FRYING WITH ALGAL OIL?

AND OTHER HOT ALGAE TRENDS

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Special report on algae

Algae are the subject of the first two articles of this issue of Inform. Readers receiving the mailed version of this issue will also receive “Industrial Algae Measurements, Version 6.0,” a report by the Algae Biomass Organization (ABO) that sets standards for measuring and comparing diverse algae industry operations. The ABO report, which is printed and distributed by AOCS Technical Services with permission from ABO, is also available as a free download.

A customized approach to frying oil

Which do you think would have a better oxidative stability index after 80 hours of frying over 10 days: high-oleic canola oil or customized high-stability algal oil? Learn what happened during an actual laboratory frying study.

Hottest trends in algae

Algae have been touted as the ultimate platform for fuels, chemicals, nutraceuticals, proteins—even cancer therapies. Here are 11 trends to watch.
2014 AOCS Fellows named

Systematic investigations in the antimicrobial efficacy of glycerine esters with fatty acids of different chain length
The antimicrobial efficacy of a homologous series of partial glycerides is compared in an attempt to understand the structure–response relationship.

General comparison of health claims with regard to food and supplement legislation frameworks in Europe, the United States, and Canada
This article compares and contrasts regulatory approaches to food and supplements in the European Union (EU), the United States, and Canada.

Is the art of HPLC method development dead?
Can the art of high-performance liquid chromatography (HPLC) method development survive high-efficiency columns and increased automation? An analytical method development chemist looks for signs of life.

Inform app and digital edition only:
• Krill sustainability
• US EPA to review 4,800 claims in 2014

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**Processing Contaminants in Edible Oils**  
**MCPD and Glycidyl Esters**  
Edited by Shaun MacMahon  
Product Code 272  
List: $155 • AOCS Member: $110  

This book serves as the single point of reference for the significant research related to monochloropropanediol (MCPD) and glycidyl esters in edible oils. These potentially harmful contaminants are formed during the industrial processing of food oils during deodorization. The mechanisms of formation for these contaminants, as well as research identifying possible precursor molecules are reviewed. Strategies which have been used successfully to decrease the concentrations of these contaminants in edible oils are discussed, including the removal of precursor molecules before processing, modifications of deodorization protocol, and approaches for the removal of these contaminants after the completion of processing. Analytical strategies for accurate detection and quantitation of MCPD and glycidyl esters are covered, along with current information on their toxicological properties.

**Trans Fats Replacement Solutions**  
Edited by Dharma R. Kodali  
Product code 271  
List: $195 • AOCS Member: $145  

Epidemiological studies have continued to increase awareness of how trans fats impact human nutrition and health. Because of the adverse effects, trans fats labeling regulations were introduced in 2006. Since then, the fats and oils industry and food product manufacturers have researched and implemented a number of novel, practical, and cost-effective solutions for replacing trans fats with alternate products. This book provides a comprehensive understanding of the trans fats chemistry, labeling regulations, and trans fat replacement technologies. It also deals with world-wide trends and scenarios in terms of regulations and trans fat replacement solutions.

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Today there is considerable research under way to determine the full potential of new algal oils in the global vegetable oil market. Custom profile microalgal oils produced using large-scale industrial fermentation processes—such as Solazyme Inc.’s Tailored™ brand oils—create new opportunities and resources for both oil producers and users. One application with great opportunities is in frying oils.

Usually frying is not considered an application for which algal oils are suitable. Often algal oils contain highly unsaturated fatty acids, such as docosahexaenoic acid (DHA) or eicosapentaenoic acid (EPA), that have poor heat stability due to oxidative sensitivity. Today algal oils are being developed using technology that allows specific fatty acid profiles to be targeted, thereby producing a wide array of functionalities. For instance, this technology can produce a monounsaturates-rich oil with very low polyunsaturates, opening the door to frying in algal oil.

In November 2012, ADM and Solazyme announced in a press release their entry into a collaboration to develop markets for algal food oils.

As part of this effort, algal oils with varied fatty acid profiles were examined in a laboratory frying study to explore...
approach to frying oil

the impact of fatty acid content on functionality. Algal oils with varied fatty acid profiles, used in the frying, were generated by Solazyme, Inc. with the company’s biotechnology platform, which uses heterotrophic microalgae to convert sugars into a wide range of customized triglycerides and bioproducts.

Solazyme is able to carefully control the environment of the microalgae inside large fermentation vessels, as opposed to pond-grown algae. Using large-scale indoor fermenters removes constraints of both seasonality and regional variability from oil production. The algal oils are produced very efficiently by the microalgae, with much more rapid growth cycles than traditional oil crops (days vs. months).

Solazyme’s technology enables production of oils that can mimic the oleic acid contents of high-stability vegetable oils, as exemplified by the “high-oleic algal oil” used in this fry study (80% oleic acid, see Fig. 1). It can also produce an oil with significant solids at low temperatures, suitable for par-frying applications, as seen in the “mid-oleic algal oil” examined (58% oleic acid/28% palmitic acid). Another profile the technology can offer is a high-oleic oil with an almost-zero content of the polyunsaturated fatty acids (PUFA) linoleic acid and linolenic acid 86% oleic acid/<0.1% PUFA). Such a fatty acid profile is not seen in today’s bioengineered oilseed crops and is only obtainable through Solazyme’s technology. This unique “high-stability algal oil” was also used in this laboratory frying study, with unprecedented results.

The three algal oils—mid-oleic algal oil (MOAO), high-oleic algal oil (HOAO), and high-stability algal oil (HSAO)—were evaluated alongside a high-oleic/low-linolenic canola oil (HOCanola) in a controlled laboratory setting. The HOCanola was used as an example of a commercial high-stability frying medium used in industry. All oils were refined, bleached and deodorized; and their initial quality was reflective of fresh oil. A standard bench-top laboratory frying protocol was used to stress the oil at frying temperatures for 80 hours in total over a 10-day period. During this time, a total of 46 kg of blanched, frozen potatoes were fried per

CONTINUED ON NEXT PAGE
fryer in even apportionments. The laboratory trial evaluated all normal frying reactions: hydrolysis, oxidation, and polymerization.

This study examined the oils “as is,” with no antifoam or added antioxidants. Initial oxidative stability index (OSI) values of fresh oil were 33.9 hours, 17.7 hours, 62.7 hours, and 18.4 hours at 110°C for MOAO, HOAO, HSAO, and HOCanola respectively (Fig. 2). As expected, after 10 days of frying, all oils, except for the HSAO, had less than 6 hours of inherent stability remaining by OSI measure. The HSAO at 10 days, with no additional protection, still had a 25 hour OSI value at 110°C. OSI is just one of the measurements that showed the impact of such low levels of PUFA in the HSAO frying oil. Another oxidation measurement that was performed was \( p \)-anisidine value (\( p \)-AV). Whereas OSI determinations survey inherent stability, \( p \)-AV measures secondary oxidation breakdown components. Increases in \( p \)-AV are expected in frying operations, with oil composition, product being fried, and frying procedures having an impact on how much the \( p \)-AV increases over time. When the \( p \)-AV for HSAO was measured at day 10 of frying, the value was 23. For comparison, the other algal oils were four times higher than the HSAO and the HOCanola was six times higher in \( p \)-AV, all treated under the same frying operations (Fig. 3).

Determination of percent total polar materials (%TPM) also showed the HSAO to have fewer breakdown components, another indication of a longer fry life vs. the oils with more polar materials. After 10 days, the %TPM for HSAO was 15%, compared to the other oils that ended the study at 38–42%TPM. During the study all oils except the HSAO foamed after 3–4 days of frying—by day 10 substantial foaming was observed. This is a normal result of frying in oils with no antifoam additive and substantial levels of breakdown components, and it is remarkable that HSAO did not show foaming under these conditions (Fig. 4). Nonetheless, even with the degree of foaming observed and the high levels of breakdown components in MOAO, HOAO, and HOCanola, no polymerized oil built up on the fryers for any of the oils tested. This preliminary examination demonstrates that algal oils do show great promise as frying oils—and one, HSAO, gives outstanding results even as a neat oil.

An additional aspect of the laboratory frying study was examination of the fried potatoes for flavor. The objective was to gain a basic understanding of what differences might exist. A panel of evaluators familiar with oil sensory characteristics tasted hot, fried potatoes throughout the 10-day period of frying. Samples were presented in blind, random order, although the panel members were aware of the oils used in the study. Feedback was that the algal oils did not have a consistent differentiation from the vegetable oil control. Development of fried flavor and inherent flavors was shown to vary at different time points, as would be expected with dissimilarities in fatty acid profiles. However, in this preliminary examination, sensory differences observed were not thought to be any more significant than what is detected with inherent differences seen in oilseeds.

Frying in algal oil may seem to be a novel concept, but with new developments in technology, algal oils have been customized to provide properties having parity or enhanced performance compared to a commercial high-oleic vegetable oil. The full potential is being determined as research continues. With Solazyme’s Tailored™ technology
permitting custom profiling to application needs, one can ask: What would be an ideal fatty acid profile for an oil specifically designed for frying flavor and stability? What are the opportunities for extending shelf life in finished goods when polyunsaturates can be lowered to trace amounts? Is there a prospect to remove waste oil from a frying operation entirely? With these possibilities on the horizon, the future is exciting for custom profile algal oils.

Michelle Peitz works in technical sales at ADM Oils in Decatur, Illinois, USA. She can be contacted at Michelle.Peitz@ADM.com.

Risha Bond is associate director, emerging business at Solazyme Inc., South San Francisco, California, USA. She can be reached at rbond@solazyme.com.
Algae has been touted as the ultimate platform for fuels, chemicals, nutraceuticals, proteins—even cancer therapies, yet commercialization has been slow despite considerable investment.

Recently, a cluster of research projects and proto-companies have been tackling the real-world challenges of yield, harvesting, dewatering, and application development.

Here are the top trends to watch.

**BIG OIL, LITTLE ALGAE**

In late February 2014, algae observers were startled to learn that Reliance Industrial Investments, the Indian oil
holding company, placed a $2.4 million purchase order for Algae.Tec algae production technology (http://tinyurl.com/Rel-Ind-Algae) as a follow-up to its initial investment in January 2014 of AU$1.5 million, with additional investments of AU$1.2 million over the next two years. The purchase order for Algae.Tec modules will be supplied and completed over approximately the next nine months. The Algae.Tec solution is less than one-tenth the land footprint of pond growth options, while its enclosed module system is designed to deliver the highest yield of algae per hectare and solves the problem of food-producing land being turned over for biofuel production.

Overall, it’s Reliance’s third algae investment. A Credit Suisse report on the company, (see page eight of the report at http://tinyurl.com/CreditSuisse-algae) revealed in 2012 that Reliance had invested a total of $116 million in two United States companies, $93.5 million in Algenol and $22.5 million in Aurora Algae.

But there’s more algae activity stirring in the world of Big Oil. In December 2013, Sapphire Energy and Phillips 66 announced a strategic joint development agreement to work together to collect and analyze data from co-processing of algae and conventional crude oil into fuels, and to complete fuel certifications to ready Sapphire energy’s renewable crude oil for wide-scale oil refining.

Under the agreement the companies will expand Sapphire Energy’s current testing programs to further validate that Green Crude can be refined in traditional refineries and meet all of the Environmental Protection Agency’s (EPA) certification requirements under the Clean Air Act. This includes determining the optimal operating conditions for processing algae crude oil into American Society for Testing and Materials-certified diesel, gasoline, and jet fuel. Once the study is finished, the companies will work together to complete the EPA certification process to register a new fuel product entering the market. Sapphire Energy is now producing crude oil daily from algae biomass cultivated and harvested at the company’s Green Crude Farm, located in Columbus, New Mexico, USA.

Meanwhile, let’s not forget the Synthetic Genomics (SGI–ExxonMobil relationship, which debuted in spectacular fashion with a $500 million initial spending target in 2009. In May 2013, SGI announced a new co-funded research agreement with ExxonMobil to develop algae biofuels. The new agreement is a basic science research program that focuses on developing algal strains with significantly improved production characteristics by employing synthetic genomic science and technology. Financial details of the agreement were not disclosed. Last year, ExxonMobil CEO Rex Tillerson said in a March 2013 interview on PBS (the US Public Broadcasting System), “We’ve come to understand some limits of that technology, or limits as we understand it today, which doesn’t mean it’s limited forever.” The venture is “probably further” than 25 years away from successfully developing fuels, according to Tillerson.

The last public update on ExxonMobil’s algae efforts appeared on the company website in 2011 (http://tinyurl.com/ExxonMobil-April2011).

**MAKING MORE BETTER**

Algae is renowned for its production potential—at all, the mass can double in as little as 24 hours—meaning that it could dwarf the productivity of terrestrial plants. But translating potential into industrial-scale “business as usual” hasn’t been a joyride.

Hence it was big news when, in March 2013, Algenol confirmed that the company had exceeded production rates of 9,000 gallons of ethanol per acre (84,000 liters per hectare) per year—and company CEO Paul Woods said, “I fully expect our talented scientific team to achieve sustained production rates above 10,000 by the end of this year” (http://tinyurl.com/Algenol-Woods-algae). Last September, in the opening plenary session at the Algae Biomass Summit, Woods revealed that the company, at its 4-acre (1.7 hectare), outdoor Process development unit in Lee County, Florida, USA, had achieved continuous production of ethanol at the 7,000 gallons per acre level.

It was a substantial increase over the company’s original target of 6,000 gallons per acre, and it was achieved in outdoor operation under normal operating conditions. With the news, Woods confirmed that the company, after completing major construction activities at its integrated pilot scale biorefinery in 2012, has fully shifted focus to demonstrating the commercial viability of Direct to Ethanol technology at its pilot facility and identifying sites for commercial projects to begin in 2014.

**SCALE**

Now, Solazyme doesn’t like to think of itself as an algae company any more than Budweiser wants to be known as CONTINUED ON NEXT PAGE
Scientists are researching the production of oil-producing algae, as well as the feasibility of commercial-scale biofuel production based on microbes discovered in Yellowstone National Park.

a yeast company—both prefer to define themselves by their products rather than around the details of their fermentation technology. Nevertheless, Solazyme does use algae fermentation—and they have been getting to massive scale.

In January 2014, the company announced that commercial operations have commenced at both Archer Daniels Midland Company’s Clinton, Iowa (USA), facility, and the downstream companion facility operated by American Natural Products in Galva, Iowa. Volumes shipped to Brazil are being utilized for market development activity in advance of the opening of the Solazyme Bunge Renewable Oils’ Moema facility. As stated previously, production at the ADM and ANP facilities is expected to ramp to a nameplate capacity of 20,000 metric tons per year (MT/yr) within 12–18 months, with targeted potential expansion to 100,000 MT/yr in subsequent years.

The company noted, in a release, that “truckloads of product are now shipping from the Iowa operations for use in applications including lubricants, metalworking and home and personal care. These shipments are being made pursuant to multiple supply agreements as well as spot purchases, and include reorder.s” (http://solazyme.com/media/2014-01-30). Highlighting the flexibility of Solazyme’s technology platform, Solazyme, ADM, and ANP have successfully manufactured three distinct and unique tailored oil products at the facilities, and products are currently being sold and distributed in both the United States and Brazil.

The Clinton news is a follow-through from the news in December 2012 that Solazyme had announced the completion of multiple initial fermentations in 500,000 liter fermenters at ADM’s Clinton, Iowa facility—about four times the scale of the vessels in Solazyme’s own Peoria, Illinois, facility. That set of runs broke through the ferment wall: namely that, hitherto, no next-generation producer had successfully achieved linear scaleup in 500,000 liter (or larger) fermenters. It’s simply impossible for fermentation-based technologies to affordably produce fuels and chemicals in small fermentation tanks—it’s way too much capex (capital expenditure), too much opex (operating expenses) to produce, say, 10,000 liters at a time.

Companies also at scale in fermentation? DSM and Alltech.

BRING ON THE APPS

“We’re like the iPhone,” said Heliae CEO Dan Simon, “and companies like Triton are bringing forward the apps” (http://tinyurl.com/Heliae-Triton-apps). “We may well see companies like Heliae selling licenses for its production technology to customers who in turn license and introduce apps, to generate fuels, chemicals, nutraceuticals, as well as complex proteins, enzymes, and other biologics that are cost effective and have immediate applications in agricultural, pharmaceutical, and other retail markets.

Progress with the “iPhone” is becoming pretty clear, with Heliae booking $4.2 million in sales already in 2014 for their raceway-based algae growing technology, after recently completing a $13 million demonstration plant. Although the company has equipment onsite to develop fuels from the algae, and the company has previously turned algae into jet fuel onsite, Heliae is focusing on the growing side of the equation. The company brought in more than $1 million in revenue last year.

So, what are the hot apps?

Proteins. Almost 10 years ago, out of Dr. Steve Mayfield’s lab at Scripps Research Institute and later at the University of California at San Diego, a series of discoveries made it possible, for the first time, that algae could be used as a platform for synthetic biology and genetic innovation—just as yeast and E. coli had been used for years. Now, there are subtle and microscopic reasons why algae could be a platform to rival E. coli—some related to superior folding (proteins that don’t fold properly are generally inactive, or can become toxic or change their function). Mayfield’s technology ultimately led to the formation of Rincon Pharmaceuticals in 2004 to pursue commercialization. Sapphire ultimately acquired it, and pursued proteins as a side project.

By 2010, Mayfield was reporting in Plant Biotechnology Journal that seven diverse human therapeutic proteins could be produced in Chlamydomonas reinhardtii, a green alga used widely in biology laboratories as a genetic model organism, with production costs of $0.60-per-gram protein (http://tinyurl.com/Mayfield-PBJ). Even then, that was “about the same cost estimates for the least expensive protein expression systems presently available, and con-
considerably cheaper than mammalian cell culture,” Mayfield and his team reported at the time.

With expected improvements in the ability to express proteins in algae, “and the continued reduction in algal biomass cost associated with the large scale efforts to use algae for biofuel production, we anticipate at least a 10-fold reduction in the costs over the next few years, which should make algal protein production the least expensive platform available.”

Ultimately, some of the old Rincon intellectual property was spun out of Sapphire and back to Mayfield and Jason Pyle, who cofounded Sapphire Energy with Mayfield and then founded Triton in 2012. Triton’s platform is known as PhycoLogix, and uses algae to produce compounds that other organisms cannot, that can be safely consumed without modification, and that can be cultivated at large scale inexpensively. Heliae invested $5 million late in 2013.

**Nutraceuticals.** In September 2013 Algaeon announced the signing of a multi-year, multi-million dollar supply agreement with Valensa International to provide high-value “condition specific” nutraceuticals to the marketplace (http://tinyurl.com/Algaeon-Valensa). Algaeon, in cooperation with Valensa, is using its extensive knowledge of algae production to bring a new level of efficiency and quality for algae-based ingredient supply to the nutraceutical market. Algaeon will develop manufacturing processes and technology while Valensa will produce finished form condition specific products that will be sold to marketers with recognized brands.

**DHA.** This is the secret ingredient in fish oil or omega-3s sold at your pharmacy for its health benefit. In late 2011, Sofiprotéol, the industrial and financial arm of the French plant oils and proteins sector, established a joint venture with Fermentalg to “industrialize, produce and market oils from microalgae that are rich in oils from the omega-3 family (EPA-DHA) [eicosapentaenoic acid-docosahexaenoic acid]”—with a goal of ensuring “the development of its patented process until the early scaleup phases of its technology.” Sofiprotéol is providing the bulk of financing. In early 2013, it was training its focus on “omega-3 fatty acids, coloring agents, antioxidants and biopolymers, etc.” according to an interview in *Algae Industry Magazine.*

A signature Series C capital raise announced on September 13, 2013—which netted $16 million and attracted existing investors ACE Management, Demeter Partners, Emertec Gestion, and Picoty Algo, plus new investors IRDI and Viveris—was in support of a focus on “industrial scaleup and commercialization” of its microalgae production for use in “animal feed, biofuels, cosmetics, food, health, and specialty chemicals.”

Also hovering around the DHA scene is Alltech. Its major move into microalgae dates to the acquisition of a former Martek Bioscience plant in Winchester, Kentucky, USA. The plant, which contains 1.26 million liters of fer-
mplementation capacity on a 17 acre (7 hectare) campus, had been originally built as a yeast production plant, then produced vitamin B2 for Coors, and ultimately was acquired by Martek, before Alltech bought the plant for $14 million. Alltech has publicly discussed a $200 million investment in transforming the plant into a heterotrophic algae production facility—with a focus on production of DHA. The renovated plant opened in April 2011. Right now, the plant can produce 20 tons (18 metric tons) of algae per 11-day campaign—a capacity of roughly 1,800 tons per year at current productivity. At this stage, 1,800 tons of algae would have a theoretical maximum of 176 tons (160 metric tons) of DHA production.

Hybrid platforms. In 2012, Cellana announced the launch of its ReNew brand and ReNew Omega-3 line of algae-based products. The ReNew brand was developed to meet the growing demand for more sustainable omega-3 human health products, animal nutrition products, and biofuel feedstocks. The ReNew portfolio comprises four main product categories: ReNew Omega-3, including both ReNewEPA and ReNewDHA; ReNew Feed as a nutritional product for the animal feed market; ReNew Fuel as an algae-based bio-crude, particularly for jet fuels for commercial and military aircraft; and ReNew Algae, available in bulk for customers to apply their own extraction technologies and develop customized solutions within these application areas. The ReNew product line is derived from Cellana’s scalable, sustainable, and patented ALDuo algae production technology. Cellana’s 6-acre (2.4-hectare) Kona Demonstration Facility on Hawaii’s Big Island has produced more than 9 tons (8 metric tons) of algal biomass for commercial testing. At this time, Cellana is raising money for a commercial-scale facility.

Another company with multiple product lines is Aurora Algae. Aurora burst onto the scene in June 2008 with the announcement that it had raised $20 million in series A financing from Oak Investment Partners, Noventi, and Gabriel Venture Partners. The company completed an 18-month pilot in early 2009 and said that it has more than doubled the productivity of its selected strains. By August 2013 Aurora said it was looking to move its planned commercial-scale project algae project to Geraldton, Australia, where it already has a test project. It has stated that it needs to expand from a 6-acre system to 250 acres to be commercially successful.

The company’s key technology—an optimized strain of saltwater algae that is lighter in color than wild-type algae—allows deeper penetration of sunlight, thereby extending the zone for algae reproduction and increasing yield. That’s the Aurora “secret sauce”: to outcompete, as a form of crop protection, simply to grow too fast for predators and competitors to get a foothold. The four product lines are: (i) A2 Omega-3—a family of omega-3 oils aimed at the nutraceutical and pharmaceutical markets. The first offering in this family, A2 EPA Pure, will make the benefits of EPA available to a broader market since it is derived from an allergen-free, vegetarian source. (ii) A2 Feed is a family of protein-rich algal grains for the animal and aquaculture markets; (iii) A2 Fuel is a family of biomass and biodiesel applications; and (iv) A2 Protein is a family of protein-rich applications.

NO MORE VENTING MONEY, OR CO2
Then there’s the flue stack—you might as well call the Money Stack, because of all the money that is vented every time a company vents CO2. One of the most interesting plays in algae is to use it as a means of monetizing CO2, turning it from a headache into an opportunity.

BioProcess Algae (Middletown, Rhode Island, USA) is helping Green Plains Renewable Energy (headquartered in Omaha, Nebraska, USA) to scale up its CO2-based algae experiment into a commercial-scale add-on facility (http://www.bioprocessalgae.com/news/101/). What started out as a lab test that grew and grew until it reached 400 foot (120 meter)-long greenhouses has led to omega-3 production as well as high-value pellets and feed selling from $1,500 to $10,000 a ton, compared to $200 a ton for corn. Omega-3 activity? The company in 2012 announced a commercial supply agreement for EPA-rich omega-3 oils with KD-Pharma for use in concentrated EPA products for nutritional and/or pharmaceutical applications.

