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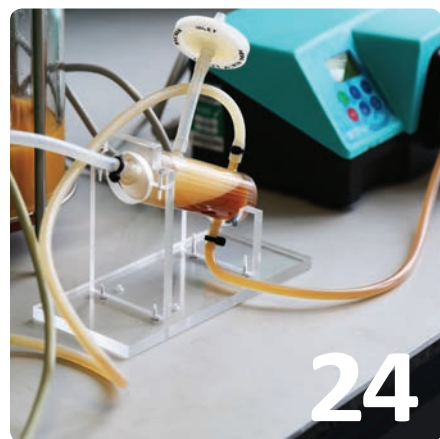
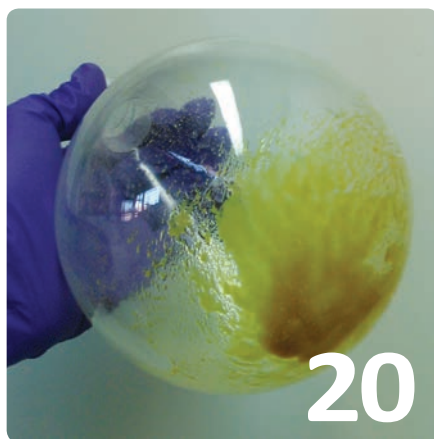
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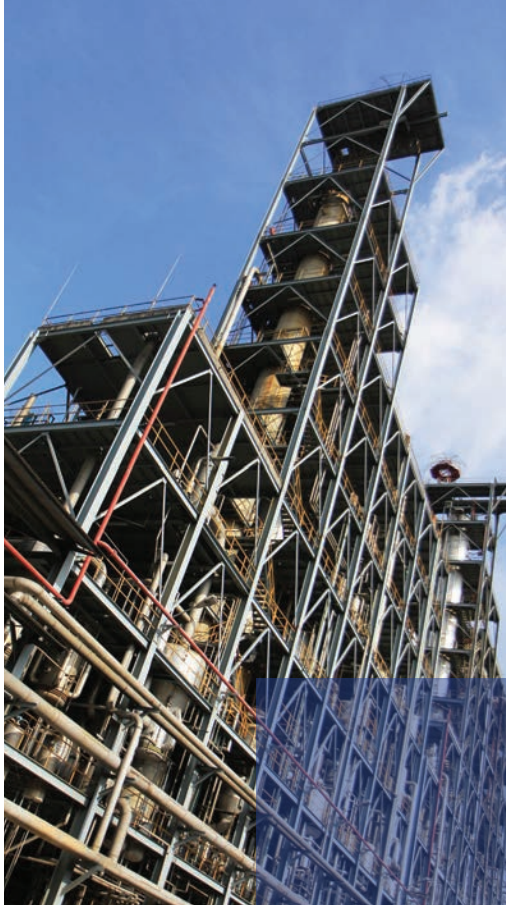
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Catherine Watkins

# The whys and wherefores of life-cycle assessment

Life-cycle assessment, or LCA, is a tool for quantifying the overall environmental impact of products and processes, from the extraction of raw materials through to product disposal, or from “cradle to grave.” Industry pioneered development of this analytical methodology beginning in the 1960s; the International Organization for Standardization (ISO) has since codified the methodology into a suite of standards (see page 11).

- Life-cycle assessment (LCA) is a tool used by organizations of all sizes to analyze the overall environmental impact of their products, processes, and services.
- LCA can advance product and process innovation as well as enhance competitive differentiation among brands. It can also be used to identify cost-savings, improve design decisions, and achieve regulatory mandates.
- If your organization isn't currently performing LCAs, you can be sure your competitors are doing them on your products.

“LCAs can be an important tool in building a company's business by identifying cost-saving opportunities, such as on energy and water, or by building gross sales or revenue by helping identify new environmentally sustainable products,” says Len Sauers. Sauers, who retired in 2016 as vice president of global sustainability for P&G in Cincinnati, Ohio, USA, currently consults on corporate sustainability strategy through LJS Consulting LLC (also in Cincinnati).

