the oil extraction; however, using absolute solvents in single-stage batch extractions at 80°C resulted in oil yields of up to approximately 80%. The solvent water content and process temperature strongly impacted the properties of the protein fraction; the nitrogen solubility index (NSI) decreased from approximately 40% for the absolute solvents to 17 and 15% for the aqueous ethanol and isopropanol, respectively, when the extraction was performed at 80°C. More of the minor nutraceutical compounds were transferred from the oleaginous matrix to the oil by aqueous ethanol than by hexane, yielding RBO with 1.53% γ -oryzanol and 769 mg/kg tocotrienols. On the other hand, absolute isopropanol exhibited a higher tocopherol extraction capacity; RBO with a tocopherol content of 98.1 mg/kg was obtained with this solvent. Based on these results, short-chain alcohols are promising alternatives to the conventional extraction solvent, because they enable high-quality protein fractions and oils to be obtained and add value to the rice production chain.







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