EXTREMOPHILES
New algae—or rather, undiscovered or otherwise underappreciated algae—well, algae companies and research organizations have their scouts traveling even more obscure paths than a major league baseball scout.

One of the hottest areas for development—extremophiles. Organisms that love unusual heat or pressure conditions that make them very robust in algae growth systems (for example, algae that can tolerate hot temperatures can outcompete other swimmers in the pond). So, consider this: scientists are researching the production of oil-producing algae, as well the feasibility of commercial-scale biofuel production based on microbes discovered in Yellowstone National Park (http://tinyurl.com/Montana-research).

Part of a multi-institutional project funded by a grant through the Sustainable Energy Pathways program at the National Science Foundation, it is one of many algal biofuel research projects at Montana State University. The project, which also includes researchers with the University of North Carolina and the University of Toledo, is part of a federal effort to tackle some of the fundamental problems in developing enough biofuels fuels to provide up to 50% of the nation’s transportation fuel. The US Department of Energy is funding the project.

THE PYROMANIAx
Hitherto, most algae systems have relied on extraction. That is, grow the algae, dewater them, then extract the valuable oils or proteins. But a number of ventures, such
as Sapphire Energy and Algenol, are looking to pyrolyze the whole algae or algae residues (http://tinyurl.com/WholeAlgae-Byproducts).

In Washington state, engineers have created a continuous chemical process that produces useful crude oil minutes after they pour in harvested algae. The research by engineers at the US Department of Energy’s Pacific Northwest National Laboratory (PNNL) was reported recently in the journal Algal Research (http://dx.doi.org/10.1016/j.algal.2013.08.005). In the PNNL process, a slurry of wet algae is pumped into the front end of a chemical reactor. Once the system is up and running, out comes crude oil in less than an hour, along with water and a by-product stream of material containing phosphorus that can be recycled to grow more algae.

CO2 services at Sapphire are provided by Linde, who is tasked with figuring out the best way to scale—through co-locating with a CO2 emitter or tapping in to the CO2 pipeline system.

With additional conventional refining, the crude algae oil is converted into aviation fuel, gasoline, or diesel fuel. And the wastewater is processed further, yielding burnable gas and substances such as potassium and nitrogen, which, along with the cleansed water, can also be recycled to grow more algae. The system runs at around 350°C at a pressure of around 3,000 PSI (20 MPa), combining processes known as hydrothermal liquefaction and catalytic hydrothermal gasification. Cautionary note? The PNNL system runs continuously, processing about 1.5 liters of algae slurry in the research reactor per hour. So, it’s pre-pilot. And it is not going to be cheap to build out, at scale, a system that requires 350°C and 20MPa.

Along those lines, Sapphire and Linde announced last year that they will expand their partnership to commercialize a new industrial-scale conversion technology needed to upgrade algae biomass into crude oil (http://tinyurl.com/SapphireLinde-pilotscale). Together, the companies will refine the hydrothermal treatment process developed and operated today by Sapphire Energy at pilot scale. In addition, they will jointly license and market the technology into an expanded list of industries, including algae, municipal solid waste, and farm waste, in order to upgrade other biomass sources into energy. The agreement spans a minimum of five years through the development of Sapphire Energy’s first commercial scale, algae-to-energy production facility.

ONE WORD: PLASTICS

What about new materials? Plastics have been promising. In December, the Institute for Plastic Technology in Valencia, Spain, profiled its European Commission program looking into various materials that can be produced from algae to create adhesives, paints, and dyes using a technology developed by Alicante-based Biofuel Sys-
tems (http://tinyurl.com/PlasticTechnology-Valencia). The 42-month research program includes 13 different companies. The first stage of the project will be to identify fast-growing algae to later be processed.

**SCRUBBERS**

Then, there are algae’s abilities not only as a product, but as a platform for scrubbing wastewater—which has been a use for algae for years. But recently, algae’s abilities to scrub out highly toxic materials have been put to the test.

In January 2014, a research group led by Yoshihiro Shiraiwa of the University of Tsukuba, Japan, identified 17 strains of microalgae as well as aquatic plants that are able to efficiently remove radioactive cesium, iodine, and strontium (http://tinyurl.com/algae-isotopes) from the environment were identified. The findings add to existing options that could help to decrease radioisotope pollution in the Fukushima area. Such measures are of utmost importance, because a large quantity of radioactivity has been released. The researchers noted that further studies are needed on the mass cultivation and efficient coagulation and sedimentation of these algal strains before their findings can be put into practice.

Plus, there are tools to keep the algae ponds free of pests, predators, competitors, and the like. Along those lines, in November 2013, OriginOil announced that academic testing verified its new Algae Screen growth optimizer effectively controls bacterial and microscopic predators in commercial algae production, helping to promote high rates of cultivation of the most valuable species (http://tinyurl.com/SCSU-AlgaeScreen). “Initial test results saw a dramatic drop in contaminant load while the culture still maintained target cell integrity,” said Matt L. Julius of the Department of Biological Sciences at St. Cloud State University, Minnesota, USA. “This is one technology that will change the industry once it is fully validated.”

**KELP IS ON THE WAY**

What about macroalgae, also known as kelp? In California, researchers from Bio Architecture Lab Inc. (BAL) published in the journal *Nature* an alginate monomer transporter they discovered that will help to significantly boost the efficiency of cellulosic ethanol production by a synthetic yeast from brown macroalgae sugars (http://dx.doi.org/10.1038/nature12771). Using fermentation, the researchers were able to achieve 83% theoretical yield from the sugars.

In 2010, BAL and Norway’s Statoil announced a wide-ranging strategic partnership for the production of renewable, sustainable, and low-cost ethanol derived from macroalgae grown off the coast of Norway (http://tinyurl.com/Statoil-BAL-macroalgae). Statoil will fund BAL’s research and development and demonstration projects, and if successful, will also fund the commercialization of BAL’s technology in Norway and elsewhere in Europe. During the initial phase of the partnership, BAL is responsible for developing the technology and process to convert Norwegian seaweed into ethanol. Statoil is responsible for developing and managing the seaweed aquafarming operations, with consultation from BAL, which already has established aquafarming operations in Chile. Upon the successful achievement of key milestones, Statoil and BAL would develop a demonstration scale facility in Norway.

**BUILDING BETTER ALGAE**

Final trend? Building better algae through genetic enhancement. That work has been mostly undertaken by Sapphire, which has been engaged in some brute force biology to get the industry going.

In late 2012, for example, Sapphire Energy and the Institute for Systems Biology (Seattle, Washington, USA) announced a strategic partnership to significantly increase oil yield and improve resistance to crop predation and environmental factors in order to further the advancement of commercialized algae biofuel production (http://tinyurl.com/ISB-Sapphire-yield). “Sapphire is dealing with one of the most complicated problems known to humans: how to make fuel from a renewable resource,” said Nitin Baliga, director of Integrative Biology at ISB. “Together, we have complementary expertise that will allow us to understand, reverse engineer, and rationally alter the gene networks for fuel production in algae.”

But the effort continues elsewhere. Last November in Tennessee, researchers at Vanderbilt University reported that when the biological clocks of cyanobacteria were stopped in their daylight setting, the amount of several biomolecules that they were genetically altered to produce increased by as much as 700% when grown in constant light (http://tinyurl.com/Daylight-cyanobacteria). “We have shown that manipulating cyanobacteria’s clock genes can increase its production of commercially valuable biomolecules,” said Carl Johnson, Stevenson Professor of Biological Sciences at Vanderbilt University.

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**Jim Lane is editor and publisher of Biofuels Digest. He can be contacted at jlane@biofuelsdigest.com.**

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Call for Nominations

Stephen S. Chang Award

The Award

The Stephen S. Chang Award recognizes a scientist, technologist, or engineer who has made significant and distinguished accomplishments in basic research that must have been utilized by industries for the development or improvement of products related to lipids. The awardee may be recognized for either one major breakthrough or an accumulation of publications.

A prospective recipient must agree to be present for acceptance of the award and to deliver an award address at the 106th AOCS Annual Meeting & Expo. The award is made without regard for national origin, place of residence, race, color, creed, or gender.

Nomination Procedures

Nominations for the 2015 award must be submitted before October 15, 2014.

The suggestions listed below may be helpful to nominators in addressing the mandatory criteria of industrial utilization.

1. Documentation of the application of research
   a. Patents received, licensing arrangements
   b. Specific examples of industrial use

2. Documentation for the development or improvement of products related to lipids
   a. Listing of new products, manufacturers, sales history
   b. Manufacturers’ testimonials regarding product improvement resulting from their direct utilization of the basic research in specific products with comparative figures on sales or consumer acceptance

The nomination must include a letter from the nominator, at least three supporting letters, the nominee’s curriculum vitae, and a list of major relevant publications, including patents.

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The spotlight is on AOCS Platinum, as of April 1, 2014

Platinum

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Valicon
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AOCS provides the resources.
About 24 years ago as a young assistant professor at Iowa State University I was looking to develop my academic career in the field of Food Science and Technology. I had spent five years in industry as a Product Development Scientist and was looking to grow my career in academics. Dr. Larry Johnson suggested that I attend a meeting of an organization called AOCS. To be frank with you I had never heard of AOCS and he explained to me about the organization. Not the information that I could find on the website about the work in Fats, Oils, Soaps, Detergents and related materials, but other strengths that would make me want to be a part of the organization. The excellent scientists and professionals who were the international leaders in their respective fields. The strong networking between industry, academia and government to identify and solve issues related to the fats, oils, and soaps and detergents industries. And the strong camaraderie and friendships between members who assist each other to be more successful in our respective professional endeavors. After the first meeting I found that he had understated this and from that first meeting I knew that I wanted to not only attend meetings but to also have an active role in this organization.

**Want to be more involved and help lead AOCS?**

Serving in leadership positions with AOCS is your opportunity to be instrumental in the future direction of the Society.

Contact us today! [www.aocs.org/leadership](http://www.aocs.org/leadership)
In early April 2014, the US Food and Drug Administration released the first final rule under the Food Safety and Modernization Act of 2010 (FSMA). The regulation is on record access requirements for food firms and includes a guidance document for industry. See http://tinyurl.com/FNSMA-Record-Rule for the final rule and http://tinyurl.com/FNSMA-Final-Records for the guidance document.

State-owned China National Cereals, Oils and Foodstuffs Corp. (COFCO) said in early April 2014 that it will pay $1.5 billion in cash for a 51% stake in the agribusiness operations of Hong Kong-based Noble Group Ltd., according to the Bloomberg news service (see http://tinyurl.com/COFCO-Noble). The move follows COFCO’s purchase in February of a 51% stake in grain trader Nidera BV of the Netherlands aimed at gaining direct access to South American oilseed and grain supplies.

AAK (Malmö, Sweden) said in a statement that it has agreed to acquire the oils and fats business of CSM Bene-lux NV in Merksem, Belgium. CSM is a bakery fats, margarine, and pumped shortenings supplier to the bakery markets in Belgium, the Netherlands, and France. The acquisition includes the CSM factory in Merksem, the sales force, and a bakery innovation center.

JoAnn Rutkowski of the former Thumb Oilseed Producers Cooperative in Ubly, Michigan, USA, was indicted in April 2014 for introducing soy flour containing Salmonella into interstate commerce between December 2010 and January 2011, according to the Huron Daily Tribune (http://tinyurl.com/Thumb-Oil). Further, she was found to have done so “with the intent to defraud and mislead.” The cooperative produced and sold soy flour, soy grits, and refined soy oil. After two US Food and Drug Administration recalls of its products (in February 2010 and October 2011) for potential contamination with Salmonella and a subsequent $7-million lawsuit brought by Ralcorp Holdings, the cooperative filed for Chapter 11 bankruptcy in March 2012. Rutkowski’s trial is expected begin on July 8.

### NEWS & NOTEWORTHY

#### Annual soy innovation contest results

A group of Purdue University students who created a soy-based filament for 3D printing won the top prize in the annual Student Soybean Product Innovation Contest. A record 15 teams completed projects in the competition, which is celebrating its 20th anniversary.

The S3D Innovations team developed Filasoy, a next-generation 3D printing material. Filasoy replaces petroleum-based plastic with a low-energy, low-temperature, renewable, and recyclable filament. It retains similar properties found in a bioplastic with an added “green” twist: It allows printing without waste.

The runner-up team, Soots, produced a 100% organic leather boot conditioner and polish by the same name. The product comes in two forms: One, made from soybean oil and beeswax, is a thick, more solid polish for genuine leather items such as boots and reins and also serves as a waterproofing agent. The second product is a much lighter conditioner, in the form of a spray, that can be used on faux leather items. It is used more for cleaning and improving appearance than waterproofing.

#### Food label changes coming to EU and US

Regulatory authorities in both the European Union (EU) and United States (US) are in the process of implementing new regulations on the labeling of processed foods.

In Europe, Regulation 1169/2011 (see http://tinyurl.com/Labeling-EU) comes into force on December 13, 2014. Also known as
the Food Information to Consumers Regulation (FIR), the new regulation requires the following:

- Annex IIIA of Directive 2000/13/EC lists all allergenic ingredients and substances that must be labelled (see http://tinyurl.com/EU-Allergens). Fully refined soybean oil is specifically exempted from allergen labeling.

- All food product labels must contain the name of the product, a list of all ingredients in descending order of weight, the amount of food in the container or package, and the use-by date.

- Nutrition labeling is voluntary. However, if a nutrition claim is made on a product label or in advertising, nutrition labeling becomes compulsory. See http://preview.tinyurl.com/CBI-labeling for a complete discussion of all aspects of the new regulation.

- The labeling of genetically modified foods, as delineated in Regulation (EC) 1829/2003 and Regulation (EC) 1830/2003 continue to apply under the new regulation. For further requirements related to traceability and authorization, see http://www.gmo-compass.org/eng/regulation/labelling.

Three tips for complying with the FIR—as provided by Stephen Spice, head of regulatory affairs for research firm Campden BRI (Chipping Campden, Gloucestershire, UK)—include acting quickly to book a printer to be sure new labels are ready by the December 2014 deadline. Further, Spice suggested firms look at their labeling policies to make sure they comply with the new regulation. Finally, he stressed that all employees must be trained on the new rules—employees both within a firm and throughout the supply chain.

**US TO GET UPDATED LABELS**

Food labels will also change in the United States, thanks to two proposed rules published in late February 2014 in the Federal Register by the US Food and Drug Administration (FDA). The proposed regulations constitute the first major revisions to US nutrition labeling since 1994.

The comment period on the proposed regulations ended on June 2, 2014. (See http://tinyurl.com/comments-label and http://tinyurl.com/comments-serving-size to access the two dockets.) The FDA has said that it plans to issue final rules by 2015 and then give industry two years to phase in new labels. The agency estimates the changes will cost industry approximately $2 billion.

“For 20 years consumers have come to rely on the iconic nutrition label to help them make [more healthful] food choices,” said FDA Commissioner Margaret A. Hamburg. “To remain relevant, the FDA’s newly proposed Nutrition Facts label incorporates the latest in nutrition science as more has been learned about the connection between what we eat and the development of serious chronic diseases . . . .”

**CURRENT VS. PROPOSED LABELS**

Key aspects of the proposed changes include:

- **Removal of “Calories from Fat.”** The proposed regulations will continue to require “Total Fat,” “Saturated Fat,” and “Trans Fat” on the label. However, “Calories from Fat” would be removed because FDA believes the type of fat is more important than the amount.

- **Updated serving size requirements.** Under the new rules, serving sizes must be based on what people actually eat, not what people “should” be eating. Thus, food makers will have to provide calorie and nutrition information for the whole package of certain food products that could be consumed in one sitting such as bottled soda and ice cream.

- **Increased prominence of serving size and calorie counts.**

- **Dual-column labels.** This will indicate both “per serving” and “per package” calorie and nutrition information for larger packages that could be consumed in one sitting or multiple sittings.

- **Declaration of sugars.** The proposed rule would require declaration of “Added sugars,” indented under “Sugars,” to help consumers understand how much sugar is naturally occurring and how much has been added to the product.

CONTINUED ON PAGE 360
Compared with the first quarter of 2013, sales of certified sustainable palm oil saw a 49% increase and sales of GreenPalm Certificates rose by 54%, according to the Roundtable on Sustainable Palm Oil (RSPO). The current estimated annual production capacity of RSPO-certified sustainable palm oil is 9.7 million metric tons, or approximately 16% of global crude palm oil production, the group said in a news release. Spread over almost 2 million hectares of certified production area, about 48% of the world’s current RSPO-certified sustainable palm oil production capacity comes from Indonesia, followed by 44% from Malaysia, and the remaining from Papua New Guinea, Solomon Islands, Brazil, Thailand, Colombia, Cambodia, and Ivory Coast.

Fast-food retailer McDonald’s honored Cargill in April 2014 with seven “Best of Sustainable Supply” awards, including two category winners for community impact and economics. The Cargill projects recognized by McDonald’s include:

- Raising per-acre yields of canola to fulfill McDonald’s requirements for high-oleic canola oil;
- Establishing Indonesia’s first palm oil teaching farm;
- Implementing renewable energy sources in chicken housing to reduce the housing’s carbon footprint while improving animal health;
- Helping cacao farmers implement sustainable practices, improve production and quality, and earn better prices;
- Collaborating with the World Wildlife Fund to boost corn production while protecting environmentally sensitive lands in Northeast China’s Jilin province;
- Setting environmental targets for stevia cultivation to reduce greenhouse gas emissions, water use and waste, and ensure stevia is not grown on conservation or protected lands;
- Working with CARE International to reduce hunger, increase farmer productivity, and improve children’s education. CARE, or the Cooperative for Assistance and Relief Everywhere, is an international humanitarian relief agency founded in 1945 that is based in Chatelaine, Switzerland.

These seven Cargill projects were among 51 honored by McDonald’s with its 2014 Best of Sustainable Supply awards.

For the first time, a field test has demonstrated that elevated levels of carbon dioxide (CO₂) inhibit plants’ assimilation of nitrate into proteins, indicating that the nutritional quality of food crops is at risk as climate change intensifies. Findings from this wheat field-test study, led by a University of California at Davis plant scientist, were reported in the

CONTINUED ON NEXT PAGE
“Food quality is declining under the rising levels of atmospheric CO₂ that we are experiencing,” said lead author Arnold Bloom, a professor in the Department of Plant Sciences.

“Several explanations for this decline have been put forward, but this is the first study to demonstrate that elevated CO₂ inhibits the conversion of nitrate into protein in a field-grown crop,” he said.

The assimilation of nitrogen plays a key role in the plant’s growth and productivity. In food crops, it is especially important because plants use nitrogen to produce the proteins that are vital for human and animal nutrition. Many previous laboratory studies had demonstrated that elevated levels of atmospheric CO₂ inhibited nitrate assimilation in the leaves of grain and non-legume plants; however, there had been no verification of this relationship in field-grown plants.

To observe the response of wheat to different levels of atmospheric CO₂, the researchers examined samples of wheat that had been grown in 1996 and 1997 in the Maricopa Agricultural Center near Phoenix, Arizona, USA.

At that time, CO₂-enriched air was released in the fields, creating an elevated level of atmospheric carbon at the test plots, similar to what is now expected to be present in the next few decades. Control plantings of wheat were also grown in the ambient, untreated level of CO₂.

Leaf material harvested from the various wheat tests plots was immediately placed on ice and then was oven dried and stored in vacuum-sealed containers to minimize changes over time in various nitrogen compounds.

A fast-forward through more than a decade found Bloom and the current research team able to conduct chemical analyses that were not available at the time the experimental wheat plants were harvested.

In the recent study, the researchers documented that three different measures of nitrate assimilation affirmed that the elevated level of atmospheric CO₂ had inhibited nitrate assimilation into protein in the field-grown wheat.

“These field results are consistent with findings from previous laboratory studies, which showed that there are several physiological mechanisms responsible for CO₂’s inhibition of nitrate assimilation in leaves,” Bloom said.

Bloom noted that other studies also have shown that protein concentrations in the grain of wheat, rice and barley—as well as in potato tubers—decline, on average, by approximately 8% under elevated levels of atmospheric CO₂.

“When this decline is factored into the respective portion of dietary protein that humans derive from these various crops, it becomes clear that the overall amount of protein available for human consumption may drop by about 3% as atmospheric CO₂ reaches the levels anticipated to occur during the next few decades,” Bloom said.

While heavy nitrogen fertilization could partially compensate for this decline in food quality, it would also have negative consequences including higher costs, more nitrate leaching into groundwater and increased emissions of the greenhouse gas nitrous oxide, he said.

• **Replacement of vitamins A and C with vitamin D and potassium.** Dietary deficiencies of vitamin D and potassium are widespread, FDA believes, referring to these two substances as “nutrients of public health significance.” Calcium and iron will still be required on the nutrition label, and vitamins A and C may be included on a voluntary basis.

• **Revised daily values.** Proposed changes for the daily values of certain nutrients include:
  ○ A decrease in sodium from 2400 mg to 2300 mg;
  ○ An increase in dietary fiber from 25 mg to 28 mg; and
  ○ A change for vitamin D from 400 international units to 20 micrograms.

• **Increased record-keeping requirements.** The FDA noted that there are no analytical methods currently that can distinguish between dietary fiber and nondigestible carbohydrates that do not meet the definition of dietary fiber, added and naturally occurring sugars, the various forms of vitamin E, or folate and folic acid. Also, there are no analytical methods that can determine the amount of added sugar in specific foods containing added sugars alone or in combinations with naturally occurring sugars, where the added sugars are subject to fermentation. Therefore, for the products with these ingredients, the agency is proposing that manufacturers must make and keep certain written records for two years “in order to verify their declarations of each of these nutrients in the labeling of the food associated with such records.”
According to CBC News (division of the Canadian Broadcasting Corp.), the Thunder Bay Generating Station in the province of Ontario burned its last supply of coal in April, making Ontario the first political unit in North America to fully eliminate coal as a fuel for electricity generation. The plant will switch to using steam-exploded biomass fuel, probably by January 2015. The operator of the generating station, Ontario Power Generation, indicated that Thunder Bay will likely be the first power plant in the world to use this fuel. A fuel supplier has not yet been selected.

The College of Agriculture at the University of Hawaii has received authorization to conduct a two-year study of hemp. The research will focus on the potential use of hemp as a biofuel feedstock and in phytoremediation, that is, the use of hemp plants to remove contaminants from soil. A precondition is that the plant stock used in the study be certified by the state Department of Agriculture as industrial hemp. The Honolulu Police Department opposed the approval, saying it would be difficult to distinguish industrial hemp from marijuana.

The Energy Information Administration of the US Department of Energy reported that the beginning stocks of biodiesel in the United States in January 2013 were 87.5 million gallons (1 gallon = 3.8 liters). Production that year was 1,339 million gallons, and imports were 315 million gallons, for a total of 1,742 million gallons. Ending stocks were 189 million gallons, for a disappearance of 1,552 million gallons. For comparison, beginning stocks in 2012 were 84.5 million gallons, production was 991 million gallons, and imports were 35.8 million gallons, for a total supply was 1,111 million gallons. Ending stocks were 87.5 million gallons (http://tinyurl.com/biodiesel-EIA-2013).

British Airways to burn garbage

British Airways announced plans on April 16 to power its flights using sustainable jet fuel made from landfill waste. Solena Fuels (headquartered in Washington, DC, USA) will partner with British Airways to build the world’s first facility to convert landfill waste into jet fuel.

The facility, called the GreenSky London fuel facility, will be located in the Thames Enterprise Park, part of the site of the former Coryton oil refinery in Thurrock, Essex, UK. The site was selected for the availability of transportation links and existing fuel storage facilities. Construction is scheduled to be completed in 2017, creating up to 150 permanent jobs.

Solena’s technology will convert about 575,000 metric tons (MT) of post-recycled waste, normally destined for landfill or incineration, into 120,000 MT of liquid fuels. British Airways has committed to purchase $0,000 MT per year of the jet fuel at market-competitive rates.