## INDUSTRY TAKES THE LEAD

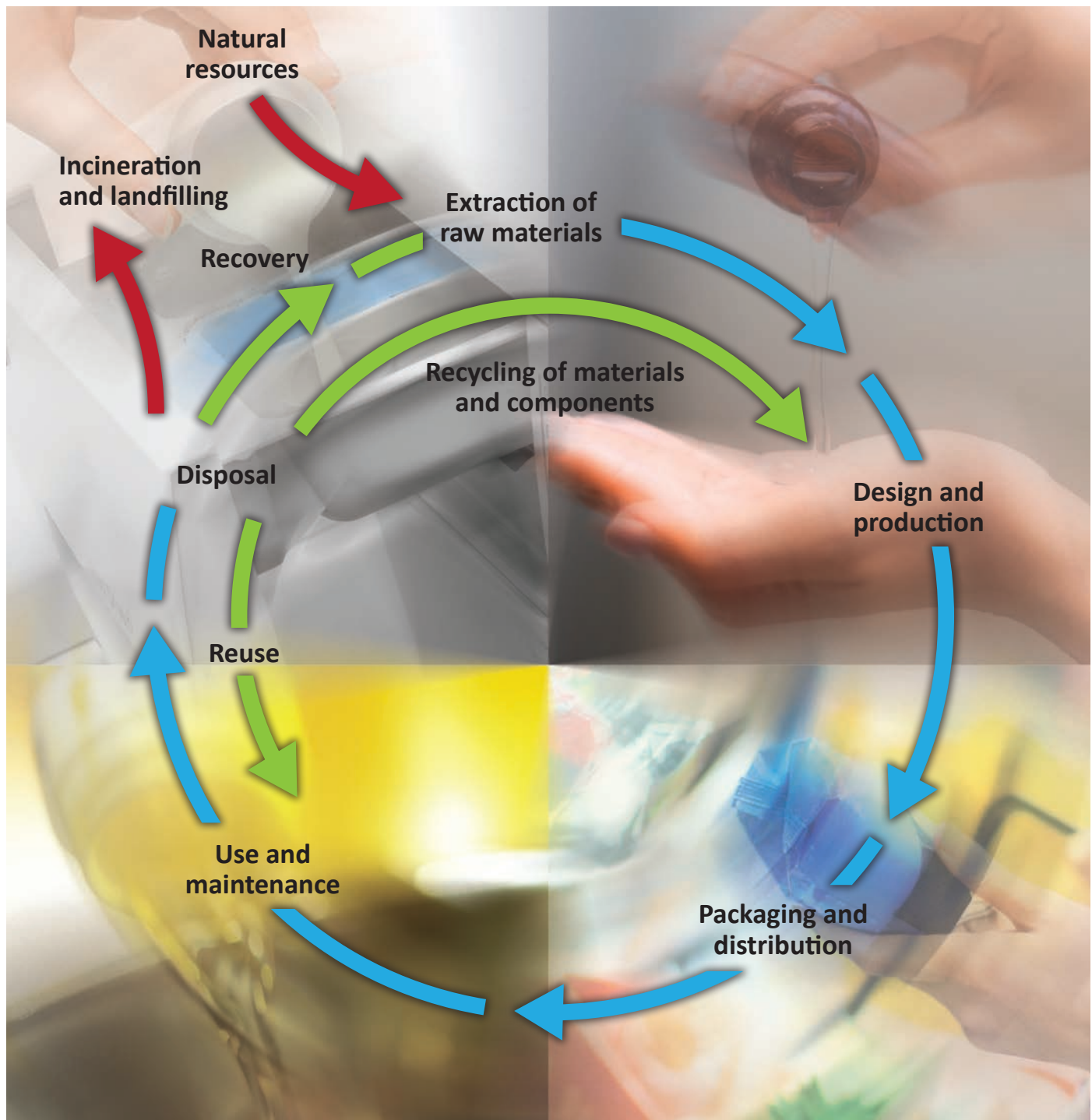
Former AOCs President Erich Dumelin, who retired from Unilever in 2007 as vice president, supply chain strategy and technology for the company's food business, remembers the early days of LCA development.

In the 1980s, he says, Unilever Switzerland began examining the environmental impact of product packaging using Ecopro software from EMPA (the Swiss Federal Laboratories for Materials Science and Technology in St. Gallen).