Solena is providing patented high-temperature plasma gasification technology to convert the waste efficiently into synthetic gas (a mixture consisting primarily of hydrogen, carbon monoxide, and often some carbon dioxide). The gas will then be converted into liquid hydrocarbons using technologies such as cleaning and conditioning the gas, a Velocys Fischer-Tropsch conversion process, hydrocracking, and electric power production.

In a company statement, Willie Walsh, chief executive of British Airways’ parent company IAG, said, “The sustainable jet fuel produced each year will be enough to power our flights from London City Airport twice over with carbon savings the equivalent of taking 150,000 cars off the road.”

ABO 2014 Industry Survey

The Algae Biomass Organization (ABO) has summarized 285 opinions and projections it
received in March 2014 from its survey on the industry’s growth barriers to advancement and policy priorities through 2020. Respondents were optimistic that algae-derived fuels will be price competitive with fossil fuels by 2020 (83% agreement); however, this was a decrease from 91% for a similar survey in 2013.

Twenty-eight percent of survey responders who produce algae biomass or algae-derived products think the cost per gallon of algae-based fuel will be below $3.00 per gallon by 2020, while 42% believe prices will be below $5.00 per gallon.

Ninety-six percent of survey respondents indicated they think that cost-competitive bioplastics or biochemicals derived from algae will be commercially available by 2020. Ninety-seven percent believe that cost-competitive algae-based feeds in agriculture or aquaculture will be commercially available by 2020.

The most important existing federal government policies for building a robust algae industry were identified in the survey as (i) research and development grants, (ii) tax credits and incentives, and (iii) the renewable fuel standard.

For further information see http://tinyurl.com/ABO-2014-Survey.

Game-changers

At the seventh Bloomberg New Energy Finance Summit, 10 “game-changing” companies in the field of clean energy technology and innovation were recognized (see http://www.newenergypioneers.com). Among the criteria for selection, the service or product provided by the company must be new, different, and disruptive. Furthermore, the company must have been operating for 10 years or less.

Two companies among this year’s recipients, named in April 2014, are involved in making commodity chemicals from biomass:

- Genomatica (San Diego, California, USA), which develops and licenses complete manufacturing processes for producing widely used chemicals from alternative bio-feedstocks, at competitive cost and with greater sustainability than petroleum-based processes.
  Among the products with which it is involved are butadiene, 1,4-butanediol, and polybutylene terephthalate.

- Renmatic Inc. (King of Prussia, Pennsylvania, USA), which licenses its Plantrose bio-based technology—utilizing supercritical hydrolysis to break down nonfood mass quickly while consuming no significant quantities of consumables—to enable the production of petrochemicals from plants.

Biodiesel mandates in Canada

The provincial government of Ontario (Canada) introduced a mandate for the use of “Greener Diesel” on April 1, 2014. The intention is to reduce the production of greenhouse gas emissions and to encourage biodiesel production in the province. It should also reduce smog-forming particulates and improve air quality in the province’s urban centers.

The present inclusion requirement for biodiesel in Ontario is 2% biodiesel in petrodiesel. This will be followed by an increase to 4% by 2017.

Other Canadian provinces already have biofuel mandates in place, namely, British Columbia, 4%; Alberta, 2%; Saskatchewan, 2%; and Manitoba, 2%. Additionally, Prince Edward Island had proposed that a 10% mandate be introduced by 2013, to be doubled by 2018, but this has not yet come to pass. Newfoundland gets a permanent exemption on renewable content in the diesel fuel sold in that province due to logistical challenges of blending biodiesel in the region (http://tinyurl.com/Canada-biodiesel).

Only six US states mandate the use of any form of biodiesel.

Neol Biosolutions to scale up MicroBiOil®-2.0

Neol Biosolutions, a 50:50 joint venture between Repsol (Madrid, Spain) and Neuron Bio (Granada, Spain), has reached an agreement with the Center for Second Generation Biofuels of CENER (the Spanish National Renewable Energy Center) to carry out scale-up testing of its second technological platform, MicroBiOil-2.0.
MicroBiOil-2.0 is able to produce large quantities of triglyceride oil from different types of agricultural waste of different origins such as wheat straw or sugarcane bagasse, thanks to the microorganism Neoleum®, which was discovered and developed by Neol. According to the company website (www.neolbio.com), Neoleum can produce in a 1.0 m³ bioreactor the same amount of oil as from 1 hectare of oil palm. The oils so obtained can be used as feedstock for the production of biodiesel, renewable diesel, and biokerosene, as well as other oleo products such as biosurfactants and biolubricants.

Neol (Granada, Spain) has a collection of over 9,000 microorganisms (bacteria, molds, fungi, and microalgae) isolated from extreme ecosystems, from which to choose in developing renewable feedstocks, bioplastics, and sustainable processes.

Ethanol news
Syngenta process: more ethanol, more corn oil. In mid-April Syngenta announced an agreement with Cellulosic Ethanol Technologies, LLC, a subsidiary of Quad County Corn Processors (QCCP), to license its Adding Cellulosic Ethanol (ACE) technology, a new process for ethanol plants.

As well as converting corn starch into ethanol, the ACE technology can, in a bolt-on process, convert corn kernel fiber into cellulosic ethanol. ACE technology is designed to increase a plant’s ethanol production in combination with the Enogen® corn trait from Syngenta. (Enogen trait technology is one of the only corn output traits designed specifically to enhance ethanol production.) ACE technology has been added to the QCCP facility in Galva, Iowa, USA. The schedule called for production runs to start in May 2014.

According to Delayne Johnson, chief executive officer of QCCP, “The integration of the Adding Cellulosic Ethanol process into the QCCP plant operation will help create a higher protein feed [DDGS], 2.5 times more corn oil, and more ethanol out of the same kernel of corn.

The supply of corn oil has grown roughly four times in the past 34 years according to materials in the US Department of Agriculture Economic Research Service Oil Crops Yearbook. (see table in this month’s supplement, digital and mobile editions only).

POET-DSM, July 2014 is the expected date for POET-DSM to start producing cellulosic ethanol from corn stover in its facility at Emmetsburg, Iowa, USA. The company anticipates producing about 12 million gallons (45 million liters) before the end of December, at which time it should be at full production. In subsequent years the plant is scaled to produce 20–25 million gallons annually. Feedstock is being drawn from an approximately 40-mile radius of the plant. Construction on the plant began in early 2012.

New uses for tobacco. Tobacco leaves are already being investigated as a source of vegetable oils for use in producing biofuels (for example, see http://tinyurl.com/ARPA-E-tobacco). Agricultural methods of growing tobacco are well worked out owing to the past use of the leaves in making smoking materials such as cigarettes, cigars, and snuff. The growth of tobacco is in decline in the USA and in Europe, owing both to greater awareness of the harmful properties of smoking tobacco and to the growing production in countries such as China.

Researchers in Spain are now investigating tobacco plants that are genetically modified to produce increased amounts of starch and sugars as a basis for producing ethanol (http://tinyurl.com/Basque-tobacco-EtOH). The plants are grown at a high

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density, allowing production of up to 160 metric tons of biomass per hectare for the growing season.

Pacific Biodiesel closes its prototype facility

In a company statement, Pacific Biodiesel (Kahului, Hawaii, USA) said on April 12, 2014, that it had vacated its production plant at the Central Maui Landfill. Built in 1996, Pacific Biodiesel’s Maui plant was the longest continually operating commercial biodiesel processing facility in America.

The Maui County Department of Environmental Management notified Pacific Biodiesel in December 2013 that continuing its current operation would require multiple permits and extensive upgrades to comply with new County requirements. Robert King, president and founder of the company, said, “With just over two years left on our contract, we couldn’t justify the costly site improvements that were required to meet the County’s demands.”

Pacific Biodiesel will continue its pumping and collection services on the islands of Maui and Lanai, as well as its collection and processing operations on Oahu and Hawaii Island. Distribution of the Company’s biodiesel fuel will continue in the state.

Worldwatch Institute examines global biofuel production

- In a report issued on April 16, 2014, Worldwatch Institute examined trends in global biofuel production as of 2012. Both ethanol and biodiesel were considered (http://tinyurl.com/Worldwatch-biofuel).
- Global ethanol production continued the decline first noted in 2010, falling to 83.1 billion liters, while biodiesel output rose fractionally from 22.4 billion liters in 2011 to 22.5 billion liters in 2012. Biodiesel accounted for more than 20% of global biofuel production.
- The top five ethanol producers in 2012 were the United States (50.4 billion liters), Brazil (21.6 billion liters), China (2.1 billion liters), Canada (1.8 billion liters), and France. The United States and Brazil together accounted for 87% of the global total.
- The United States also led in biodiesel production in 2012 with 3.6 billion liters. Argentina was second, with 2.8 billion liter, and Germany and Brazil each produced about 2.7 billion liters. China’s output of biodiesel was only 200 million liters, lagging far behind its output of fuel ethanol. The European Union as a whole accounted for 41% of global biodiesel output despite a decline of 7% in 2012.
- Worldwide, biodiesel production grew at an average annual rate of 17% from 2007 through 2012, although the rate of growth slowed considerably over that period.
- Mandates or targets for the use of biofuels have been established in 13 countries in the Americas, 12 in the Asia-Pacific region, and 8 in Africa. In Europe, the EU-27 group of countries is subject to a Renewable Energy Directive (RED) that called for 5.75% biofuel content in transportation fuels in 2012. The United States and China have established—and Brazil has already achieved—targets of between 15% and 20% no later than 2022; India has also mandated 20% ethanol by 2017. Politics has a way of influencing the implementation of these mandates, however.
- Biofuels for transportation, which at present means ethanol and biodiesel, account for about 0.8% of global energy use, 8% of global primary energy derived from biomass, 3.4% of global road transport fuels, and 2.5% of all transport fuels.
- Global investment in biofuels was about $5 billion in 2012, down 40% from 2011. Of this amount, about $3.8 billion was in industrial nations and the rest in developing ones.
US researchers have discovered and validated a lipid panel blood test that can predict with greater than 90% accuracy if a healthy person will develop mild cognitive impairment or Alzheimer’s disease (AD) within three years.

Described in *Nature Medicine* (http://dx.doi.org/10.1038/nm.3466, 2014), the study heralds the potential for developing treatment strategies for AD at an earlier stage, when therapy would be more effective at slowing or preventing onset of symptoms. It is the first known published report of blood-based biomarkers for preclinical AD.

The test identifies 10 lipids in the blood that predict disease onset. It could be ready for use in clinical studies in as few as two years and, researchers say, other diagnostic uses are possible.

“Our novel blood test offers the potential to identify people at risk for progressive cognitive decline and can change how patients, their families, and treating physicians plan for and manage the disorder,” says the study’s corresponding author Howard J. Federoff, professor of neurology and executive vice president for health sciences at Georgetown University Medical Center (GUMC) in Washington, DC.

There is no cure or effective treatment for AD. Worldwide, about 35.6 million individuals have the disease and, according to the World Health Organization, the number will double every 20 years to 115.4 million people by 2050.

Federoff explains there have been many efforts to develop drugs to slow or reverse the progression of AD, but all of them have failed. He says one reason may be the drugs were evaluated too late in the disease process.

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“The preclinical state of the disease offers a window of opportunity for timely disease-modifying intervention,” Federoff says. “Biomarkers such as ours that define this asymptomatic period are critical for successful development and application of these therapeutics.”

The study included 525 healthy participants aged 70 and older who gave blood samples upon enrolling and at various points in the study. Over the course of the five-year study, 74 participants met the criteria for either mild AD or a condition known as amnestic mild cognitive impairment (aMCI), in which memory loss is prominent. Of these, 46 were diagnosed upon enrollment and 28 developed aMCI or mild AD during the study (the latter group are known as “converters”).

In the study’s third year, the researchers selected 53 participants who developed aMCI/AD (including 18 converters) and 53 cognitively normal matched controls for the lipid biomarker discovery phase of the study. The lipids were not targeted before the start of the study, but, rather, were an outcome of the study.

A panel of 10 lipids was discovered, which researchers say appears to reveal the breakdown of neural cell membranes in participants who develop symptoms of cognitive impairment or AD. The panel was subsequently validated using the remaining 21 aMCI/AD participants (including 10 converters), and 20 controls. Blinded data were analyzed to determine if the subjects could be characterized into the correct diagnostic categories based solely on the 10 lipids identified in the discovery phase.

“The lipid panel was able to distinguish with 90% accuracy these two distinct groups: cognitively normal participants who would progress to [a]MCI or AD within two to three years, and those who would remain normal in the near future,” Federoff says.

The researchers performed stable isotope dilution–multiple reaction monitoring mass spectrometry to identify and quantify lipids, amino acids, and biogenic amines; “this would discriminate our groups with emphasis on differences that might predict phenocconversion from normal control to aMCI/AD,” write the authors. This analysis revealed significantly lower plasma levels of serotonin, phenylalanine, proline, lysine, phosphatidylcholine (PC), taurine, and acylcarnitine (AC) in converter participants who later phenocverted to aMCI/AD.

“A notable finding of this targeted metabolomic and lipidomic analysis was the identification of a set of 10 metabolites, comprising PCs [PC diacyl (aa) C36:6, PC aa C38:0, PC aa C38:6, PC aa C40:1, PC aa C40:2, PC aa C40:6, PC acyl-alkyl (ae) C40:6], lysophosphatidylcholine (a C18:2), and AC [propionyl AC (C3) and C16:1-OH] that were depleted in the plasma of the converter participants but not in that of the normal control group. These metabolites remained depleted after phenocconversion to aMCI/AD and were similar to the levels in the aMCI/AD group.”

The researchers also looked to see if the presence of the APOE4 gene, a known risk factor for developing AD, would contribute to accurate classification of the groups but found it was not a significant predictive factor in this study.

“We consider our results a major step toward the commercialization of a preclinical disease biomarker test that could be useful for large-scale screening to identify at-risk individuals,” Federoff says. “We’re designing a clinical trial where we’ll use this panel to identify people at high risk for AD to test a therapeutic agent that might delay or prevent the emergence of the disease.”

In addition to Federoff and other researchers at GUMC, study authors included researchers from the Temple University School of Medicine in Philadelphia, Pennsylvania; Unity Health System, the University of Rochester School of Medicine, and Rochester General Hospital, all in Rochester, New York; the Regis University School of Pharmacy in Denver; and the University of California-Irvine.

The precise reason for the health benefits of dark chocolate?

The health benefits of eating dark chocolate are well accepted, but the exact mechanisms have remained a mystery. New work, however, suggests a solution. In brief: Score another positive health effect for the gut microbiome.

Findings discussed by researchers at the 247th National Meeting & Exposition of the American Chemical Society, held March 16–20, 2014, in Dallas, Texas, USA, suggest that certain bacteria in the stomach ferment it into compounds that are good for the heart.

“We found that there are two kinds of microbes in the gut: the ‘good’ ones and the ‘bad’ ones,” explained Maria Moore, an undergraduate student at Louisiana State University in Baton Rouge, USA, and one of the study’s researchers.

“The good microbes, such as Bifidobacterium and lactic acid bacteria, feast on chocolate,” she said. After a person eats dark chocolate, the gut bacteria grow and ferment the chocolate, producing compounds that are anti-inflammatory. The other bacteria in the gut are associated with inflammation and can cause gas, bloating, diarrhea, and constipation. These include some clostridia and some strains of E. coli.

“When these compounds [the fermentation products of the ‘good’ microbes] are absorbed by the body, they lessen the inflammation of cardiovascular tissue, reducing the long-term risk of stroke,” said John Finley, who led the work. He said that this study is the first to look at the effects of dark chocolate on the various types of bacteria in the digestive tract.

The team tested three cocoa powders using a model digestive tract, comprised of a series of modified test tubes, to simulate normal digestion. They then subjected the non-digestible materials to anaerobic fermentation using human fecal bacteria.

Finley explained that cocoa powder contains several polyphenolic, or antioxidant, compounds such as catechin and epicatechin, and a small amount of dietary fiber. Both antioxidant components are poorly digested and absorbed, but when they reach the colon, the desirable microbes take over. “In our study
we found that the fiber is fermented and the large polyphenolic polymers are metabolized to smaller molecules, which are more easily absorbed. These smaller polymers exhibit anti-inflammatory activity,” he said.

Finley also noted that combining the fiber in cocoa with prebiotics is likely to improve a person’s overall health and help convert polyphenols in the stomach into anti-inflammatory compounds. Prebiotics are carbohydrates found in foods such as raw garlic and cooked whole wheat flour that humans can’t digest but that beneficial bacteria can readily metabolize.

Progress made developing creamier low-fat food products

Adjusting the calcium level and acidity of processed foods could be the key to developing better-tasting, more eye-appealing, and creamier reduced-fat sauces, desserts, and salad dressings, according to research conducted at the University of Massachusetts Amherst (USA).

In a laboratory study, AOC member D. Julian McClements and his team reduced the fat content and, therefore, calories in a model white sauce from 10% to 2% without sacrificing the look and feel of the food. “By controlling pH and calcium content, we are able to regulate the interactions among fat droplets,” explained Bicheng Wu, a graduate student. “This makes them stick together and form flocs, or clumps. We believe the water trapped inside these flocs makes the sauce seem fattier than it really is and preserves the look, feel, and flavor.”

Wu presented the findings at the 247th National Meeting of the American Chemical Society, held in Dallas, Texas, in March 2014. She noted that fat plays various roles in determining the overall sensory attributes of food products. “It carries flavors, so cutting the fat content lessens the intensity of the flavor. The appearance, meaning the opacity or lightness, of a food mixture largely depends on light scattering by fat droplets, so high fat content gives a milky appearance to a sauce or dressing.” High fat content is also related to the thick, smooth, and creamy mouth-feel of many products, she said, such as pudding, owing to how fat droplets affect the flow of the food matrix.

Yet another problem with cutting the fat content is that the food then doesn’t make people feel as full, McClements noted. “Due to the high calorie count in fat and how the body digests it, fat also affects the feeling of satiety.”

The team plans to conduct extensive taste and odor tests. “Then we will be able to adjust the composition and incorporate other seasoning ingredients into the foods,” he said.

The study was supported by ConAgra Foods.
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The International Peanut Genome Initiative (IPGI)—a multinational group of crop geneticists who have been working with The Peanut Foundation (http://www.peanutfoundation.org)—announced on April 2, 2014, that they have successfully sequenced the peanut (groundnut) genome. The new sequence is now available to researchers and plant breeders around the world for use in breeding more productive and more resilient peanut varieties.

**Implications.** Richard Wilson—former president of AOCS, retired US Department of Agriculture National Program Leader for oilseed research and now a consultant dealing in oilseeds and biosciences—said that the genomic sequences now available at the Peanutbase.org website (http://peanutbase.org/files/genomes) are “extremely high-quality roadmaps to gene locations on each peanut chromosome.” He indicated that this resource will enable and expedite efforts to develop enhanced peanut cultivars in the United States, China, India, South America, and Africa. At least four future outcomes from this work should be of interest to AOCS members:

- Early efforts will be directed toward increasing the supply of peanuts with higher oleic acid content. This trait will help extend the shelf life of peanut products, particularly in the United States where about 80% of the peanuts grown are eaten whole, in confections, or in peanut butter products.
- The time required to develop new varieties will be cut considerably, from a typical six years to perhaps as low as two years.
- Farmer costs per acre to grow peanuts should fall. At present peanut plants are susceptible to a number of fungal infections,
requiring farmers to treat their crops with fungicides many times during the growing season. With a blueprint of the peanut genome in hand, researchers will be able to identify genes in strains known to have some resistance to fungal infection and develop new crosses that enhance disease resistance.

- Greater availability of high-quality peanuts will help ensure food security in nations that suffer from chronic hunger.

**Background.** According to FAO statistics, in 2012 farmers in a total of 113 countries raised peanuts on 24.7 million hectares and harvested about 41 million metric tons.

Although peanuts (*Arachis hypogaea*) have been bred for intensive cultivation for thousands of years, relatively little had been known about the legume's genetic structure because of its complexity, according to Peggy Ozias-Akins, a plant geneticist with the University of Georgia (UGA)-Tifton (USA) who also works with the IPGI and directs the UGA Institute of Plant Breeding, Genetics, and Genomics (http://tinyurl.com/peanut-genome-sequenceIPGI).

The peanut in fields today is the result of a natural cross between two wild species, *Arachis duranensis* and *Arachis ipaensis*, which occurred in northern Argentina 4000–6000 years ago. Because its ancestors were two different species, today's peanut is a polyploid, meaning the species can carry two separate genomes, designated A and B subgenomes.

To map the peanut's structure, researchers sequenced the genomes of the two ancestral parents, because together they represent the cultivated peanut. The sequences provided researchers access to 96% of all peanut genes in their genomic context, providing the molecular map needed to more quickly breed drought- and disease-resistant, lower-input and higher-yielding varieties of peanuts.

The two ancestor wild species had been collected in nature decades ago. *Arachis duranensis* is widespread in nature today, but *A. ipaensis* has only ever been collected from one location and may now be extinct in the wild. Long-sighted efforts toward germplasm collection and conservation of these species provided IPGI with the materials to understand the peanut genome better.

Knowing the genome sequences of the two parent species will allow researchers to recognize the cultivated peanuts' genomic structure by differentiating between the two subgenomes present in the plants. Being able to see the two separate structure elements also will aid future gene marker development—the determination of links between a gene's presence and a physical characteristic of the plants.

In addition, these genome sequences will serve as a guide for the assembly of the cultivated peanut genome that will help to decipher genomic changes that led to peanut domestication, which was marked by increases in seed size and plant growth habit.

Scientists from all around the world worked together through the IPGI to delineate peanut genome sequences, characterize the genetic and phenotypic variation in cultivated and wild peanuts, and develop genomic tools for peanut breeding. Research was carried out in US universities (California-Davis, Georgia, Texas A&M, North Carolina), the USDA Agricultural Research Service (USDA-ARS; Georgia, Mississippi, Iowa), the National Center for Genome Resources (New Mexico, USA), Brazil (University Brasilia, Embrapa Recursos Genéticos e Biotecnologia), China (Academy of Agricultural Sciences, Beijing Genome Institute, Shandong Academy of Agricultural Sciences, Henan Academy of Agricultural Sciences), ICRI SAT (International Crops Research Institute for the Semi-Arid Tropics), Japan, Israel, and Australia. A complete list of the institutions involved with the project and funding sources is available at http://www.peanutbioscience.com.

The initial sequencing was performed by the Beijing Genomic Institute (BGI). Assembly was carried out at the BGI, USDA-ARS (Ames, Iowa), and the University of California-Davis.

### Camelina to be field-trialed

The UK Department for Environment, Food and Rural Affairs (DEFRA) granted permission on April 16, 2014, for Rothamsted Research (Harpenden, UK) to carry out field trials with *Camelina sativa* plants genetically modified (GM) to produce omega-3 fish oils in their seeds. Rothamsted applied in late January 2014 for permission to carry out this field trial in the growing seasons of 2014, 2015, 2016, and 2017. The independent Advisory Committee on Releases to the Environment (ACRE) reviewed the risk assessment of the project, and DEFRA carried out a 60-day public consultation. Also, Rothamsted scientists spoke to and answered questions directly from the public and from special interest groups that expressed interest in the research project and the trial.

At the end of its review, ACRE expressed satisfaction that all scientific issues raised by the public with respect to this application had been addressed.

In a statement issued by Rothamsted, Johnathan Napier, lead scientist of the project, said, “The project is a core element of our strategic program grant Designing Seeds for Nutrition and Health, which is funded by the BBSRC [Biotechnology and Biological Sciences Research Council]. Being able to carry out the field trial with our GM plants means that we have reached a significant milestone in the delivery of our research program.”

The trials will test whether GM *Camelina sativa* can make significant quantities of omega-3 long-chain polyunsaturated fatty acids, particularly docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA), in its seeds under field conditions.

Camelina seeds were scheduled to be planted by mid-May 2014 in secure fields in Hertfordshire, with harvest anticipated in August/September 2014. A small amount of seed will be analyzed for its oil content, and the rest of the seed and plant material will be destroyed according to the conditions under which the study is being carried out.

Jackie Hunter, chief executive of the BBSRC, said, “This research is seeking to provide an alternative source of omega-3 oil for the aquaculture industry that is seeking new ways to maintain and increase its sustainability. After many years of BBSRC supported laboratory research this project has reached the point where only a field trial will show scientists if this could work in real world conditions.”

For further information see http://tinyurl.com/BBSRC-Rothamsted-camelina.
Food labeling

US Congressmen Mike Pompeo (Republican-Kansas) and G.K. Butterfield (Democrat-North Carolina) introduced legislation on April 10 that would block efforts being made in a number of states to require mandatory labeling of food “produced from, containing, or consisting of a bioengineered organism.”

The bill, entitled the Safe and Accurate Food Labeling Act of 2014 (HR4432), would modify the US Food, Drug and Cosmetic Act to establish a federal labeling standard for genetically engineered ingredients. The US Food and Drug Administration would have sole authority to require mandatory labeling of such foods if they are ever found to be unsafe. The FDA could regulate optional labeling, according to the Politico website (http://tinyurl.com/politico-HR4432).

If passed, this bill will negate efforts by anti-GE groups and pesticide, grocery, and agriculture corporate trade groups that are fighting mandatory labeling efforts at the state and local level.

Rothamsted researchers boost seed oil content

A team of researchers from Rothamsted Research (Harpenden, UK) published in April 2014 the first study to target multiple genes that control a series of steps in the pathway for seed oil production. The study was carried out in the laboratory using the model oilseed plant Arabidopsis thaliana, which is related to oilseed rape.

The study established that specific combinations of individual genes involved in triacylglycerol metabolism could be used to achieve an additive effect, resulting in enhanced yield.

Seed-specific overexpression of WRINKLED1 (a transcriptional regulator of glycolysis and fatty acid synthesis) and DIACYLGLYEROL ACYLTRANSFERASE1 (a triacylglycerol biosynthetic enzyme) combined with suppression of the triacylglycerol lipase SUGAR-DEPENDENT1 results in a higher percentage seed oil content and greater seed mass than could be achieved by manipulating each gene individually.

Furthermore, seed vigor in plants with these “stacked” genes was not significantly different from controls. The average time for 50% of seeds of each genotype to germinate was not significantly different from controls, nor were the initial rates of seedling growth of the genetically modified seeds statistically different from the control plants.

For further information, see H. van Erp, A.A. Kelly, G. Menard, and P.J. Eastmond, Multi-gene engineering of triacylglycerol metabolism boosts seed oil content in Arabidopsis, CONTINUED ON NEXT PAGE
Differences between transgenic genotypes and parent varieties

A team of researchers headed by Owen A. Hoekenga of the Boyce Thompson Institute, Cornell University (Ithaca, New York, USA) published a comparison of the metabolome of a genetically modified (GM) tomato and those of a wide variety of garden, heirloom, and other non-GM varieties.

The GM tomato they analyzed had been engineered to delay fruit ripening. For the study, a water-alcohol solvent was used to extract the fruits, and roughly 1,000 metabolites from the GM tomato were compared with similar extracts from the non-GM tomatoes.

Hoekenga and his colleagues found through their analyses that there were differences between the GM fruit relative to its parent, although these changes were mostly seen in biochemicals related to fruit ripening. Hoekenga said, “So that’s part of an intended effect.”

The scientists next compared the metabolome of the GM tomato with an assortment of non-GM tomatoes. They found no significant differences overall. Although the GM tomato was distinct from its parent, its metabolic profile still fell within the “normal” range of biochemical diversity exhibited by the larger group of varieties.

The US Food and Drug Administration already requires developers of GM crops to compare a handful of key nutritional compounds in GM varieties relative to their non-GM parents. Part of the biotechnology risk assessment, the process is designed to catch instances where genetic manipulation may have affected nutritional quality.

Hoekenga’s team used nontargeted liquid chromatography–mass spectrometry metabolic fingerprinting to compare the cultivars they considered. This method is an especially good tool for guiding plant improvement, as it can record larger numbers of metabolites simultaneously without making a priori judgments of which will be biologically important. To analyze their data, the researchers used network analysis, a type of statistics, to identify clusters of metabolic similarities and to compare those patterns across different varieties.

In a statement released by the Crop Science Society of America, Hoekstra said, “The method can be applied to any plant or crop.” His group has already characterized the corn metabolome.

The group acknowledges, though, that his type of nontargeted metabolomics is expensive. Furthermore, the chemistry methods it uses are not yet robust enough for official safety assessments.

Solvay SA (Brussels, Belgium) announced in April 2014 that it has completed the acquisition of the specialty chemical assets of ERCA Química, Ltda. in Brazil. The acquisition effectively doubles Solvay’s surfactant production capacity in that country. “The transaction includes ERCA’s local specialty chemical assets, its portfolio of agrochemicals as well as home and personal care products, which strengthens Novecare’s commercial network, closeness to customers and innovation pipeline,” according to www.che.com.

Rivertop Renewables (Missoula, Montana, USA; www.rivertop.com), a producer of novel chemicals derived from plant sugars, announced in April 2014 that it has raised $26 million from Cargill, First Green Partners, and existing investors. Rivertop said it will leverage these funds “to produce market development quantities of salts of gluconic acid for select customers.” The company’s initial focus will be on the dishwasher detergent and corrosion inhibitor markets, according to a news release. Rivertop will also explore additional opportunities from among several large industries including home and personal care products, oil and gas, building and infrastructure, agriculture, and food.

Swiss specialty chemicals manufacturer Clariant announced in April 2014 that it has joined the “Together for Sustainability” (TfS; www.tfs-initiative.com) initiative, a chemical industry project founded in 2011 and aimed at improving sustainability practices within the industry’s supply chains. Clariant joins AkzoNobel, BASF, Bayer, Evonik Industries, Henkel, Lanxess, and Solvay in jointly operating global supplier assessments and third-party audits and by sharing scorecard ratings within the TfS member group on a web-based collaborative platform.

The American Cleaning Institute (ACI), a trade group based in Washington, DC, has released a four-minute video that summarizes a weight-of-evidence risk assessment on the low predicted

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Stepan introduces solvent-replacing surfactant

It took almost four years, but the joint research and development agreement (signed in 2010) between Stepan Co. and Elevance has led to the introduction of STEPOSOL® MET-10U, a novel surfactant derived from plant oils that is being marketed as a replacement for solvents. Stepan manufactures basic and intermediate chemicals and is based in Northfield, Illinois, USA. Elevance Renewable Sciences, Inc. produces biobased specialty chemicals and has headquarters in Woodridge, Illinois.

The new product utilizes a naturally derived, metathesized feedstock to form a biobased surfactant with a number of possible applications, according to the companies, including adhesive removal, paint and coating removal systems, kitchen degreasers, and all-purpose cleaners for both consumer and industrial uses.

Performance details about the new non-ionic surfactant, according to a Stepan news release (see http://tinyurl.com/Steposol), include the following:

- As a household cleaner, the surfactant reportedly delivers better and faster cleaning performance at half the solvent loading with neutral pH in comparison to alkaline degreasers (pH 11–13);
- As an adhesive remover, the surfactant provides equivalent removal of various adhesives at 5% concentration in aqueous phase compared with 100% N-methyl pyrrolidone; and
- As a paint remover, the surfactant achieves safe and efficient removal of latex paint at 5% concentration in aqueous phase compared with 100% solvent systems with methylene chloride.

Specific product attributes of STEPOSOL MET-10U include a Kauri-butanol value (a measure of solvent power of a hydrocarbon solvent) greater than 1,000; a boiling point of 297°C; a Biorenewable Carbon Index of 75%; and formulating pH range of 3–12. “These

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parameters create a high-strength and nonvolatile surfactant, which provides faster and more effective cleaning by fulfilling the role of both a low HLB (hydrophilic-lipophilic balance) surfactant and a strong solvent,” according to the news release.

A company spokesperson told Inform that all of the Elevance biorefineries will produce the same end products and are capable of running on multiple renewable oil feedstocks, including palm, mustard, soybean, canola, and, when they become commercially available, jatropha or algal oils. In addition, the Gresik (Indonesia) biorefinery is now operating on palm oil; the Natchez (Mississippi, USA) biorefinery will initially operate using canola or soybean oil. “The feedstocks can be sourced locally, enabling Elevance and its customers to reduce their carbon footprint across the entire supply chain,” the spokesperson added.

Wilmar buys Huntsman’s European surfactants business

Huntsman Corp. (The Woodlands, Texas, USA) and Wilmar International Ltd. (Singapore) announced in April 2014 that Wilmar’s wholly owned subsidiary, Wilmar Europe Holdings BV, has agreed to purchase Huntsman’s European commodity surfactants business. Completion remains subject to customary closing conditions, including regulatory procedures in France. Financial details of the agreement were not disclosed.

Under the terms of the agreement, Huntsman plans to sell to Wilmar its ethoxylation facility in Lavera, France. In addition, Wilmar will enter into a multiyear arrangement to purchase sulfated surfactant products from Huntsman’s facilities in Saint-Mihiel, France, and Castiglione delle Stiviere, Italy.

Separately, Huntsman has also announced its intention to cease production by October 2014 at its Patrica, Italy, commodity surfactants facility.

In October 2013, Huntsman announced plans to improve its annual EBITDA by $20 million by exiting a number of commodity surfactant product lines in Europe and to focus on developing and growing the remaining differentiated surfactants businesses. “The closure of the Patrica facility and the implementation of other restructuring activities, including the focus on differentiated surfactants following the sale to Wilmar, will result in a smaller, more profitable surfactants business for Huntsman in Europe going forward,” said the company in a news release.

Peter Huntsman, president and CEO of Huntsman Corp., said: “This restructuring and portfolio repositioning will benefit our Performance Products division in Europe and preserve jobs that otherwise would have been lost. The sale and offtake agreement with Wilmar will further cement the already strong relationship between our two companies. We plan to work closely together with Wilmar to ensure a smooth and seamless transition for customers and other stakeholders.”

Kuok Khoon Hong, Chairman and CEO of Wilmar International Ltd., said: “We are very pleased with this agreement with Huntsman, which enables Wilmar to broaden its existing footprint in Europe and extend its integrated chain to better serve customers in all geographies with responsible and quality products. The ongoing and successful relationship between our two companies will serve us both very well in our respective growth strategies.”

P&G makes “no deforestation” pledge

The Procter & Gamble Co. (P&G; Cincinnati, Ohio, USA) said in April 2014 it will require palm oil suppliers in Indonesia to submit plans that “demonstrate how they will ensure no deforestation in the supply chain for their mills by 2020.”

“The announcement came after the environmental advocacy group Greenpeace had pressured P&G to stop using palm oil from plantations that have clear-cut forests. Nine Greenpeace activists were arrested on March 4 after they entered P&G headquarters and unfurled protest banners from the 12th floor of the building. “We are committed to driving positive change throughout the entire supply chain, not just for us, but for the industry and for the small farmers who depend on this crop,” said Len Sauers, P&G’s vice president of Global Sustainability, in a news release. “Our aim is to develop effective long-term solutions to the complicated issue of palm oil sustainability.”

In related news, Ecover—the “green” cleaning company based in Malle, Belgium—announced in April 2014 that it will introduce a liquid laundry detergent in which 7% of its oil-based ingredients will come from algal sources. The company told The Guardian newspaper that it intends to increase the amount of algal oil “as more is learned about its use.” (See http://tinyurl.com/Ecover-algae.)

Toward sustainable laundry behavior

“Understanding the role of habit—and how to influence it—is a critical determinant of behavior,” notes the Cleaning Products Europe (CPE; www.cleaningproductseurope.com) 2014 website.

Why would a conference on home care be interested in how to influence consumer habits? Because as much as the home care industry has done to increase the sustainability of its products through reformulation and new packaging, the consumer is still key to the success of such efforts.

The UK’s Department for Environment Food & Rural Affairs commissioned a study (or “action-based research”) in 2012 to test the use of Implementation Intention Plans (IIP) as an approach to breaking and reforming new habits—based on its effective use predominantly in the health field. The technique involves the formation of simple “if-then plans,” which individuals adopt and rehearse, until new behavioral responses become encoded in their day-to-day environments. These plans help people to initiate new behaviors when good intentions alone are insufficient.

“This research worked closely with three major businesses and a small sample of consumers to understand whether IIP is effective in changing the sustainable habits of consumers,” said CPE in a blog post, “and whether businesses have the potential to adopt the use of IIP, and provide an effective channel for influencing the sustainable habits of their consumers.”

The CPE blog post continues: “Over a six-week trial period, an IIP-centered intervention was evaluated using six sustainable
behaviors, comprising IIP habit plans, investigative interviews, sharing experiences via social media, using a science expert, and the use of prompts. A mixed methods evaluation was adopted to determine in-depth insights and measure changes in habit strength.

“The research demonstrated that using an IIP-centered intervention approach is effective in changing sustainable habits, and achieving a high degree of habitual behavior change in the six sustainable behaviors tested (comprising laundry, showering, and food-related behaviors). The findings show that a mix of factors is important in influencing habit. Further, the research established that adopting IIP-centered interventions was attractive to the three businesses in engaging and influencing the sustainable habits of their consumers. However, more work is required to develop a scalable model and a tailored business case.”

The detailed results are currently subject to peer review before the results are published in third quarter of 2014, according to CPE.

US demand for lubricants stalls

US demand for lubricants is forecast to expand less than 1% annually to 2.5 billion gallons (about 10 billion liters) in 2018, valued at $27.5 billion. Although demand growth will be modest, this will represent a reversal of an outright drop in demand between 2008 and 2013.

According to analyst Jason Carnovale of The Freedonia Group market research firm, “Growth will result from an improving economic climate and an acceleration in manufacturing activity, supported by relatively low natural gas prices and strength in key lubricant-consuming industries.”

Stronger gains will be held back by losses in the light vehicle market, where the long-term trend of falling lubricant demand will continue. Improved efficiency and less frequent oil replacement will be a common trend not just in light vehicles but throughout all lubricant markets, serving to prevent faster growth. Longer oil change intervals will result from the greater use of higher-quality, better-performing products, such as synthetics. However, growth will remain healthy in value terms as average prices benefit from this shift toward premium products.

These and other trends are presented in Lubricants, a new study from Freedonia, which is based in Cleveland, Ohio, USA.

Among product types, those seeing use in commercial and industrial markets, such as heavy-duty diesel engine oils and gas turbine oils, will see the fastest growth. The impact of efficiency gains will be felt most strongly for products such as light vehicle engine oils and automatic transmission fluids. There will be opportunities in most markets for premium products that can offer improved lubrication, longer drain intervals, or increased environmental friendliness. Synthetic, re-refined, and biobased oils will substantially outpace conventional petroleum lubricants. For example, despite declines in the overall light vehicle market, synthetic engine oils will post strong gains.

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James Kenneth Daun

Former AOCS president James K. Daun died on April 4, 2014, in Winnipeg, Canada, at the age of 68. He is survived by his wife Lynda, whom he married in 1968; son Kyle (Caryn); daughter Meredith (Darryl); and granddaughter Mira.

Daun grew up in Winnipeg and received his B.S. in chemistry in 1967 from United College, University of Manitoba, Canada. He then worked two years as an analytical technologist for Atomic Energy of Canada Ltd. in Pinawa, Manitoba, before returning to the University of Manitoba. There he received his M.S. in plant science in 1973 and his Ph.D. in 1975 in grain technology under a Rapeseed Utilization Assistance Program Grant. Daun then joined the Canadian Grain Commission (CGC) in Winnipeg, where he remained until he retired in 2006. He also served as an adjunct professor at the University of Manitoba in the Faculty of Graduate Studies. In retirement he also was a consultant through his own company, AgriAnalytical Consulting.

Daun had his first contact with AOCS in 1972, when he attended an AOCS-sponsored short course in Champaign, Illinois. He subsequently joined the organization, and was a founding member of the Canadian section of the AOCS in 1986, serving as president of the latter in 1990–1991. He was a very willing volunteer for AOCS, serving on any number of committees during the 1980s and 1990s. He chaired the technical program for the 1992 joint meeting of AOCS/ISF, and was general chairperson for the Annual Meeting in 1994.

During 2011–2013, he was an associate editor for the Journal of the American Oil Chemists’ Society.

He served as a member-at-large of the AOCS Governing Board in 1992–1994 and then as secretary from 1994 to 1996. He was elected vice-president in 1996, and became president in 1997. During his association with AOCS, Daun was especially keen to expand outreach activities of AOCS and to encourage international harmonization of analytical methodology.

AOCS recognized Daun’s work and service on a number of occasions. He received the Archer Daniels Midland Award in 1989, the Herbert J. Dutton Award in 2005, and the Alton E. Bailey Award in 2011. He was named an AOCS Fellow in 2005.

He also received the Queen Elizabeth II Silver Jubilee Medal in 1977, the Queen Elizabeth II Golden Jubilee Medal in 2002, and the Eminent Scientist Award at the 12th International Rapeseed Congress in 2007.

In May 2012 Daun was made an honorary life member of the Canola Council of Canada (CCC) in recognition of his “work to define canola.” Much of his career was spent exploring composition and quality issues related to canola, rapeseed, flaxseed, and other oilseed crops grown in Canada. He helped create the official definition of canola by establishing specifications for glucosinolates and erucic acid and the method of glucosinolate measurement. In his acceptance of his life membership in the CCC, Daun said, “Of all the things I accomplished, I am most proud of my work to assist in the definition of canola.” These contributions helped transform canola into one of Canada’s most important agricultural commodities.

In retirement, Daun enjoyed traveling, taking photographs—some of which he emailed to AOCS headquarters to share his delight in what he was seeing—reading, curling, golfing, volunteering, and relaxing at Caddy Lake, near Whiteshell, Manitoba.

BRIEFS (cont. from page 373)

The US Food and Drug Administration’s (FDA) Center for Food Safety and Applied Nutrition has reorganized the cosmetics content of the FDA website to make it easier for consumers and other stakeholders to navigate the site (www.fda.gov/cosmetics). “Visitors to the website will see changes in the ‘Navigate the Cosmetics Section’ box on the www.fda.gov/cosmetics homepage, with new section titles that group cosmetics-related web content into logical categories intended to help users locate needed information more quickly,” FDA said in a news release.
Composition of biodegradable gear oil


A composition of biodegradable gear oil that mainly contains modified non-edible vegetable oils. Mono-esters are hydrogenated, epoxidized, or aryl alkylated or mixture thereof, and C7–C12 primary alcohol. In addition to chemically modified non-edible vegetable oils, the composition also contains an additive pack, which comprises at least one: antioxidant, an extreme pressure additive, an anti-foaming agent, a pour point depressant, a corrosion inhibitor, and a detergent-dispersant additive. The product of this invention has utility as industrial and automotive gear oil GL 4 grade. The compositions are significantly biodegradable, eco-friendly, reduce use of petroleum, have lower cost than synthetic oil, are miscible in mineral and synthetic fluids, and are safe to use due to higher flash point.

Aqueous polymer compositions obtained from epoxidized natural oils


Aqueous polyurethane dispersions are made from urethane prepolymer comprising one or more polyhydroxy compounds from ketone functional molecules derived from an epoxidized natural oil. Addition of a hydrazine functional moiety to the prepolymer dispersion can further provide a crosslinking mechanism resulting in the formation of azomethine linkages in the resulting polyurethane during drying. When the ketone functional molecule is derived from levulinic acid and epoxidized vegetable oil, the resulting urethane dispersion can also be converted into a hybrid polyurethane-vinyl dispersion by adding and polymerizing one or more vinyl monomers in the polyurethane prepolymer or polyurethane dispersion.

Production of soluble protein solutions from soy ("S701" CIP)


A soy protein product, which may be an isolate, produces transparent heat-stable solutions at low pH values and is useful for the fortification of soft drinks and sports drinks without precipitation of protein. The soy protein product is obtained by extracting a soy protein source material with an aqueous calcium salt solution to form an aqueous soy protein solution, separating the aqueous soy protein solution from residual soy protein source, adjusting the pH of the aqueous soy protein solution to a pH of about 1.5 to about 4.4 to produce an acidified clear soy protein solution, which may be dried, following optional concentration and diafiltration, to provide the soy protein product.

Wax compositions and methods of preparing wax compositions


Described are morphology stable wax compositions comprising the (trans)esterification product of a wax-forming composition comprising: (i) a triglyceride stock, a fatty acid stock, or a mixture thereof; and (ii) a glycerol-based composition comprising glycerol, polyglycerol, or a mixture thereof. Methods of making the waxes are also described. The wax compositions may be used in candles, stack waxes, jell candles, cosmetics, food coatings, adhesives, board manufacturing (e.g., particle board and oriented strand board), urethane foams or coatings, alkyd coatings, coating formulation additives, printing additives, polymer processing agents, extrusion processing aids, polymer plasticizers, mold releases, polishes, and stick markers.

Stable whippable and whipped food products


The present invention provides stable whippable or prewhipped food products comprising exudate gums in combination with proteins and optionally comprising cellulosic hydrocolloids; triglycerides; sweetener/bulking agents and water. The whippable liquid product is free of fat-based emulsifiers. The product is stable through processing and freeze/thaw and when whipped the resulting product can be used as icing, topping, and filling in a dessert. The prewhipped product further comprises non-hydrogenated fat-based emulsifiers but is free of other fat-based emulsifiers. This product can be stored frozen and when thawed, can be directly applied as icing, topping, and filling.

Coffee-derived surfactants


Coffee-derived surfactants are provided by transesterification reactions of sugars and coffee oil. The coffee-derived surfactants are especially useful in the production of coffee oil emulsions for use with, or incorporation into, coffee products.
Serum docosahexaenoic and eicosapentaenoic acid and risk of cognitive decline over 10 years among elderly Japanese


The purpose of the study was to clarify the association of serum docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA) levels with cognitive decline over 10 years. This study was part of the National Institute for Longevity Sciences—Longitudinal Study of Aging and was conducted with 232 male and 198 female Japanese community-dwelling subjects aged 60–79 years in the second wave (2000–2002).

Cognitive function was assessed with the Mini-Mental State Examination (MMSE) in both the second and seventh (2010–2012) waves. Fasting venous blood samples were collected in the morning, and serum DHA and EPA levels were measured. Multiple logistic regression analysis was performed among participants with an MMSE score ≥24 in the second wave (n = 430) to estimate the odds ratio (OR) and 95% confidence interval (CI) for MMSE score ≤23 or MMSE score decline ≥4 ten years later. These estimates were based on baseline tertiles of serum DHA or EPA levels, and controlled for age, sex, education, MMSE score at baseline, alcohol consumption, current smoking, body mass index and disease history. Fifteen (3.5%) subjects whose MMSE score was ≤23 and 36 (8.3%) subjects whose MMSE score declined to ≥4 showed cognitive decline. Multivariate-adjusted OR (95% CI) for the lowest through highest tertiles of serum DHA to MMSE score ≤23 or decline ≥4 were 1.00 (reference), 0.11 (0.02–0.58) and 0.17 (0.04–0.74), or 1.00 (reference), 0.22 (0.08–0.61) and 0.31 (0.12–0.75), respectively (P for trend = 0.01 or 0.04). Serum EPA was not associated with cognitive decline. The study gives some indication that a moderately high level of serum DHA might prevent cognitive decline among community-dwelling elderly Japanese individuals.

CONTINUED ON NEXT PAGE
Arachidonic acid synthesis from biodiesel-derived waste by Mortierella alpina


The synthesis of arachidonic acid (15–20% of lipid) from biodiesel-derived waste by Mortierella alpina strains was demonstrated. An inverse correlation between lipid and arachidonic acid production was revealed in media with different concentrations of carbon substrate. A method of increasing the arachidonic acid content of M. alpina lipid based on the additional incubation of harvested mycelium was developed. The effect of mycelium age, time of incubation, and temperature for 4–7 days, the arachidonic acid content of lipid in M. alpina strains NRRL-A-10995 and LPM-301 grown on biodiesel-derived waste and in the pure glycerol-grown strain LPM-301 increased by 162–199% of initial level (from 14.6 to 29.0%, from 19.8 to 36.5%, and from 35.0 to 56.8%, respectively).

Optimization of enzymatic synthesis of cocoa butter equivalent from high oleic sunflower oil


High-oleic sunflower oil (HOSO) and a fatty acid (FA) mixture wereinterested in a solvent-free system catalyzed by Lipozyme RM IM to produce a cocoa butter equivalent (CBE). The effects of reaction conditions on the percentage of saturation–oleoyl–saturate (SOS) and saturate–saturate–oleoyl (SSO) triacylglycerols (TAG) were studied. The process was further optimized by response surface methodology. A five-factor response surface design was used to investigate the influences of the five major factors and their mutual relationships. The five factors were: substrate ratio (A, FA/HOSO, mol mol−1), enzyme load (B, wt% based on substrates), water content (C, wt% based on substrates), reaction temperature (D, °C) and reaction time (E, in hours) varying at three levels together with two star point levels. The highest yield (59.1% SOS) and lowest acyl migration (2.9% SSO) was obtained at 10% enzyme load, 1% water content, 1:7 substrate mole ratio, 65°C reaction temperature, and 6 h reaction time. All the investigated factors except substrate ratio had significant effect on acyl migration. The quadratic response models sufficiently described the acylidosis reaction. All parameters had significant effect on the percentage of SOS TAG. Based on the models, the reaction was optimized to obtain a maximum yield of SOS TAG.

More Extracts & Distillates can be found in this issue’s supplement (digital and mobile editions only).
• CO2/N2 triggered switchable surfactants with imidazole group, Chái, M., Z. Zheng, L. Bao, and W. Qiao
• Enhancing of corrosion inhibition and the biocidal effect of phosphonium surfactant compounds for oil field equipment, Aiad, I.A., S.M. Tawfik, S.M. Shaban, A.A. Abd-Elaal, and M. El-Shafie
• Synthesis and surface properties of N,N-dimethyl-N-dodecyl polyoxyethylene amine-based surfactants: amine oxide, betaine and sulfobetaine, Hou, L., H. Zhang, H. Chen, Q. Xia, D. Huang, L. Meng, and X. Liu
• Synthesis and investigation of surface active properties of counterion coupled Gemini surfactants, Noori, S., A.Z. Naqvi, W.H. Ansari, M. Akram, and Kabir-ud-Din
• Cationic gemini surfactant as a corrosion inhibitor and a biocide for high salinity sulfidogenic bacteria originating from an oil-field water tank, Labena, A., M.A. Hegazy, H. Horn, and E. Müller
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• Synthesis and thermodynamic properties of rosino-based cationic Gemini surfactants, Deng, W., Y. Zhang, Y. Zhong, and J. Peng
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• Sugar-based ester quaternary ammonium compounds and their surfactant properties, Gan, C., H. Wang, Z. Zhao, and B. Yin
• Synthesis of quaternary, long-chain N-alkyl amides and their corrosion inhibition in acidic media, Öztürk, S., A. Yildirim, M. Çetin, and M. Tavasli
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• Fatty acids in heterocyclic synthesis. Part XIV: synthesis of surface active agents from some novel class of oxadiazole, thiazi-dazole and triazole derivatives having microbiological activities, Gad El-Karim, I.A., M.S. Amine, A.A. Mahmoud, and A.S. Gouda
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• Recovery of saponins from jua (Ziziphus joazeiro) by micellar extraction and cloud point preconcentration, Ribeiro, B.D., D.W. Barreto, and M.A.Z. Coelho
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• Triolein and trilinolein ameliorate oxidized low-density lipoprotein-induced oxidative stress in endothelial cells, Luo, T., Z. Deng, X. Li, H. Rao, and Y. Fan
Consumers worldwide are becoming more interested in what is in the foods they consume and how those foods are made. Edible oils, a leading source of dietary fats worldwide, are processed to improve consumer acceptance by removing or modifying components that can negatively impact appearance, taste, and shelf stability.

**Green Vegetable Oil Processing, Revised First Edition**

The revised first edition includes much of the content of the first edition, but incorporates updated data, details, images, figures, and captions. This book addresses alternative green technologies at various stages of oilseed and vegetable oil processing, including: oil extraction technologies such as expeller, aqueous, and supercritical methods; and green modifications of conventional unit operations such as degumming, refining, bleaching, hydrogenation, winterizing/dewaxing, fractionation, and deodorization. While most chapters describe soy oil processing, the techniques described are equally applicable to oils and fats in general.

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**Bleaching and Purifying Fats and Oils Theory and Practice, 2nd Edition**

Since the original publication of Patterson’s book in 1992, the bleaching process has continued to attract the attention of researchers and the edible oil industry. In this second edition, the reader is directed to more modern techniques of trace-metal analysis such as flame-atomic adsorption, graphite furnace atomic adsorption, atomic emission spectrometry involving direct current plasma (DCP), and inductively coupled plasma (ICP). It also discusses the Freundlich Equation and reports on high-temperature water extraction, high-temperature oxidative aqueous regeneration, and extraction with supercritical CO2. Finally, various degumming methods improved over the past several decades are discussed.

**Practical Guide to Vegetable Oil Processing**

Practical Guide to Vegetable Oil Processing is a comprehensive and made-easy review of all vegetable oil processing steps, including critical process control points, their significance, proper control ranges, and the troubleshooting process. This book is a must-have for vegetable oil processing and maintenance personnel, as well as equipment manufacturers. It discusses processing equipment and its functions, along with everything one needs to know to maintain the operation in good order.

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**Fats and Oils Handbook (Nahrungsfette und Öle)**

This book describes the raw materials predominantly used in fats and oils processing. It is the updated and revised English version of Nahrungsfette und Öle, originally printed in German. It contains 283 tables, 647 figures, and over 850 references.

Save 15% and get free shipping on these titles with promo code PROCESSING. Expiration July 11, 2014.
Experts in industrial processing, analytical chemistry, and toxicology from around the globe have collaborated with AOCS to provide books addressing how these oils are produced, laboratory resources for quality assurance testing, and analytical techniques for detection and mitigation of contaminants.

From AOCS Technical Services

Validated Methods for MCPD Ester Analysis

Process contaminants are a group of unrelated compounds formed in the production of finished food products. Of particular interest is the occurrence of variable levels of 3-MCPD esters in refined vegetable oils. Other process contaminants include pyrrolizidine alkaloids and acrylamide.

Discrepancies among different measurement techniques have hindered the assessment of levels of contamination. Although AOCS has favored solving this analytical problem using direct approaches, AOCS members recently adopted 3 indirect methods for determination of 3-MCPDs, 2-MCPDs, and glycidol in edible oils and fats following a collaborative study using defined reference materials.

- **AOCS Official Method Cd 29a-13**
  2- and 3-MCPD Fatty Acid Esters and Glycidol Fatty Acid Esters in Edible Oils and Fats by Acid Transesterification

- **AOCS Official Method Cd 29b-13**
  Determination of Bound Monochloropropanediol- (MCPD-) and Bound 2,3-epoxy-1-propanol (glycidol-) by Gas Chromatography/Mass Spectrometry (GC/MS)

- **AOCS Official Method Cd 29c-13**
  Fatty-acid-bound 3-chloropropane-1,2-diol (3-MCPD) and 2,3-epoxi-propane-1-ol (glycidol), Determination in Oils and Fats by GC/MS (Differential Measurement)

AOCS Laboratory Proficiency Program

The AOCS Laboratory Proficiency Program (LPP) is the most extensive and respected collaborative proficiency testing program for oil- and fat-related commodities, oilseeds, oilseed meals, and edible fats.

For those with an interest in contaminants analysis resources, the LPP offers the following:
- Mycotoxin Series: Fumonisin
- Aflatoxin in: Cornmeal, Milk, Peanut Butter, Peanut Paste

P: +1 217-693-4803  |  F: +1 217-693-4847  |  technical@aocs.org  |  www.aocs.org/LabService

For more resources on processing contaminants, visit www.aocs.org/3mcpd
Describe your career path.

I earned an undergraduate degree in biochemistry from the University of Nigeria. I then moved to the United States and earned a master’s degree in biochemistry at Washington State University (WSU; Pullman, Washington), working on terpene biosynthesis. At that point I could have gone to work in the flavor and fragrance or cosmetic industry and earned a little more than other biochemists, but I wanted to complete my Ph.D. first. Thus, I accepted a graduate assistantship in the Department of Food Science & Human Nutrition at WSU to study the chemical synthesis of reduced-calorie and zero-calorie fat substitutes.

The challenge was to create a product not infringing on the Procter & Gamble patent on sucrose fatty acid polyesters (Olestra™) intended for use as fat substitutes. We successfully produced other lipid molecules that can serve as fat substitutes based on chemical interesterification reactions between the component carbohydrates and various fatty acid ester mixes from vegetable oils and fats. The satisfaction that came with this accomplishment gave me the motivation to always try to do something new, to explore, to be creative, and to contribute to knowledge.

The two years I spent doing postdoctoral research in nutritional biochemistry before joining the University of Georgia faculty helped balance my research prospectus and gave me deeper understanding of the implications of my research.

What do you love about your job?

I enjoy the freedom to determine what type of research to pursue, the enabling environment, and the knowledge that my contributions will somehow improve the consumer’s quality of food and health. I have a boss, but in some ways I am my own boss. It’s quite fulfilling to interact with students from all over the world, learn from them, impact their knowledge, challenge them to think, and help them succeed. Traveling to other countries to help students and faculty design research, execute their plans, and publish their findings is very rewarding.

What is the biggest challenge you have encountered in your career and how did you address it?

My decision to change from basic biochemical research to applied chemistry in a food science department was difficult. I figured I could apply all the organic chemistry and biochemistry basics I had acquired to solve food science problems and benefit mankind. That move and my fundamental background in chemistry and biochemistry were instrumental to my successful career.

Why did you join AOCS?

I joined AOCS in 1985 for professional enrichment and to advance my career. AOCS offered me opportunities to learn from the best scientists in my profession, to grow, to network, and to serve society.

Professional Pathways is a regular Inform column in which AOCS members discuss their professional experiences and share advice with young professionals who are establishing their own careers in oils and fats-related fields.

Casimir C. Akoh is a distinguished research professor in the Department of Food Science at the University of Georgia (Athens, USA). Akoh was president of AOCS from 2008–2009 during its centennial year.
How has your industry changed since you entered the field?
I do not and have not worked in industry. That being said, the fats and oils industry has been adapting to meet consumer demand for healthful foods and food ingredients. The industry has added genetic engineering to breeding to produce healthful oilseed crops, sustainable plant-based sources of omega-3 (n-3) fatty acids, and high-stearate soybean oil. Frying and cooking with lard and tallow have given way to the use of highly stable monounsaturated oils. Trans-fat has been essentially banned in most countries, and blending and interesterification are accepted alternatives. Enzymatic modification of lipids to produce trans-free fat alternatives, cocoa butter, infant formula fat analogs, specialty fats, and healthful and functional lipids is now accepted by the industry. Indeed, industries are investing a lot of dollars to support the development of such fats and oils. Even animals foods (pet foods) are designed with healthful lipids.

Do you have any advice for those looking to enter your field?
There are a lot of job opportunities in the fats and oils industry for someone with a background or education similar to mine. Master the basic sciences such as chemistry, biochemistry, biology, nutrition, physics, and mathematics. Have an understanding of the social sciences (e.g., sociology), the English language, logic, and economics.

Start early in life: volunteer for service, join professional societies as a student, network, and identify a mentor or someone in your field or life to look up to. Ask questions when in doubt, work hard, define your targets, and try new things.

Be confident in yourself, trust your capabilities, work on your limitations, and self-motivate by being passionate in whatever you do. Be willing to try something new most of the time. Believe that hard work will eventually pay dividends. Remember that nothing good comes easy. Have a long-term goal and many sequential short-term achievable milestones. Be forward-looking and positive. Have self-discipline and humility. Give back to your profession and help others along the way. Apply for things you qualify for. I believe that anything worth doing is worth doing well. If you accomplish most of these things and possess some of these qualities—you will succeed in this field or any field.

I urge you to upgrade to active membership in AOCS after your student membership expires and remain active in the organization.

How do you see the industry changing in the next five years?
I can see more outsourcing or collaborative research and integrated problem solving between industry, academia, and even government. No one entity can solve all the problems posed by research, manufacturing, processing, quality, and safety or by a demanding consumer. Communication between the players needs to improve, and there should be no more lack of communication across disciplines. I see more team efforts across complementary disciplines and individual skills. I also envision global partnerships.

Describe a memorable job experience.
I remember receiving reviews back for a few of my graduate student research manuscripts submitted to the *Journal of the American Oil Chemists’ Society*, with the phrase “Accept as Is.” You cannot imagine what that did to boost my confidence and my motivation to continue to improve as a researcher and a writer. Serving as AOCS president during the celebration of the 100th anniversary of AOCS was the hallmark of my service to our profession and will be cherished forever. I owe my career and achievements to my involvement, mentoring (informal), and the nurturing environment of the AOCS community.

Please describe a course, seminar, book, mentor, or speaker that has inspired you in ways that have helped you advance your career.
It is difficult to point at one event that inspired me. Rather, I will describe few events and people that helped me advance in my career. Growing up, I had a neighbor who was a professor of mathematics and later became a university vice-chancellor. I admired his lifestyle and achievements and always looked up to him.

I owe a lot of my success to the opportunity given to me by a visiting professor from the United States, Dr. R.H. Filby, from the WSU chemistry department. He encouraged me to apply to his university for graduate studies in biochemistry when we met at the University of Port Harcourt, Nigeria, where I was newly employed.

My Ph.D. advisor, Dr. Barry G. Swanson, played a big role in my career. He guided me and gave me the freedom to design my research experiments, the opportunity to be creative, and the responsibility to write manuscripts and respond to reviewers’ comments with minimal supervision. I learned how to be an independent researcher by doing it myself.

Last and not least are my parents. Even though they did not have college degrees, they knew the value of education. They encouraged me to always strive to be the best, have self-discipline, be humble, and never settle for mediocrity.

Do you have any advice for young professionals who are trying to develop an effective network of other professionals?
I would encourage young professionals to make use of any available opportunity to attend meetings of organizations in
their profession, make presentations, meet people at these meetings, and introduce themselves or have someone introduce them to their friends, and network. I advise students to attend division meetings and student activities, and to volunteer to serve as an elected officer. Also, endeavor to meet with potential future employers from industry and government and make connections. Follow up after annual meetings by dropping them a line, and always keep in touch with your network and your major professor. If you are with the government or industry, request and justify the need to your supervisor to allow you to attend at least one professional meeting a year.

If you were starting your career again, what would you do differently?

I have had a rewarding and fulfilling career and would not change anything. One regret, though, is that I did not have the opportunity to intern in industry. However, I do interact and consult with the industry. I did do sabbatical work with the government, and that gave me another perspective as I look at my career and network.

What are the opportunities for advancement in your career/field, and how can someone qualify for such advancements?

There are many opportunities for advancement, but you have to seek and take them as they present themselves. Active participation in your profession opens many doors through networking. Job opportunities are available at entry level in many industries if you are a fresh graduate with little or no experience. Having an internship with potential industry employers during your university days is a plus and will help in securing a job. Through the network you build over the years, you can also move from one industry job to another or from academia to government or industry and vice versa. The way to qualify for these positions is to start early and build your resume with professional activities, publications, patents, and society service.

How would you describe the culture in your field, and how has it developed?

In academics, the culture has been: publish peer reviewed papers, attract extramural grants, train graduate students, be a good teacher, and be internationally recognized in your field—or perish. In the early 1970s to late 1990s in the United States, state and federal governments supported teaching, research, and extension education relatively well. Following the economic downturn in 2008, federal government and industry are cutting financial support and grants to the universities, making it difficult for young assistant professors to truly establish themselves as internationally recognized experts in their field. The amount of individual investigator-initiated research grants from the government has decreased. Instead, interdisciplinary, multi-investigator, multi-university, collaborative research grant applications are encouraged and awarded. It sounds good politically (and it may make sense) to say that public funds were used to fund many investigators in many universities to comprehensively solve a research problem. Often the funds that were allocated to the actual experiments are small due to the administrative costs to the principal investigator and the various institutions involved.

In your area/field and considering today’s market, is it more important to be well-rounded or a specialist?

It is important to specialize and have a focus while building a general knowledge of other fields. The key is flexibility and adaptability. The research questions and priorities of funding agencies change with time, as do those of industry or government. Employers would prefer to do minimal retraining in order to switch their current employee to a new and closely related area rather than to hire a new specialist at a higher cost. The same goes with academia; professors pursue research dollars based on the priorities set by the funding agencies for that year. If you are not able to adapt and prepare a proposal addressing one of the priority areas, you will be left in the cold for that grant cycle. Quick thinking and flexibility are desirable assets to a specialist tool box.

What is your opinion toward the value of obtaining or possessing a graduate degree during a challenging economy?

You can never go wrong with or regret having a graduate degree at any time, challenging economy notwithstanding. Knowing that you have the educational qualifications to exit or enter the job market bring joy and internal peace. First, the pay scale is different between a B.S. and an M.S. or Ph.D. degree holder. Second, the opportunity to advance your career within the same company or another company is greater with a graduate degree. It will take longer for a B.S. degree holder to reach the same rank in a company than someone with an advanced degree. The only caveat is that in a bad economy and layoffs, it may be more difficult for the graduate degree holder to obtain a new lower-paying job due to overqualification. Personally, I support having all the studies/degrees you need before entering the job market. It is more difficult to go back to school once you are already enjoying a certain lifestyle and possibly have children and a mortgage.
Seven new AOCS Fellows were inducted at the recent AOCS Annual Meeting & Expo in San Antonio, Texas, USA. They join a select group of veteran AOCS members whose achievements in science entitle them to exceptional recognition, or who have rendered unusually important service to the Society or to the profession. The seven are Moghis U. Ahmad, John L. Harwood, Rodney J. Mailer, F. Xavier Malcata, W. David Nes, Ian Purtle, and Randall J. Weselake.

Moghis U. Ahmad is a distinguished lipid chemist and currently vice president, Chemical Technology and Manufacturing, at Jina Pharmaceuticals Inc. in Libertyville, Illinois, USA. He has contributed to the field of lipid chemistry in numerous ways, including through the search for new industrial oils, new product development, process research and scale-up, and large-scale synthesis of lipid products. He has more than 50 peer-reviewed publications and 33 patents and patent applications. He has discovered and marketed new lipid products for chemical, pharmaceutical, and biotechnological applications. His expertise in the pharmaceutical industry and in the field of phospholipids brings together the latest cutting-edge technologies to satisfy the needs of both consumers and the health industries.

Ahmad, who is also a Fellow of the Royal Society of Chemistry, has been a member of AOCS since 1979. He has served on a number of committees within the Society, as editor and contributing author for AOCS Press books, as a session organizer, and as a member of the Phospholipid Division leadership team, culminating in his work as chairperson in 2010.

John L. Harwood, deputy director of the School of Biosciences, Cardiff University, Wales, UK, is an internationally known and respected lipid scientist. He has had major impact on our understanding of many aspects of plant lipid biochemistry. Whereas plants have been his primary interest, Harwood has also made important contributions to lipid biochemistry in humans, animals, and microbes. Some of his work has had application within medicine, such as his examination of the role of surfactants in respiratory distress, of the mechanisms of endotoxic shock, and of new treatments for malaria. More recently, his lab has been engaged in research on the role of n-3 polyunsaturated fatty acids in alleviating arthritis, dementia, and cardiovascular disease. He has also made significant contributions to research into the role of lipids in the adaptation to environmental stress.

Harwood has been a member since 1999 and has served as a committee member for the European Section, has presented numerous papers at AOCS meetings, and has written two articles for Inform. At present, he is joint editor-in-chief of The AOCS Lipid Library. Harwood is an elected Fellow of the Learned Society of Wales (2012), an honorary member of the Hungarian Academy of Sciences (2010), and is an elected Fellow of the International Society of Biocatalysis and Biotechnology (2013). He has received the AOCS Supelco/Nicholas Pelick-AOCS Research Award (2011), the ISF International Award (1999), the Terry Galliard Medal for plant lipid biochemistry (1998), the Phytochemical Society of Europe award for a younger European scientist (1990), and is the 2014 Euro Fed Lipid Chevreul Medal awardee.

Rodney J. Mailer of Australian Oils Research in New South Wales, Australia, is an internationally recognized expert on rapeseed and olive oils. He has been a principal research scientist with the NSW Department of Agriculture, where he was responsible for establishing the Australian Oils Research Laboratory—the central oilseed and olive research facility in Australia. He was also associated with canola breeding and quality improvement programs that released 22 canola cultivars for commercial use. His recent research has been instrumental in developing analytical methods and standards to ensure that high-quality extra virgin olive oils reach the international market.

Mailer has been a member since 1983, was one of the founding members of the Australasian Section and served as that section’s first president, and has been active on the AOCS Olive Oil Expert Panel. He was president of the International Society for Fat Research 2007–2009) and is an elected Fellow of the Royal Swedish Academy of Agriculture and Forestry (2012), an honorary member of the Australian Olive Association, and a life member of the Australian Oilseed Federation. He has also received the Farrer Memorial Trust Medal (2009; Farrer Memorial Research Scholarship Fund) for distinguished service in agricultural science, and the Timothy Mounts Award (2008, AOCS).

CONTINUED ON NEXT PAGE
F. Xavier Malcata, director of the integrated masters (five-year program) on bioengineering, College of Engineering, University of Porto, Portugal, has generated an enormous number of original research publications, and has had a productive and noteworthy research career.

He has more than 400 journal publications, has edited or co-edited more than 45 book chapters, and has edited or co-written 15 books. His work focuses primarily on basic and applied biotechnology pertaining to oils and fats, and his research has produced many innovative ideas on reactor design and characterization, membrane systems, enzyme applications in dairy products, and bioprocessing of lipids using microalgae. His research has contributed significant advances to lipid biochemistry, lipid enzymology, and lipid transformation.

Malcata has been an AOCS member since 1991 and has received the AOCS Young Scientist Research Award (2001) and the Ralph Potts Memorial Award (1991). He has also received the Foundation Scholar Award in Dairy Foods, the Danisco International Dairy Science Award and the Distinguished Service Award (American Dairy Science Assoc.; ADSA), the Canadian & International Constituency Investigator Award in Physical Sciences and Engineering (Sigma Xi), the Scientist of the Year Award (European Federation of Food Science and Technology), and the International Leadership Award and the Elmer Marth Award (International Association for Food Protection). He is also a Fellow of ADSA, the Institute of Food Technologists, and the International Academy of Food Science and Technology.

W. David Nes, director, Center for Chemical Biology and Paul Whitfield Horn Professor at Texas Tech University, Lubbock, USA, is considered to be one of the foremost international authorities on sterols, sterol biosynthesis, and lipid metabolism. His research is a combination of organic chemistry, the refined analysis of steroids and their derivatives, which is now united with a deep understanding of biological processes in plants, microorganisms and parasites, including the use of modern methods in molecular biology to elucidate enzymatic reaction mechanisms, and the rational design of mechanism-based inhibitors.

Nes originally joined AOCS in 1979 and in 2002 began co-chairing the AOCS Sterol Symposium Series. Furthermore, David has served as an associate editor on a number of journals including *Lipids* (1986–1993), *Inform* (1989–1999), and *JAOCS* (1987–1988). He has tenure at the National Science Foundation as program director of metabolic engineering/

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**Supelco/Nicholas Pelick—AOCS Research Award**

Since 1964, AOCS has recognized scientists, researchers, engineers, and others for their original research in fats, oils, lipid chemistry, or biochemistry.

Candidates must be individuals who are actively associated with research, and who have made discoveries that have influenced his or her field of endeavor. In addition, candidates must have published technical papers of high quality.

**Award recipient will receive:**

- $10,000 honorarium
- a plaque
- travel-and-expense allowance to attend the AOCS Annual Meeting & Expo to present the award address

For nomination procedures, deadlines, and full award details, visit: [www.aocs.org/awards](http://www.aocs.org/awards).

*Sponsored by Supelco, a subsidiary of Sigma Aldrich Corp, and Nicholas Pelick, a past president of AOCS.*
molecular biochemistry in the Molecular and Cellular Biosciences Division.

Ian Purtle, consultant and retired vice president, Cargill, Wayzata, Minnesota, USA, is responsible for shaping much of the philosophy and programs that have contributed to the recent success of the Society. His global experience, technical expertise, and commercial insight have helped AOCS better serve its members’ needs in oilseed production, biofuels, energy conservation, and bio-industrial products.

He served on numerous committees and held positions in AOCS including as Governing Board president (2009) during the Society’s 100th-year jubilee. As Technical Steering Committee chairperson (2002–2008), his guidance, vision, and counsel led to the growth of the technical services area. This growth has helped establish the AOCS brand throughout the world, thus strengthening other AOCS programs as well.

Randall J. Weselake, professor, Canada Research Chair in Agricultural Lipid Biotechnology and scientific director of the Alberta Innovates Phytola Centre, University of Alberta, Canada, has distinguished himself as a lipid biochemist and contributed to our understanding of the enzymology and molecular biology of storage lipid formation in plants, yeast, and mammals. He is author or co-author of over 140 papers in peer-reviewed journals and books, and over 200 conference abstracts. He is a recipient of the Imperial Oil Limited Research Excellence Award and the University of Lethbridge Board of Governors Research Chair. In 2011, he received the distinction of Academician/Fellow of ISBAB (the International Society of Biocatalysis and Agricultural Biotechnology) and was selected as an ASTech Honoree by the Alberta Science and Technology (ASTech) Leadership Foundation in the category of “Outstanding Leadership in Alberta Science.”

Weselake has been an AOCS member since 1989. He has served on numerous committees and was also involved in Section activities and Society governance. He was president of the Canadian Section (2003–2005), chairperson of the Biotechnology Division (2011–2012), Governing Board member-at-large (2004–2008), Technical Program chairperson for the 2002 AOCS Annual Meeting & Expo, and has been an associate editor of Lipids since 2005. At present, he is joint editor-in-chief of The AOCS Lipid Library.

Young Scientist Research Award

Call for Nominations

Sponsored by the International Food Science Center A/S.

Do you know a young scientist who should be recognized for their outstanding research contributions?

We invite you to nominate yourself or a colleague who meets the following criteria:

By June 1, 2015, the candidate must be:
- Younger than 36 years of age
- OR have received his/her highest degree within the previous 10 years

Additionally, the candidate’s research must have had a significant impact on advances in one of the areas represented by AOCS’s eleven Divisions.

Award recipient will receive:
- $1,000 honorarium
- a plaque
- travel-and-expense allowance to attend the AOCS Annual Meeting & Expo to present the award address

For nomination procedures, deadlines, and full award details, visit: www.aocs.org/awards.
Systematic investigations in the antimicrobial efficacy of glycerine esters with fatty acids of different chain length

Marion Kaß, Sonja Lüthje, Sabine Herweg, and Sven Stepan

Formulators are aware of the necessity to adequately preserve their products in order to ensure product safety and be in compliance with the respective legislations although the regulatory framework differs regionally. Whereas in the EU (1), Japan (2), and the ASEAN (3) countries preservatives are listed in positive lists, the United States (4) does not regulate preservatives in that way. However, every cosmetic manufacturer has the legal responsibility to ensure the safety of its products.

But not only legislation limits the number of accepted preservative actives. More and more public discussions on preservative actives that have been evaluated as safe for use in cosmetics influence marketing requirements, resulting in demands for claims such as globally approved, soft preservation, “free of,” and the like. Due to this situation cosmetic formulators try to avoid using these discussed actives and start looking for new possibilities to keep their products free of antimicrobial growth.

Hurdle technology—which includes good manufacturing practice (GMP), appropriate packaging, water activity, as well as pH control—and the intelligent combination of multi-functional additives can help to design microbiologically stable products (5, 6).

The addition of multifunctional additives is becoming especially more and more popular. Multifunctional additives in this sense are cosmetic ingredients that have some antimicrobial efficacy besides their main purpose as a surfactant, emulsifier, emollient, and the like. Among those are alcohols, glycols, and glycerine ethers as well as fatty acids and their corresponding esters (5–9).

Glycerine esters with fatty acids of different chain length

Partial glycerides are an important class of emulsifiers for the cosmetic industry. Frequently partial glycerides also occur as secondary ingredients in other emulsifiers (10).

Partial glycerides are easily accessible raw materials, which can be produced via the processes enumerated in Table 1.

Another newer process that should be mentioned is the synthesis of partial glycerides by enzyme catalysis (11).

Partial glycerides perform many different functions over a wide application range in cosmetic formulations. Certainly the most frequently used long-chain partial glyceride is glyceryl stearate, which is used as a consistency-modifying co-emulsifier in emulsions. Long-chain partial glycerides, such as glyceryl oleate, are also used in cosmetic cleansers as a so-called refatting agent.

Medium-chain partial glycerides (chain length $C_8$–$C_{12}$) are found less often in cosmetics. Partial glycerides having a chain length of $<C_8$ are found only in pharmaceutical applications, as...
use in cosmetic formulations is not recommended due to the characteristic odor of caproic acid.

It is often more difficult to formulate with medium-chain partial glycerides because of their large influence on the emulsifier system, due to their high hydrophilicity. However, this can also be a desired characteristic of medium-chain partial glycerides because cost-effective significant changes in the polarity of the oil phase can be achieved by using only small amounts. It can also be an advantage for the solubility of actives, ultraviolet filters, and other ingredients with polar structures (12).

It should be noted that ethylhexyl triazone has a higher solubility in a mixture of caprylic/capric glycerides (IMWITOR® 742) and caprylic/capric triglyceride (MIGLYOL® 812) than in each of the individual substances.

Actives in cosmetic formulations can only become active when they are at least able to penetrate the stratum corneum. For this purpose it is important that they are already dissolved and penetrate the stratum corneum via suitable enhancers. Medium-chain partial glycerides, as illustrated in Table 2 (page 392), are representatives of typical chemical enhancers.

Caprylic/capric glycerides are suitable to achieve optimized hydrophilicity for solubilizing very hydrophilic actives in lipophilic oils.

In addition to their already described solubility effects, medium-chain partial glycerides are easy to incorporate into surfactant systems, which lead to a thickening of SLES [sodium laureth sulfate]/betaine systems. Besides thickening, positive effects on the foam have been observed, which has finer bubbles and is creamier.

Medium-chain partial glycerides are small surface-active molecules and are therefore able to stabilize foam. Stabilization of the foam is concentration dependent and passes through a maximum. Above this maximum, destabilization of the foam can result, which is explained by the Gibbs-Marangoni effect.

**ANTIMICROBIAL EFFICACY TESTS**

The monoglyceride esters of the fatty acids caprylic acid (glyceryl caprylate) and capric acid (glyceryl caprate) are well-known emulsifiers for cosmetic formulations. In addition both have a certain antimicrobial efficacy that is already used in combination with other multifunctional additives to sufficiently stabilize cosmetic formulations (5,6).

To effectively develop new intelligent combinations of multifunctional additives to keep cosmetic products antimicrobially stable, it is important to understand how current systems work. This work shows exemplarily the antimicrobial efficacy of glycerine esters depending on their chain length \((C_6–C_{12})\) as well as their degree of esterification.

An overview of the tested glycerides is given in Table 2. For all substances, antimicrobial efficacy tests were performed. A range of five different monoesters with increasing chain length was used. Additionally the two well-known monoglycerides with a chain length of 8 and 10 carbon atoms were tested in a mixture with their diglycerides. The monoglyceride content of each glyceride is shown, along with the diglyceride content.
for the mixtures. The deviations in the monoester content are related to the synthesis conditions and certainly have an influence on the antimicrobial efficacy. But the range of deviations allows an estimation of the trend of the antimicrobial properties with regard to chain length.

To investigate the antimicrobial efficacy, a germ count reduction test with four different germs was performed. The tested germs are shown in Table 3.

For the determination of the germ count 25 mL portions of the samples were inoculated with 0.1 mL microorganism suspension (the initial microorganism count is given in Table 3) and stirred.

The solutions were streaked out onto tryptone soya agar or Sabouraud-dextrose 4% after 24, 72, 168, and 336 hours. The cultures were incubated for 48 hours at 37°C, except for Aspergillus brasiliensis, which was incubated for 72 hours at 25–27°C. The evaluation was made on the basis of semiquantitative assessment of the microbial growth of the streaks.

The germ count reduction tests were performed in a simple oil-in-water lotion according to the formulation listed in Table 4. The use-concentration of the actives in the oil-in-water lotion was varied from one to two percent.

Almost all of the partial glycerides showed good antimicrobial efficacy in our tests. Only the glyceride with the longest chain (lauric acid monoglyceride: C₁₂) showed insufficient activity. This effect may be due to the poor water solubility and the resulting limited availability as a preservative in the formulation used.

The efficacy of the partial glycerides against molds increases with the chain length. A shorter chain length coincides with better efficacy against bacteria. Thus, as a single active the middle chain length (caprylic acid monoglyceride: C₈) exhibits the best efficacy against all germs. The C₆ monoglyceride (capric acid monoglyceride) alone shows a significant and reproducible weak effect against gram-negative bacteria (Pseudomonas aeruginosa). This effect is observed even in a higher use-concentration. The C₈ monoglyceride (caprylic acid monoglyceride) has a low efficacy against molds (A. brasiliensis). In this case a higher use-concentration leads to better efficacy. For maximum effectiveness one can combine the monoglycerides with the C₈ and C₁₀ chain. A caprylic/capric acid monoglyceride mixture showed an optimum efficacy and was sufficiently effective against all tested germs in a use-concentration of 1%.

To compare the antimicrobial efficacy of corresponding mono- and diglyceride, mixtures with different contents of mono- and diester have been tested. The results are shown in Figure 3, and they demonstrate a better efficacy of mixtures with a higher content of monooest.
Due to their nature as an emulsifier, all partial glycerides exhibited an influence on emulsion stability. In application, this can be achieved if the monoglyceride is incorporated in an early stage of the formulation development. Then the emulsifying and antimicrobial properties can be skillfully combined.

The shorter the chain length of the monoglyceride, the better is the antimicrobial efficacy against bacteria. The efficacy against mold increases with longer chain length. At a certain point the chain length limits the antimicrobial effectiveness related to solubility. In general, a high monoglyceride content increases efficacy. A caprylic/capric acid monoglyceride combination (C8/C10) exhibits the best antimicrobial efficacy, especially in relation to the use-concentration.

Marion Kaß, Sonja Lüthje, and Sabine Herweg are with Schulke & Mayr GmbH in Norderstedt, Germany. They can be contacted at sai@schuelke.com. Sven Stepan is with Cremer Oleo GmbH & Co. KG in Witten, Germany. He can be reached at sven.stepan@cremer.de.

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**TABLE 4. Composition of the oil-in-water (O/W) lotion that was used as test basis**

<table>
<thead>
<tr>
<th>O/W lotion</th>
<th>INCI name</th>
<th>Trade name</th>
<th>Function</th>
<th>% w/w</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Phase A</strong></td>
<td>Arachidylalcohol, behenyl alcohol, arachidyl glucoside</td>
<td>Montanov 202&lt;sup&gt;a&lt;/sup&gt;</td>
<td>Emulsifier</td>
<td>5.00</td>
</tr>
<tr>
<td></td>
<td>Cetearyl ethylhexanoate</td>
<td>Lanol 1688&lt;sup&gt;b&lt;/sup&gt;</td>
<td>Emollient</td>
<td>20.00</td>
</tr>
<tr>
<td><strong>Phase B</strong></td>
<td>Aqua</td>
<td></td>
<td></td>
<td>Ad 100</td>
</tr>
<tr>
<td></td>
<td>Glycerin</td>
<td>Glycerol 85%&lt;sup&gt;b&lt;/sup&gt;</td>
<td>Humectant</td>
<td>9.40</td>
</tr>
<tr>
<td></td>
<td>Monoglyceride</td>
<td></td>
<td></td>
<td>x %</td>
</tr>
<tr>
<td><strong>Phase C</strong></td>
<td>Polysorbate 13, polyisobutene, polysorbate 20</td>
<td>Sepiplus 400&lt;sup&gt;a&lt;/sup&gt;</td>
<td>Viscosity control</td>
<td>0.20</td>
</tr>
</tbody>
</table>

Procedure: Heat phase A to 85°C and B to 80°C. Add phase C to phase A while stirring until homogeneity. Add this mixture to phase B while stirring. Homogenize at 65°C. Cool down to approximately 30°C and homogenize again for a short time. The glycerides were added to phase B in the respective concentration.

<sup>a</sup> Seppic.
<sup>b</sup> Oleon.

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General comparison of health claims with regards to food and supplement legislation frameworks in Europe, the United States, and Canada

Nigel Baldwin and Theresa Poon

In the EU, it is important point to note that, while supplements have their own legislation for basic presentation, vitamins and minerals lists, warning labels and the like, they are fundamentally considered to be foods. This means that the same legislation that applies to the manufacture, recommended daily allowances, nutritional labeling, and packaging font sizes in foods also applies to supplements. For example, Regulation 1924/2006 on health and nutrition claims on foods (1) applies to supplements, just as it does to foods.

Much has been written about the nutrition and health claims regulation. Essentially there are three types of claims.

1. Article 13-related claims are about maintenance or support of healthy physiology or psychological function.
   a. Article 13.3 applies to grandfathered products.
   b. Article 13.5 applies to new full submissions.
2. Article 14.1(a)-related claims refer to disease risk (factor) reduction.
3. Article 14.1(b)-related claims refer specifically to children’s development and health (a general population claim that also includes children is Article 13 in most cases).

Now that the dust has settled and the “grandfathering” of existing claims is more or less complete, we are left with a submission process and a positive list of health claims. To get on the list, you make a dossier and submit via a Member State. Then the European Food Safety Authority (EFSA) reviews and delivers an opinion. This is followed by a one-month public comment period before the dossier is either refused or approved; in the latter case the dossier becomes an approved claim that is adopted into EU legislation and added to the Commission database. In theory, you can withdraw before your claim is refused, but legally you have to do this before the EFSA Panel on Dietetic Products, Nutrition and Allergies (NDA Panel) reaches an opinion, and often you do not know whether the decision is positive or negative until its adoption. If you want to see the EFSA claims “Register” for yourself and read things in more detail, review the Commission’s web page (2); and for more details on EFSA’s approach, have a look at EFSA’s very informative site (3).

Reference to general, nonspecific health claims is acceptable only if used in conjunction with an authorized health claim. Also, endorsement by charities and medical doctors is severely restricted. To avoid misleading consumers, food business operators have the responsibility to demonstrate the link between the general, nonspecific benefits of the food and
the specific, accompanying, permitted health claim. In December 2012, representatives from 17 of the 28 Member States met to discuss recommendations for the general principles on flexibility in claim wording. Among the recommendations agreed upon are (i) alternate claim wording must have the same meaning to the consumer as the authorized claim wording and (ii) the health claim must clearly link the proper food constituent to the health claim. For examples of EU health claims under Regulation 1924/2006, see Table 1.

The success rate for approval of specific and general health claims has not been very high but is improving as dossiers of higher quality have been submitted and as the guidance has improved. Currently, claims that are “on hold” include claims on caffeine, because EFSA has been asked to review the safety implications of approving them, and on “botanicals” (i.e., “food herbals”), because the Commission and Member States are still trying to decide and agree on an alternative (if there is one) to the currently pure scientific review process to allow for traditional use to be taken into account. In the meantime, claims are also subject to standard EU claims law, which means that, on a case-by-case basis, they must not be judged to mislead the consumer to a material degree.

In the United States, a “dietary supplement” is regulated separately from a food, but, for the most part, claims rules apply to both product categories. However, where the EU has Article 13.3 or 13.5 “health claims”, the United States has “structure/function claims”; and where the EU has Article 14.1 (a) disease risk (factor) reduction claims, the United States has disease (direct not factor) risk reduction health claims that are referred to as authorized/notified health claims or qualified health claims.

Structure/function claims generally do not need presubmission approval, apart from the requirement for notification to US Food and Drug Administration (FDA) within 30 days of first marketing of dietary supplements. FDA does not check the science of the claim (as that is the responsibility of the marketer, but FDA does check to ensure that the claim does not contravene medicines and other relevant consumer legislation). Structure/function claims must be based on “competent and reliable scientific evidence” and must:
1. Describe the role of a nutrient or dietary ingredient intended to affect normal structure or function in humans, or

### Table 1. Examples of European Union health claims under Regulation (EC) No 1924/2006

<table>
<thead>
<tr>
<th>Article</th>
<th>Claim</th>
</tr>
</thead>
<tbody>
<tr>
<td>Article 13.1</td>
<td>Beta-glucans contribute to the maintenance of normal blood cholesterol levels.</td>
</tr>
<tr>
<td>Article 13.5 (proprietary)</td>
<td>Water-Soluble Tomato Concentrate (WSTC) helps maintain normal platelet aggregation, which contributes to healthy blood flow&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
<tr>
<td>Article 14.1a (disease risk factor reduction)</td>
<td>Barley beta-glucans have been shown to lower/reduce blood cholesterol. High cholesterol is a risk factor in the development of coronary heart disease&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>Article 14.1b (children)</td>
<td>Docosahexaenoic acid (DHA) maternal intake contributes to the normal brain development of the fetus and breastfed infants.</td>
</tr>
</tbody>
</table>


2. Characterize the action by which a nutrient or dietary ingredient maintains such structure or function.

The following disclaimer is required on dietary supplements that bear structure/function claims: “This statement has not been evaluated by the Food and Drug Administration. This product is not intended to diagnose, treat, cure, or prevent any disease.”

For more information on structure function claims, for both dietary supplements and foods, FDA offers an excellent webpage (4).

Health claims that are authorized by the FDA under the Nutritional Labeling and Education Act (NLEA) or notified to the FDA under Food and Drug Administration Modernization Act (FDAMA) must demonstrate by submission to FDA that the level of scientific substantiation meets the standard of “significant scientific agreement” (SSA). Further information is available on the FDA website (5). An example of an authorized health claim is “Diets containing foods that are good sources of potassium and low in sodium may reduce the risk of high blood pressure and stroke” (6).

Qualified health claims are those for which the totality of scientific evidence falls short of the SSA standard but that are supported by credible scientific evidence; thus, qualifying language is used to convey to the consumer the level of scientific evidence in support of the health claim. Historically, qualified health claims have been of reduced value because of limited consumer understanding of claim wording. For example, “Supportive but not conclusive research shows that eating 1.5 ounces per day of walnuts, as part of a low saturated-fat and low-cholesterol diet, and not resulting in increased caloric intake, may reduce the risk of coronary heart disease. See nutrition information for fat content” (7).

Overall, there has been a greater success rate for approval of health claims in the United States than in the EU.

For Canada, the situation regarding “health claims” is complicated by the fact that supplements are called “Natural Health Products” (NHPs), which are regulated more or less as medicines on a product-by-product basis via a licensing procedure. Again, there is a distinction between function claims (referred to as “nutrient function or other function claims”) and disease risk reduction claims. Disease risk reduction and therapeutic claims can be made on foods or NHPs. In addition, even stronger claims, such as diagnostic, prevention, and cure claims can be made for NHPs, as shown in Table 2. All health claims on NHPs require full submission and scientific review by the Natural Health Products Directorate of Health Canada. For more information on health claims for foods and NHPs, please see web pages for the Health Canada and the Natural Health Products Directorates (8,9).

<table>
<thead>
<tr>
<th>Claim type</th>
<th>Definitions</th>
</tr>
</thead>
<tbody>
<tr>
<td>General health</td>
<td>Describe the effect of a medicinal ingredient on restoration, correction, or modification of a structure or physiological function in the human body in a manner that maintains, supports, or promotes health.</td>
</tr>
<tr>
<td>Antioxidant</td>
<td>For products with at least one medicinal ingredient that has antioxidant properties; should be worded as general health support claims or as “source of” claims.</td>
</tr>
<tr>
<td>Diagnostic</td>
<td>Relate to the diagnosis of a disease, disorder, or abnormal physical state or its symptoms in humans.</td>
</tr>
<tr>
<td>Risk reduction</td>
<td>Based on significantly altering a major risk factor(s) for a disease or health-related condition; should ensure that consumers do not interpret as prevention claims.</td>
</tr>
<tr>
<td>Prevention</td>
<td>Relate to interventions which are proven to significantly reduce the incidence of the disease.</td>
</tr>
<tr>
<td>Treatment</td>
<td>Relate to the treatment or partial treatment and mitigation of a disease, disorder, or abnormal physical state or its symptoms in humans.</td>
</tr>
<tr>
<td>Cure</td>
<td>Describe a therapeutic effect that results in the elimination of a disease, disorder, or abnormal physical state in humans, either permanently or for a significant length of time.</td>
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Having reviewed the three jurisdictions, we can summarize the different approaches as follows:

1. For nondisease-related claims, the EU specifically approves generic claims based on ingredients, the

CONTINUED ON PAGE 400
Is the art of HPLC method development dead?

Scott Fletcher

Please believe me when I say that I take no pleasure in asking this question but I feel I must, given the experience I had during my ten years working as a method developer in the pharmaceutical industry and last six years as a professional trainer in analytical chemistry.

For me, the main benefit of (reversed phase) HPLC is its selectivity – hydrophobic (non-polar), hydrophilic (polar), ionisable and ionic compounds can all be separated (under certain conditions albeit rarely at the same time). Very similar compounds that only differ in fairly subtle aspects can be relatively easily separated, identified and then quantified. The reasons behind this almost universal applicability are the many factors that can be adjusted in order to affect how a

CONTINUED ON NEXT PAGE

• In many companies, high performance liquid chromatography (HPLC) has become so automated that all one has to do is prepare solutions and press go.

• While such automation leads to the development of more robust and better methods in a shorter period of time, preselected pHs and columns do not always provide the selectivity that is needed.

• Such situations require experienced method developers who have a deep understanding of the science, but will such individuals stick around in jobs where they’re filling solutions and pushing buttons most of the time, and will practicing their higher-level art so infrequently cause them to become rusty?
particular analyte will interact with both the stationary and mobile phase. A few factors that can be manipulated in order to affect a specific analyte’s retention are: polarity and composition of modifying ligand, surface coverage, endcapping, silanol confirmation, monomeric vs. polymeric ligand binding of the stationary phase and polarity, type of organic modifier, $pH$, ionic concentration, temperature for the mobile phase to name but a few. It had been thought (hoped) that with all the variables that could be controlled around the mobile phase that we would not see the proliferation of stationary phases that has blighted GC – where only stationary phase type and temperature can be manipulated to adjust selectivity. As shown in Figure 1 resolution, the ultimate goal and in most cases prerequisite of any chromatographic separation, is the culmination of retention factor ($k$), efficiency ($N$) and selectivity ($\alpha$).

At very low values retention factor has the largest impact on resolution but at even very modest values this becomes negligible with efficiency, but predominately selectivity, being the main driver.

The huge inherent efficiency which benefits capillary GC means that even very similar compounds, in terms of volatility or polarity, that possess similar selectivity values can be fully resolved from one another purely down to their efficiencies. A great many GC separations could be carried out on any number of different stationary phases. Even very modern UHPLC separations using state of the art instrumentation and column technologies cannot match the efficiencies enjoyed in GC and therefore a greater emphasis is placed on selectivity. Eli Grushka, then at the State University of New York at Buffalo who in 1974 would seem almost visionary now – discouraging the proliferation of stationary phases; rather than producing many phases all with a particular separation in mind, encouraging the understanding of a few phases that could be utilised for multiple separations [1]. The 1000’s of reversed phase columns that are currently available bear testament to his visage not coming true. The vast numbers of stationary phases currently available need to be further characterised and better designated – although there has been recent advances from manufacturers of HPLC columns to adopt are more transparent and industry accepted approach to classifying stationary phases. The Hydrophobic Subtraction Model [2] proposed by Snyder, Dolan and Carr seems to be generating significant interest and is being reviewed by the USP (PQRI approach) currently – over 650 columns, and counting, have been tested, characterised and added to this database.

An alternative is the principal component analysis approach postulated by Euerby and Petersson [3-5] based on earlier work by Tanaka [6]. These are just two amongst many approaches which have been proposed to better understand selectivity in reversed phase chromatography and to get an idea which mechanisms are most accurate requires input from ALL chromatographers.

The best definition I have ever heard for defining method development was ‘optimum resolution in the minimum time’. Method development is simply the process of making logical and informed changes to a methods conditions based on current knowledge and sound science – essentially a logical and structured ‘suck and see’ approach. Having worked in method development groups for a number of years and at various organisations, both in the public and private sector, most 29 method developers, and I include myself, will install their favourite C18 column and run a gradient from low organic to high organic at a low pH – usually in an unbuffered mobile phase. When this initial ‘method development’ does not work, in terms of resolution, peak shape or sensitivity, my heart sinks to think of the number of times analysts have reached into the column drawer or got on the phone to our favourite supplier when our tried and tested column doesn’t provide the selectivity we need. Given the vast number of options available to us in order to interrupt and affect particular analytes phase preference why do we not try these in the first instance – especially when they are so much quicker and convenient, not to mention cheaper and when we the mixture in hand contains a number of similar compounds. When I started out in method development roles I can count on one hand the number of times I really looked at my analytes before my initial screening run and selected the most appropriate column and mobile phase pH unless it was glaringly obvious. There are even fewer instances where I have looked to swap my low UV absorbing and low viscosity acetonitrile for methanol; even when prices were soaring.

So, back to the my original question, “why do I have such a downhearted view of the current state of HPLC method development in mainstream laboratories”;

1) As I have mentioned above, I feel there is a real lack of understanding and this is compounded by an environment where people see knowledge as power and do not pass on the fruits of their wisdom. More commonly, the senior analysts do not possess the understanding and then go onto to confuse the junior, eager to learn members of staff with fanciful tales of exotic interactions in order to cover up their own knowledge gap. It takes a brave scientist to put their head above the parapet and ask for a theory to be explained to them. As scientists we seem to see asking for things to be explained to us a sign of weakness and somehow indicating that we do not possess the correct knowledge for someone operating in a method development role all this time. The longer we are working in a method development environment the harder it is to ask for concepts to be explained. When I train people in method development I start off by asking what things we
can do to affect the analytes phase preference and I usually get some pretty good answers, most of them centred around the polarity of the mobile phase and stationary phase. I then follow up this by asking someone to define polarity for me – in most instances I am stared back at blankly until usually it is the newest, most junior member of staff who volunteers the correct answer, being able to recall their university / college days more readily. I do not ask this question to intimidate people or put them on the spot but merely to show the concepts behind chromatographic retention are based on sound scientific principles which are fairly basic.... when understood and properly explained. Forums are great places for people to ask questions, anonymously if they chose, of their peers without fear of ridicule or being shown up as a charlatan, masquerading in a method development role. There are also various on-line learning tools for people to develop their understanding from whatever starting point they choose at their own pace, and, if they choose, at their leisure.

2) Many companies (initially large pharmaceutical companies from my experience but now this is expanding into most other industry sectors too) have method development systems that incorporate column switching valves, a quaternary pump and are connected to chromatographic modelling software. All the method developer needs to do is vial up the solution and press go. The sample is then screened at a few preselected pHs, sometimes with different organic modifiers, over a mobile phase gradient and this is then repeated through a number of preselected columns. The data is then fed back into the modelling software which will predict the optimum conditions for the separation. Whilst I am always impressed with automation of this process it does leave me wondering what science the method developers in these groups actually do. Surely those of us still interested in the science will look for a role that will challenge us and we will be replaced with people who are satisfied to prepare solutions and press start. I am not belittling this role as it often leads to more robust and better methods being developed in a shorter period of time but what happens when the preselected pHs and columns do not provide the selectivity needed – this is when you need the experienced method developers but they have either moved on for new challenges or have become rusty as they practice the art so infrequently.

3) My main reason for some of the shoddy methods I developed was due to the time constraints given to me by my manager, which were taken from the overall company strategy which seemed to have been put in place to satisfy the shareholders. Sometimes it was an excuse, I admit, but in most instances it was because I had to develop a validation ready method containing a host of related and retained substances from the manufacturing process, degradation products and post formulation matrix in under a week. I invariably found that managers didn’t appreciate that not all methods would take the same amount of time to develop – ‘The last method only took you a couple of days, why is this one taking so much longer?’ was a rhetorical argument thrown back at me on numerous occasions. It stands to reason that the more time spent developing a method, the better chance it has of standing the test of time. An extra week or two in the method development phase is inconsequential compared to the time it would take to re-develop the method post validation, especially if the method had been filed with a regulatory body. My latter experience of Pharmaceutical Development was a prime example of time being a major factor on both, the quality of the methods developed and the calibre of people attracted to my method development group. We had adopted a milestone driven submission strategy where certain critical points had to be met at predefined time periods, irrespective of the potential impact on the quality of the method.

4) The last reason for my assumption that the art of method development is dying is due to the advent of high efficiency columns. This may sound a little contradictory, but now much higher efficiency separations are capable by employing sub-2 µm fully porous particles or modern core-shell particles of varying diameters (1 - 5µm), we seem to be relying on super efficiencies to separate complex mixtures and forgetting about selectivity. Let us not forget that the main driver behind resolution, above a very moderate retention factor (k), is selectivity. If I was still practicing method development on a regular basis I’m sure my strategy would have only differed in that I would be now employing my favourite C18 but in a smaller particle size or as a core-shell. Let us not forget that these advances should come as no real surprise as Martin and Synge [7] described how increased efficiencies could be enjoyed by reducing the size of the particle in their pivotal plate theory paper over 70 years ago. Core-shell (then called pellicular) particles, and their benefits, were first described by Hovarth and Lipsky [8,9] over 50 years ago (there is still some debate as to exact reason for their higher efficiencies but the benefits are real and can be enjoyed by anyone – even those of us without the budget for UHPLC instrumentation [10]).
Health claims (cont. from page 396)

United States relies on self-regulation and enforcement discretion, and Canada has pre-approved generic claims for foods but not for product-based claims for supplements.

2. For the EU, applicant-exclusive claims based on proprietary data are permitted.

3. For disease-related claims, all three jurisdictions require premarket approval.

4. The EU is the only jurisdiction that assigns a specific class of health claims for children.

5. The United States is the only jurisdiction where claims supported by different levels of evidence are permitted.

Arguably, the EU appears to be most rigorous overall; the United States appears to allow for the most flexibility with respect to claims; and Canada may be somewhere in the middle.

But since Canada’s NHPs are regulated as products, risk-benefit assessments are conducted and claims are approved on a case-by-case basis. This is something the EU definitely lacks because safety and efficacy assessments of new ingredients are completely separate regulatory processes. This deficit denies scientists, regulators, and consumers the ability to judge the product on the basis that it “is probably good for me” and is safe anyway.

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Scott Fletcher is technical business development manager at Crawford Scientific and Senior Tutor, CHROMacademy.

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- NTD (non tower / agglomeration) process
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- Batch / Continuous

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- Fatty Alcohols
- Methylesters
- Glycerine
- Biodiesel

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- Ethyl Alcohol
- Starch & Yeast
- Fatty Amines

INORGANIC CHEMICALS
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- Sulphuric Acid
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SUPPLEMENT

Krill sustainability

TABLE 32. Corn oil: supply, disappearance, and price, United States, 1980/81–2013/14

US EPA targeting review of 4,800 CBI claims in 2014

Statistical analysis from Mintec

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Antarctic krill are small crustaceans—like prawns or shrimps—which swim in the Southern Ocean. Species of krill occur in most oceans but it’s only around Antarctica that one species, known as Antarctic krill, plays such a dominant role in the marine ecosystem.

Antarctic krill sit in the middle of the ecosystem between the microscopic plants and animals, which form the base of the food chain, and the larger animals such as seals, penguins and whales that depend on them for food. They can reach a length of 6 cm and weigh 2 grams, so they are quite large compared to other krill species.

• The Antarctic krill fishery has been a source of controversy for several years, mainly because of the perception that it is not sustainable.

• Based on several studies and research reports, many experts have found the opposite to be true.

• This article explains why the Antarctic krill fishery is actually one of the most sustainably managed in the world.

Stephen Nicol

Antarctic krill are small crustaceans—like prawns or shrimps—which swim in the Southern Ocean. Species of krill occur in most oceans but it’s only around Antarctica that one species, known as Antarctic krill, plays such a dominant role in the marine ecosystem.

Antarctic krill sit in the middle of the ecosystem between the microscopic plants and animals, which form the base of the food chain, and the larger animals such as seals, penguins and whales that depend on them for food. They can reach a length of 6 cm and weigh 2 grams, so they are quite large compared to other krill species.
In terms of behavior, Antarctic krill swim in vast, dense swarms that can stretch for miles, making them a particularly appealing food source for larger animals such as whales. Swarming krill are also attractive to a fishery because they are large, easy to catch, and hugely abundant. In fact, there are estimated to be between 120 and 600 million tons of krill, making them among the most abundant animal species on the planet (Atkinson et al., 2009).

WHERE IT ALL BEGINS

Krill start life as microscopic eggs that are spawned to the ocean surface. They then sink to great depths to develop and hatch before swimming to the surface to begin feeding. A female krill can lay thousands of eggs, several times during the short Antarctic summer. When the newly hatched larvae arrive at the ocean surface in autumn they must eat fast, so they can survive the long ice-covered winter.

As far as feeding, krill larvae eat ice-trapped algae, using the complex under-ice habitat as a nursery ground. In spring, the ice melts and the larvae—now juveniles—are released into the open ocean where they begin to form aggregations like those of the adults.

Antarctic krill thus have a complicated life history, changing size, shape, and habitat as they grow (Nicol, 2006). They mature at two years old and can live for up to 11 years. Adult krill are now known to be capable of living anywhere in the Southern Ocean—from the very surface layer to the seafloor, and from inshore areas to the deep open ocean. Antarctic krill perform daily migrations, approaching the surface at night and remaining in the dark, deeper waters during the day.

But they are not simple animals and adopt different behaviors in response to a changing seasonal environment, staying deeper in winter and migrating offshore to spawn. Their huge schools or swarms can form or disperse unpredictably in response to subtle environmental cues, and their distribution and abundance are undoubtedly affected by currents, tides, and storms. At the same time, there are many aspects of krill behavior that remain a great mystery.

ENVIRONMENTAL CHANGE AND THE IMPACT ON KRILL

Because larval Antarctic krill are associated with the sea ice that grows during winter, they are thought to be vulnerable to any climatic changes that might affect the amount of ice that forms around the Antarctic continent annually. Krill larvae are also most sensitive to fluctuations in food availability (the adults can tolerate starvation for extended periods), and they have also been shown to be affected by increasing acidity of the ocean (Kawaguchi et al., 2013). Environmental changes that affect krill have repercussions that flow onto the rest of the ecosystem, so considerable research is under way to examine the potential effects of a warmer, more acidic ocean on the populations of krill in the Southern Ocean (Flores et al., 2012).

Antarctic krill exist in a vast area estimated to be at least 19 million km² (approx. 7 million square miles), which is more than twice the area of the United States (Atkinson et al., 2009). Because the home range of krill is so large, it has proved impossible to measure how much is out there at any one time. Further complicating matters, this region is impenetrable for much of the year due to ice cover and the fact that the Southern Ocean is the stormiest in the world. Technical problems also mean that measuring krill is an imprecise science (Nicol and Brierley, 2010). As a result, it is extremely difficult to know with any degree of certainty whether the krill population is increasing or decreasing.

ESTABLISHING THE MODERN-DAY KRILL FISHERY

It has been suggested that the removal of the Great Whales (which consumed an estimated 150 million tons of krill a year prior to their exploitation) in the last century would have allowed the krill population to explode and that this would have also allowed populations of other species, such as seals and penguins, to increase dramatically (Ballance et al., 2006). But there is little unambiguous historical data that shows this took place. Experts have also suggested that the krill population crashed in the 1980s, but again it is difficult to find supporting evidence for this theory (Atkinson et al., 2004).

Today, most of the large Antarctic animals that depend on krill as a food source are relatively healthy, with populations of species such as Adélie penguins, fur seals, and humpback whales increasing dramatically during the last 30 years. There are penguins in some areas that are in decline but this is likely the result of multiple environmental changes rather than simply krill shortages (Trivelpiece et al., 2011).

Krill population sizes vary naturally from year to year, and in extreme circumstances krill shortages at South Georgia have been shown to affect the breeding success of seals and penguins (Heywood et al., 1985). In other regions of the Antarctic it has been difficult to directly link seasonal or annual fluctuations of krill abundance to the health of seal and penguin populations. But one thing is for certain: There has been no evidence to suggest that the krill fishery is affecting the krill population to the extent that populations of whales, seals or penguins are suffering.

Due to uncertainties over krill’s total stock size, the fishery is managed using regional estimates of abundance that are very conservative, so that any management action is highly precautionary (Hewitt et al., 2004). Because measuring krill abundance directly is challenging, scientists use multiple approaches so that a realistic range of values can be determined (Atkinson et al., 2012). These approaches can include: direct estimates using nets or echosounders, determining how much krill is required by their predators, working out how much food there is to support a population of krill, and by examining the catches from the fishery.

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The krill fishery has been operating for more than 40 years. Catches peaked in the early 1980s with Japanese and Soviet vessels catching over half a million tons a year (see Fig. 1).

Today, about 200,000 tons are caught from the South West Atlantic, largely by Norwegian vessels, producing high-end aquaculture feed and krill oil supplements for human consumption. It has proved very difficult to make a marketable product from krill and harvesting it is very expensive, so the fishery has always been held in check by economic forces (Foster et al., 2011).

When the krill fishery was established there was concern that it might cause irreversible damage to the Antarctic ecosystem, so a unique international treaty was signed to ensure it would be managed using an approach that took into account the needs of the entire ecosystem. This treaty was the Convention on the Conservation of Antarctic Marine Living Resources (CCAMLR). The management of the krill fishery has been guided by its principles since the early 1980s.

In the 1970s, there was concern that the krill fishery might develop into a multi-million ton fishery and, indeed, the global precautionary catch limits (PCLs) that have been set for the krill fishery currently total over 8 million tons. However, the high level of krill catches in the early 1980s was not sustained and annual catches dropped to less than 100,000 tons in the mid-1990s. Since then, krill catches have slowly increased to around 200,000 tons per year.

**HOW (AND WHY) KRILL LIMITS ARE STRICTLY REGULATED**

While the krill fishery is the largest in the Southern Ocean, it is relatively small by world standards. In 2011 the tonnage of krill caught made it the world’s 65th largest fishery and the 7th largest crustacean fishery (FAO figures). By comparison, the top 10 fisheries in 2011 all caught more than one million tons each.

The world’s largest fishery is Peruvian anchovy, which occupies a similar ecological niche to krill but lives in a much smaller area. In this part of the world, on average, 8 million tons of anchovy are caught each year. No other commercially harvested marine species has such a large biomass, such a huge range, and such a high turnover as the krill population.

The Commission for the Conservation of Antarctic Marine Living Resources (also known as CCAMLR) was established to implement the principles of the CCAMLR Convention. It manages the krill fishery because it is responsible for marine conservation in the Antarctic region.

CCAMLR meets annually and adopts conservation measures for all the fisheries operating in the waters around Antarctica (Kock et al., 2007). For krill, the fishery is regulated through a series of measures that specify how much can be caught, where it can be caught, acceptable levels of by-catch, and other operational requirements. The amount of krill that can be caught in any one year is set through PCL, which are far more conservative than normal fishery quotas because of the difficulties in measuring the Antarctic krill biomass, the animals that depend on it as a food source, and the unique environment in which they live.

Catch limits are calculated for a particular area by working out how much krill is in that area and by determining the long-term annual yield from that area. The actual catch limit is based on a percentage of that biomass. The krill fishery is currently limited to 620,000 tons a year, or approximately 1% of the biomass, which is a highly precautionary figure when compared to other fisheries. For example, the Lenfest Forage Fish Task Force (2012) recommends that the biomass of lower trophic level species not be allowed to slip below 30–80% of
the unfished biomass depending on the level of ecosystem knowledge. CCAMLR’s allocation of less than 10% of the krill biomass to the fishery far exceeds this level of precaution.

PCLs have been set for several large areas of the Southern Ocean, totaling more than 8 million tons per year. The catch limits apply to areas where a recent survey of krill abundance was carried out (see Fig. 2). In Area 48, where most of the modern krill fishery operates, there is a PCL of 5.6 million tons.

All areas currently being fished for krill as well as most areas where fishing has been carried out in the past are now covered by these PCLs. Only very limited experimental fishing is allowed in areas where krill stocks have not yet been surveyed and where there is no established catch limit.

Catch limits are calculated using a projection from a single biomass estimate derived from independent scientific surveys using research vessels equipped with echosounders carried out in 1996, 2000, and 2006. Although annual surveys are not required to manage the krill fishery, CCAMLR takes into account changes in the krill stocks that are revealed through annual scientific research, some of which is now carried out from krill fishing vessels. Some of the catch limits have also been further subdivided into smaller areas in an effort to ensure that large portions of the annual catch do not come out of restricted areas to the detriment of the ecosystem.

A major focus of CCAMLR is to further spread out the catch so that colony-based predators such as penguins and seals are not affected by catches in their vicinity. As an added element of precaution, CCAMLR has applied a “Trigger Level” of 620,000 tons throughout the main fishing grounds (e.g., Area 48)—a level of catch that cannot be exceeded until the biomass has been evaluated and determined to be healthy enough for fishing to continue. The figure of 620,000 tons was calculated by summing the maximum annual catches from each subarea. Thus, the Trigger Level is an arbitrary limit but for each subarea it represents a level of catch that had already been reported.

Developments in krill fishery management are discussed at the annual CCAMLR meetings, and any revisions that are made to the way in which the fisheries are managed must be based on the best scientific advice available. There are 25 members of CCAMLR, including six countries that currently fish for krill, and all decisions are adopted by consensus. Additionally, the annual meetings are attended by a range of other interested parties, including environmental NGOs (non-governmental organizations), fishing industry associations, and other international bodies that have a stake in the conservation of the Antarctic ecosystem.

MAKING SURE HISTORY DOESN’T REPEAT ITSELF

Several decades ago, there was concern that the overharvesting of whales and seals in the Southern Ocean would be repeated for krill with disastrous ecological consequences. In fact, these events are precisely what led to the creation of the Convention on the Conservation of Antarctic Marine Living Resources (CCAMLR).
When it was signed in 1980, it was hailed as a ground-breaking approach to resource management because it aimed to ensure the sustainable harvesting of marine resources using an ecosystem approach. CCAMLR has since overseen the management of the krill fishery.

The conservation measures that now regulate the Antarctic krill fishery make up a regime that would be considered comprehensive and innovative in any other environment. Still, because the Antarctic is such a special place and Antarctic krill are such special creatures, the fishery is perpetually under considerable scrutiny.

A whole range of measures govern the operation of the krill fishery, including: by-catch regulation, notification rules, environmental stipulations, observer requirements, and the establishment of catch limits. Additionally, CCAMLR has implemented an ecosystem monitoring program to detect changes in the status of animals that are dependent on krill and to determine whether these changes might be a result of fishing activity.

Because the krill biomass is so large, estimates of the sustainable harvest are also correspondingly large, despite the high degree of precaution. The current annual catch of krill averages about 200,000 tons a year and most of it comes from the South Atlantic in Area 48. The most recent estimate of the biomass of krill in Area 48 is 60.3 million tons (SC-CCAMLR, 2010).

The biomass in the CCAMLR 2000 survey area is thought to be 28% of the global krill biomass (Atkinson et al., 2009), which is conservatively estimated to be around 215 million tons. The seals, seabirds, and whales need about 47.8 million tons of krill for their needs (see Fig. 3). The krill fishery’s highly precautionary trigger level of 620,000 tons per year is only 1% of the biomass. Current estimates of global krill consumption by whales are not available because of uncertainties over the sizes of whale populations, but because they are such mobile predators they are unlikely to be affected by the highly localized and relatively small krill fishery.

So even if the fishery expanded to the current catch limit in the South Atlantic—5.6 million tons a year—it would still be harvesting less than 10% of the stock. There are few other fisheries in the world where the allowable catch is set at such a low proportion of the biomass and where the actual catch is so much lower than that allowed.

**THIRD PARTY SUSTAINABILITY VERIFICATION**

There has been a trend in the last decade to use independent third party certification to confirm the sustainability of seafood products. The Marine Stewardship Council (MSC) is the premier certifying organization, known for its rigorous assessment process and credible standards for sustainable fishing and seafood traceability.

Aker BioMarine’s krill fishery was certified as sustainable and 100% traceable in 2010, allowing products from this fishery to carry the distinctive blue eco-label. In 2012, for a second time, MSC was ranked by Accenture as the leading eco-label for seafood products. Aker’s fishery is currently undergoing the MSC recertification process, which will be completed in 2015.

To achieve MSC certification a fishery must be assessed against several criteria that address issues such as the fishing methodology, the management of the fishery, and the impact of the fishery on the stock and on the species dependent on krill. The certification comes with several responsibilities, including a commitment to research to ensure the sustain-

**FIG. 3.** Estimated predator demand of krill in Area 48.
able management of the resource. Aker’s commitment to MSC certification indicates that it takes the issue of sustainability seriously, a move that has been recognized by environmental groups such as the World Wild Life Fund for Nature (WWF-Norway).

WWF-Norway works with Aker BioMarine to help it meet key MSC certification requirements, including mapping its fishing activities against local predator populations and ensuring that its fishing operations do not cause serious harm to the ecosystem.

Environmental NGOs like WWF play an active role within CCAMLR, focusing on the issue of ecosystem management and on the sustainability of the region’s fisheries. Aker Biomarine works constructively with these NGOs and has involved WWF-Norway in an initiative to bring together krill fishing companies to work together to ensure that CCAMLR’s goals are being met. This initiative—the Association of Responsible Krill (ARK) Fishing Companies—aims to ensure that the industry develops sustainably in support of the long-term viability of the krill stocks and dependent predators.

THE NEED FOR COOPERATIVE RESEARCH
There are very few scientific research vessels operating around Antarctica, and they are only present in the region for short periods each year. In contrast, krill fishing vessels operate on the fishing grounds for most of the year and are extremely well positioned to collect scientific data that can be used in the management of the fishery. Most fishing vessels have the equipment and the capacity to carry out valuable research, which can be used to better understand the biology of krill and provide information on changes in krill stocks.

An initiative of ARK is to conduct annual krill surveys in areas that are not generally accessed by researchers and to encourage the collection of scientific data from fishing vessels. The active involvement of the fishing industry in research will be of benefit to fishers, managers, and scientists. Organizations such as ARK can assist with communication between the fishing industry and CCAMLR, so that management action can be adopted seamlessly.

GOING ABOVE AND BEYOND
Over the course of a decade, Aker BioMarine developed, perfected, and patented a technology called Eco-Harvesting®. This technology, using a specially designed trawl system and direct hose connection between the trawl and the vessel, holds a special mechanism that singles out unwanted by-catch (non-krill species) and releases it unharmed.

In terms of its operation, the equipment stays underwater while a continuous stream of water flows through the hose, bringing the krill live and fresh directly into the factory vessel, which allows for immediate processing of fresh raw material with superior product quality.

Harvesting krill in a commercially viable and environmentally sound way is challenging. Traditional trawling methods where the catch is hauled up on deck and emptied into holding tanks before processing is unsuitable, as krill contains highly digestive enzymes and basically self-destructs before it can be processed.

Furthermore, unwanted by-catch (e.g., invertebrates, fish, and seals) is a problem with regular trawling in the South Atlantic and may pose a threat to fragile marine ecosystems in the Antarctic.

Aker’s Eco-Harvesting fishing system allows the fishing net to stay underwater during the entire operation. Independent observers have verified that the proprietary technology and novel harvesting method ensures no by-catch of other species than Euphausia superba.

WHY ARE ALL THESE EFFORTS NECESSARY?
The Antarctic is a special place, and industries that work there have a responsibility to ensure its conservation. There have been major ecological catastrophes as a result of earlier harvesting activities (i.e., whales, seals, and fish), so it is essential that current and future fishing proceeds in a responsible and sustainable fashion.

The CCAMLR convention provides an excellent framework to ensure the sustainability of the krill fishery, but it is also up to the fishing industry to assist with the task of collecting data and contributing positively to the management process. A krill fishery that is sustainable in the long term is in the best interest of the industry and the ecosystem.

Stephen Nicol is an adjunct professor at the Institute for Marine and Antarctic Studies and an honorary fellow at the Antarctic Climate and Ecosystems Cooperative Research Centre, University of Tasmania. He has extensive experience in krill conservation research, has worked within CCAMLR for more than 25 years, and is a provider of independent scientific advice to the Association of Responsible Krill.

This article was underwritten by AkerBiomarine.

- Ballance, L., et al., The removal of large whales from the Southern Ocean: evidence for long-term

CONTINUED ON NEXT PAGE

### Table 32.
Corn oil: supply, disappearance, and price, United States, 1980/81–2013/14

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\(^b\)Includes inedible distillers corn oil.

\(^c\)Forecast values. Asterisk (*): USDA estimate.
The US Environmental Protection Agency (EPA) aims to complete, this year, the review of 4,800 confidential business information (CBI) claims, potentially containing health and safety studies of existing chemicals.

The agency also expects to review another 350 new CBI cases.

Under a program begun in 2010, the EPA planned – during fiscal years 2011–15 – to review, and challenge where appropriate, more than 22,000 Toxic Substances Control Act (TSCA) CBI claims for chemical identity, "potentially containing health and safety studies."

Last year, 375 health and safety studies, previously treated as CBI, were declassified and made available on the agency website. “The EPA is in the process of doing final reviews, prior to posting, of another 200 filings, which were CBI reviewed and tentatively

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**Dinesh Kumar**

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declassified last year," the agency told Chemical Watch. The majority of those were voluntary industry declassifications, it said. But some were the result of "agency corrections of mischaracterized CBI elements, the CBI claim [having] expired, or was otherwise invalid."

In 2013, the agency also reviewed about 350 new TSCA section 5 and section 8 filings, "believed" to contain health and safety studies. In the vast majority of these cases, the chemicals were found either not to be in commerce or, for new chemical submissions, a generic name was provided when the chemical name was claimed as CBI, the agency said.

The EPA says that industry has been "receptive and responsive" to the agency’s efforts, recognizing that it is limited to health and safety studies on industrial chemicals in commerce, and excludes chemicals such as those in research and development and pre-pesticide filings.

That sentiment is largely reciprocated by industry groups, which say the program has been an example of the success of EPA-industry collaboration.

The impression has been that there is a “significant over-claiming of CBI and I think this decategorization effort goes a long way to debunking that myth,” says Christina Franz, senior director of regulatory and technical affairs at the American Chemistry Council. “When more than 74% of cases reviewed turn out not to even contain CBI claims at all, I think this is significant.”

The only "reservation" that the Society of Chemical Manufacturers and Affiliates has, with an otherwise “very successful” program, is with its treatment of new and specialty chemicals, says Dan Newton, senior government relations manager. “We hope the EPA recognizes that early in a product’s lifecycle, CBI is of critical importance and it prioritizes the CBI that it might challenge," he says. The agency should focus more on chemicals that have been in commerce over a period of time.

The Consumer Specialty Products Association supports an approach that “carefully balances the continued need for CBI protection with the public’s demand for increased disclosure,” says Phil Klein, executive vice president for legislative and public affairs. The agency’s plans to challenge CBI claims more aggressively, as indicated in its budget, “signal that claimants should, going forward, provide upfront justification for information eligible for CBI protection," he adds. The EPA may also require re-substantiation of certain eligibility claims.

Ernie Rosenberg, president of the American Cleaning Institute, said the two TSCA reform measures in Congress also reform CBI provisions. The basic concept of the Chemical Safety Improvement Act and the draft Chemicals in Commerce Act is that “there is some information that is presumptively CBI, other information that requires substantiation and other information that maybe even with substantiation doesn’t qualify as CBI like the content of the health and safety study.”

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Effects of UV-B radiation levels on concentrations of phytosterols, ergothioneine, and polyphenolic compounds in mushroom powders used as dietary supplements


Compositional changes of powder dietary supplements made from mushrooms exposed to different levels of UV-B irradiation were evaluated for the bioactive naturally occurring mushroom antioxidant, ergothioneine; other natural polyphenolic compounds, e.g., flavonoids, lignans, and the like; and selected phytosterols. Four types of mushroom powder consisting of white, brown (Agaricus bisporus), oyster (Pleurotus ostreatus), and shiitake (Lentinula edodes) mushrooms from three different treatment groups (control, low and high UV-B exposures) were evaluated. Ergothioneine concentrations found in mushroom powders were 0.4–10.4 mg/g dry weight (dw) and were not appreciably affected by UV-B radiation. No individual polyphenols were detected above 0.1 μg/g. Phytosterols ergosterol (2.4–6.2 mg/g dw) and campessterol (14–43 μg/g dw) were measured in mushroom powder samples. Ergosterol concentrations decreased significantly with the increased level of UV-B treatment for all mushroom powder types except for white. These results provide some new information on effects of UV-B radiation on these important natural bioactive compounds in mushrooms.

Accumulation of high-value lipids in single-cell microorganisms: a mechanistic approach and future perspectives


In recent years attention has been focused on the utilization of microorganisms as alternatives for industrial and nutritional applications. Considerable research has been devoted to techniques for growth, extraction, and purification of high-value lipids for their use as biofuels and biosurfactants as well as high-value metabolites for nutrition and health. These successes argue that the elucidation of the mechanisms underlying the microbial biosynthesis of such molecules, which are far from being completely understood, now will yield spectacular opportunities for industrial-scale biomolecular production. There are important additional questions to be solved to optimize the processing strategies to take advantage of the assets of microbial lipids. The present review describes the current state of knowledge regarding lipid biosynthesis, accumulation, and transport mechanisms present in single-cell organisms, specifically yeasts, microalgae, bacteria, and archaea. Similarities and differences in biochemical pathways and strategies of different microorganisms provide a diverse toolset to the expansion of biotechnologies for lipid production. This paper is intended to inspire a generation of lipid scientists to insights that will drive the biotechnologies of microbial production as uniquely enabling players of lipid biotherapeutics, biofuels, biomaterials, and other opportunity areas into the 21st century.

Micronutrient content of cold-pressed, hot-pressed, solvent-extracted and RBD canola oil: implications for nutrition and quality


In this study the quality characteristics and content of healthy minor components of four crude canola oils as an effect of different oil extraction method (solvent extraction, hot pressing, and cold pressing) were studied. Cold-pressed canola oils had lower concentrations of free fatty acids, peroxide value, p-anisidine value, and chlorophylls than solvent-extracted and hot-pressed canola oils. Oils obtained via the different extraction methods had different fatty acid profiles as well as dissimilar amounts of tocopherols, phytosterols, and polyphenols. The amount of total tocopherols in solvent-extracted canola oil was 493 mg/kg compared to 388 mg/kg for hot-pressed canola oil. The tocopherol content for two other cold-pressed and one other refined-bleached-deodorized (RBD) canola oil was 366, 354, and 327 mg/kg, respectively. Solvent-extracted canola oil exhibited the highest free phytosterol content (178 mg/100 g), while RBD canola oil only had 129 mg/100 g of free phytosterols. While cold-pressed canola oil had the lowest amount of polyphenols, traditional refining resulted in almost complete removal of polyphenols from canola oil.

Total phospholipids in edible oils by in-vial solvent extraction coupled with FTIR analysis

A simple procedure for the determination of total phospholipids (TPL) in edible oils was developed by combining a single-step, in situ methanol/acetone (MeOH/ACN) extraction of the oil sample followed by Fourier transform infrared (FTIR) spectroscopic analysis of the extract. Spectral analysis of extracts in a 25 μm CaF<sub>2</sub> cell obtained using 1:1 MeOH/ACN added to oil in a 2:1 ratio indicated that measurements made using only the asymmetric phosphate diester PO<sub>2</sub>-stretching band at 1243 cm<sup>-1</sup> in second-derivative spectra were sufficient for the accurate measurement of TPL with minimal coextracted triglyceride interferences being encountered. FTIR calibration spectra were devised using only phosphatidylcholine (PC) as a representative phospholipid standard, covering a range of 0–50,000 μg/g TPL and spiked into 1:1 MeOH/ACN, capable of tracking the added PC with a standard deviation of <200 μg/g. The FTIR method was initially validated using model PC-spiked degummed canola oil and subsequently with commercial crude and refined soy and rapeseed oils as well as a lecithin tablet with the FTIR TPL predictions compared to those of the AOCS Ca 12-55 molybdate method. The FTIR method tracked the AOCS results well, being somewhat more reproducible than the reference method (±3.2 vs. ±4.9%), which limited its accuracy relative to the AOCS reference procedure (±2.2%). The in-vial solvent extraction procedure, followed by FTIR analysis of the extract, is a simple, efficient, and rapid procedure that is also amenable to automation using an autosampler-equipped FTIR if multiple samples are to be analyzed.

Ionic liquid-based extraction of fatty acids from blue-green algal cells enhanced by direct transesterification and determination using GC × GC-TOFMS


Blue-green algae, commonly referred to as cyanobacteria, are known to grow in freshwater bodies when they are provided with suitable growth conditions such as nutrients, temperature, and light. Algae biomass is known to contain a large amount of lipids, such as saturated and unsaturated fatty acids. In this study, fatty acids from algal cells were extracted using a newly developed extraction protocol using ionic liquid enhanced by direct transesterification at an elevated temperature. The identification and quantification of fatty acids was performed using gas chromatography coupled to a time-of-flight mass spectrometer (GC × GC-TOFMS). The extracted fatty acids were dominated by those with carbon chain of C16 and C18 [i.e., 7-hexadecenoic acid (C16:1) and hexadecanoic acid (C16:0) for C16, whereas C18 includes γ-linolenic acid (γ-C18:3); linoleic acid (C18:2); linolenic acid (C18:3); 6,9,12,15-octadecatetraenoic acid (C18:4); oleic acid (C18:1); and octadecanoic acid (C18:0)]. The obtained fatty acid composition was then compared with that obtained by organic solvent extraction using a mixture of chloroform and methanol. Statistical evaluation was performed using one-way ANOVA and found that there was no statistically significant difference (P = 0.908) between the two extraction methods, a finding that indicates the usefulness of ionic liquid as a solvent to replace volatile organic solvent to minimize environmental pollution.

Improving method reliability in carotenoid analysis through selective removal of glycerolipid interferences by lipase treatment


Saponification is most often used to hydrolyze glycerolipid interferences during carotenoid analysis. Ester bonds of other plant metabolites such as carotenoids are, however, also hydrolyzed during saponification, thus altering the natural carotenoid composition. A straightforward and selective cleanup procedure was therefore developed involving the enzymatic hydrolysis of matrix glycerolipids. The optimized procedure [100 μL of extracted vegetable or algal oil in 20 mL of 50:50 phosphate buffer/methanol with 25 μL of sodium n-octyl sulfate, 30 mg of bile salts, and 250 μL of NaCl solution (5 mM), magnetic stirring for 2 h at 40°C with 1 mL of Lipozyme TL 100 L and 1 mL of Lipozyme CALB L] removed the greater part of triglycerides (94.8–100.0%) and diglycerides (88.2–99.8%) while preserving the natural carotenoid composition.

Factors influencing the contents of coenzyme Q10 and Q9 in olive oils


The health effects of olive oil are attributed to its high content of oleic acid and other constituents, particularly its phenolic fraction. Olive oil also contains other substances with potential health effects such as coenzyme Q10 (CoQ10) and coenzyme Q9 (CoQ9). The objective of our study was to investigate some factors that could influence the quantity of coenzyme Q<sub>10</sub> (CoQ) in olive oils. We analyzed almost 100 samples of commercial oil blends and fresh extra virgin olive oils of various cultivars using high-performance liquid chromatography. With the investigation of various monocultivar samples we determined that genetic parameters (cultivars) have an important influence on the composition of olive oils, particularly the content of CoQ10. Possible effects of the degree of ripeness were also studied for the cultivars Istrska belica and Leccino. We determined that the highest levels of both CoQ10 and CoQ9 can be found in early maturation stages.

CONTINUED ON NEXT PAGE
Antimicrobial activities of novel mannosyl lipids isolated from the biocontrol fungus *Simplicillium lamellicola* BCP against phytopathogenic bacteria


The antagonistic fungus *Simplicillium lamellicola* BCP has been developed as a microbial biopesticide that effectively controls the development of various plant diseases caused by both pathogenic bacteria and pathogenic fungi. Antibacterial bioassay-directed fractionation was used to isolate mannosyl lipids from *S. lamellicola* BCP, and the structures of these compounds were elucidated using spectral analysis and chemical degradation. Three novel mannosyl lipids were characterized and identified using spectral analysis and chemical degradation. They were elucidated using spectral analysis and chemical degradation.

Impact of environmental stresses on orange oil-in-water emulsions stabilized by sucrose monopalmitate and lysolecithin


The food and beverage industry is trying to replace synthetic functional ingredients with more “label-friendly” ingredients in many commercial products. This study therefore examined the influence of environmental stresses on the stability of emulsions stabilized by a combination of lysolecithin and sucrose monopalmitate (SMP). Orange oil-in-water emulsions [5% (w/w) oil] stabilized by SMP (1%) and lysolecithin (0.1–0.5%) were prepared using high-pressure homogenization (pH 3). In the absence of lysolecithin, all emulsions were highly unstable to droplet aggregation, which was attributed to low droplet charge (weak electrostatic repulsion) and small SMP headgroup size (weak steric repulsion). Incorporation of 0.1–0.5% lysolecithin into the emulsions greatly improved their stability to droplet aggregation. Incorporation of 0.1–0.5% lysolecithin into the emulsions greatly improved their stability to droplet aggregation, which was attributed to the increase in negative charge on the droplets (strong electrostatic repulsion). The addition of high levels of salt (NaCl) to the emulsions promoted droplet aggregation and creaming. Emulsions containing 0.5% lysolecithin were stable to heating (30–90°C) in the absence of salt but exhibited droplet aggregation and creaming when held at high (>50°C) temperatures in the presence of 300 mM salt. This study has implications for the development of emulsion-based delivery systems for use in food and beverage products.

Phospholipid architecture of the bovine milk fat globule membrane using giant unilamellar vesicles as a model


Giant unilamellar vesicles (GUVs) were constructed using an electroformation technique to mimic the morphology of the native milk fat globule membrane (MFGM) for the purpose of structural investigation. Bovine milk-derived phospholipids were selected to manufacture GUVs that were characterized by confocal laser scanning microscopy after fluorescent staining. Circular nonfluorescent dark regions were observed in a 3:7 (mol/mol) surface mixture of 1,2-dipalmitoyl-sn-glycero-3-phosphocholine (DPPC) and 1,2-dioleoyl-sn-glycero-3-phosphoethanolamine. Linear-shaped dark lipid domains were found in GUVs containing sphingomyelin (SM) in the absence of cholesterol. The dark regions were interpreted as a gel phase formed by a high gel–liquid phase transition temperature (T_{gel}) of DPPC and SM. This study provides a strategy for investigating the lipid structural organization within the native MFGM using a model lipid bilayer system.

Oxidized frying oil and its polar fraction fed to pregnant mice are teratogenic and alter mRNA expressions of vitamin A metabolism genes in the liver of dams and their fetuses


We previously observed a higher incidence of congenital malformations in the fetuses of dams fed an oxidized frying oil (OFO)-containing diet during pregnancy. In this study, we hypothesized that, during pregnancy, maternal ingestion of OFO, specifically the oxidized components (i.e., the polar fraction),
Characterization and quantification of diacylglycerol species in biological extracts after one-step derivatization: a shotgun lipidomics approach


Diacylglycerols (DAG) are important intermediates of lipid metabolism and cellular signaling. It is well-known that the mass levels of DAG are altered under disease states. Therefore, quantitative analysis of DAG in biological samples can provide critical information to uncover underlying mechanisms of various cellular functional disorders. Although great efforts on the analysis of individual DAG species have recently been made by utilizing mass spectrometry with or without derivatization, cost-effective and high throughput methodologies for identification and quantification of all DAG species including regioisomers, particularly in an approach of shotgun lipidomics, are still missing. Herein, we describe a novel method for directly identifying and quantifying DAG species in biological extracts after facile one-step derivatization with dimethylglycine based on the principles of multidimensional mass spectrometry-based shotgun lipidomics. The established method provided substantial sensitivity (low limit of quantification at amol/μL), high specificity, and broad linear dynamics range (2,500-fold) without matrix effects. By exploiting this novel method, we revealed a 16-fold increase of total DAG mass in the livers of ob/ob mice compared to their wild type controls at 4 months of age (an insulin-resistant state) vs. a fivefold difference between 3 month old mice (with normal insulin). These results demonstrated the importance and power of the method for studying biochemical mechanisms underpinning disease states.

Chemical and microbiological considerations of phytosterols and their relative efficacies in functional foods for the lowering of serum cholesterol levels in humans: a review


The controversy of the relative efficacies of sterols and stanols in the lowering of blood cholesterol in humans continues without resolution. Basic physical, chemical, and microbiological characteristics of phytosterols were reviewed in the context of the animal physiology and study design. Not all sterols are alike chemically. Shape and size of the test molecule do matter. Involuntary microbial transformation of sterols into stanols in the human gut would inevitably afford different outcome, even if clonal humans were available as test subjects. The current discourse on the relative merits of different forms of phytosterols for lowering serum cholesterol levels in humans might be a futile exercise if the different physical, chemical, and microbiological reactivities of the molecule being studied were ignored. Theoretical considerations and clinical-study evidence suggest that stanol would be the principal bio-reactive species to cause the lowering of serum cholesterol in humans.

Liposomes: versatile and biocompatible nanovesicles for efficient biomolecules delivery


Since the revolutionary discovery that phospholipids can form closed bilayered structures in aqueous systems, liposomes have become a very interesting topic of research. Because of their versatility and amazing biocompatibility, the use of liposomes has been widely accepted in many scientific disciplines. Their applications, especially in medicine, have yielded breakthroughs with anticancer-drug carriers over the past few decades. Specifically, their easy preparation and various structural aspects have given rise to a broadly usable way to internalize biomolecules such as drugs, DNA, RNA, and even imaging probes. This review article reports recent developments in liposomal drug delivery and gene delivery, and thoroughly covers the synthesis and different kinds of liposomal surface modification techniques that have resulted
in higher stability and efficiency with respect to the use of liposomes in tumor cell targeting, site-specific release, and extending blood retention times.

The impact of cholesterol, DHA, and sphingolipids on Alzheimer’s disease


Alzheimer’s disease (AD) is a devastating neurodegenerative disorder currently affecting over 35 million people worldwide. Pathological hallmarks of AD are massive amyloidosis, extracellular senile plaques, and intracellular neurofibrillary tangles accompanied by an excessive loss of synapses. Major constituents of senile plaques are 40–42 amino acid long peptides termed β-amyloid (Aβ). Aβ is produced by sequential proteolytic processing of the amyloid precursor protein (APP). APP processing and Aβ production have been one of the central scopes in AD research in the past. In the last years, lipids and lipid-related issues are more frequently discussed to contribute to the AD pathogenesis. This review summarizes lipid alterations found in AD postmortem brains, AD transgenic mouse models, and the current understanding of how lipids influence the molecular mechanisms leading to AD and Aβ generation, focusing especially on cholesterol, docosahexaenoic acid (DHA), and sphingolipids/glycosphingolipids.

Silver ion solid-phase extraction chromatography for the analysis of trans fatty acids


This article describes the application of silver ion solid-phase extraction (Ag+-SPE) to the separation of the fatty acid methyl esters (FAME) prepared from a partially hydrogenated fish oil (PHFO) with a trans fatty acid (FA) content of 41%. The complex FAME mixture was resolved into the following FAME fractions: saturated FA, trans-monoensaturated FA (trans-MUFA), cis-MUFA, and several polyunsaturated FA (PUFA) fractions with mixed geometric and positional isomers. Fractions were analyzed by gas chromatography using a 100 m cyanopropyl polysiloxane coated capillary column. The gas chromatographic analysis of the Ag+-SPE fractions allowed for the identification of the trans- and cis-MUFA and other FAME prepared from PHFO.

Plastids with or without galactoglycerolipids


In structural, functional, and evolutionary terms, galactoglycerolipids are signature lipids of chloroplasts. Their presence in nongreen plastids has been demonstrated in angiosperms and diatoms. Thus, galactoglycerolipids are considered as a landmark of green and nongreen plastids, deriving from either a primary or secondary endosymbiosis. The discovery of a plastid in Plasmodium falciparum, the causative agent of malaria, fueled the search for galactoglycerolipids as possible targets for treatments. However, recent data have provided evidence that the Plasmodium plastid does not contain any galactoglycerolipids. In this opinion article, we discuss questions raised by the loss of galactoglycerolipids during evolution: How have galactoglycerolipids been lost? How does the Plasmodium plastid maintain four membranes without these lipids? What are the main constituents instead of galactoglycerolipids?

HDL hypothesis: Where do we stand now?


There is robust epidemiological evidence dating back to the original Framingham Heart Study from 1977 that indicates an important inverse relationship between high-density lipoprotein cholesterol (HDL-C) and risk of incident coronary artery disease (CAD). Despite this body of scientific information demonstrating that low levels of HDL-C are an independent predictor of subsequent CAD events, multiple therapeutic attempts to raise HDL-C levels have failed to demonstrate a consistent reduction in prognostically important endpoints such as death, myocardial infarction (MI), and stroke. Recently, several major randomized trials using different therapeutic interventions have raised appropriate concerns about our basic understanding of HDL-C and whether the “HDL hypothesis” of lowering cardiovascular events through therapeutic interventions directed at raising HDL-C is a scientifically viable one. While two recent randomized controlled trials (AIM-HIGH and HPS2-THRIVE)
failed to show a reduction in cardiovascular events in patients treated to optimally low levels of low-density lipoprotein cholesterol (LDL-C) at baseline with extended-release niacin on a background of simvastatin, these clinical trials studied specific populations of stable ischemic heart disease patients. The data from these two contemporary trials cannot be extrapolated to all patient populations, such as those with acute coronary syndromes or myocardial infarction or those with significant residual mixed dyslipidemia not treated with optimal doses of intensive statin therapy, as these patients were excluded by trial design in both studies. Therefore, at the present time, there is insufficient evidence from clinical trials to recommend HDL-targeted therapy for additional event reduction in CAD patients. However, we will review the relevant data from recent major trials (AIM-HIGH, HPS2-THRIVE, ILLUMINATE, and dal-OUTCOMES) and highlight the potential clinical implications of these trials in modern pharmacotherapy as it relates to HDL-C raising and potential cardiovascular event reduction.