“As far as I know, this was the first institution to develop a program to assess the impact of industrial activities on the environment, starting with packaging material,” Dumelin notes. “Later, during my time at the Unilever Center in the 1990s, the first talks about CSR (corporate social responsibility) began on a much broader level, and we became more aware and concerned about the global impact of our products and services.”

One of Unilever's initial ideas was to choose more sustainable product packaging materials, product formulations, and technologies, according to Dumelin. “The ability to choose alternative materials and processes to produce ‘greener’ products was often constrained by other factors. These included the nutritive value, product functionality, sensory qualities, and cost. Therefore, our emphasis shifted from replacement with ‘cleaner’ alternatives to improving the environmental footprint of the raw materials and processes. For the assessment of





Source: United Nations Environment Program Division of Technology, Industry and Economics.

the environmental aspects of our sustainability efforts, the LCA tool came in handy and today is probably the method of choice,” he concludes.

Dumelin notes that following the complete edible product supply chain from cradle to fork, including disposal, revealed that the environmental impact of the raw materials was usually much greater than the sum of what followed further down the chain. It also became clear that consumer behavior and preference could have a significant effect on the upstream environmental impact of a product.

“Evaluation of products or processes for environmental impact was done for decades before it was called life-cycle assessment,” explains Len Sauers, adding that P&G was also an early pioneer in LCA development.

LCAs sometimes return surprising results that run counter to a company’s assumptions, he says. A classic example is the comprehensive LCA P&G conducted in 2008, which looked at energy use and greenhouse gas (GHG) emissions for all the company’s product categories. Before conducting the LCA, P&G researchers intuitively assumed the greatest envi-

ronmental impact of its detergent products would relate to manufacturing.

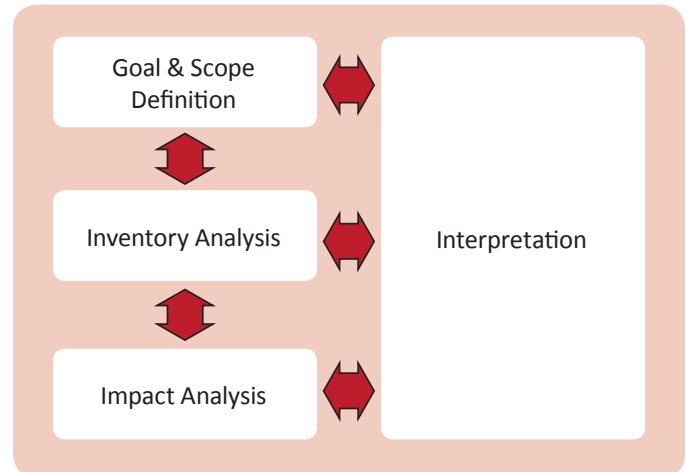
“We found that the greatest use of energy and largest emissions of GHG came from the consumer-use phase of laundry detergents,” Sauers noted in an email. “More specifically, it came from the heating of water to do laundry. This information formed the basis for a research program into the development of new technologies that improve the performance of laundry detergents in cold water. A new product was introduced—Tide Cold Water. The technologies were also used to improve the performance of our premium brands. We also set a corporate environmental sustainability goal of having 70% of machine loads washed in cold water by 2020.”

## THE BASICS OF LCA

The methodology for conducting LCAs is contained in a set of standards from the International Organization for Standardization (ISO; see page 11). These include full assessments (ISO 14044–14073) as well as streamlined analyses (ISO 14064–14067 and ISO 14069).

In brief, full LCAs comprise four repeating steps: 1) defining the goal and scope of the study, 2) completing a Life-Cycle Inventory (LCI), 3) performing a Life-Cycle Impact Assessment (LCIA), and 4) interpreting the results. As each step is completed, the results of that step are validated against the pre-

**FIG. 1. Methodological framework for life-cycle assessment (ISO 14040 and 14044). This figure visualizes the iterative flow of information in the LCA process.** Source: American Center for Life Cycle Assessment (Washington, DC, USA; [www.aclca.org](http://www.aclca.org))



ceding steps for accuracy and adherence to the original objectives of the study (see Fig. 1).

The *goal and scope of the assessment* must be stated explicitly, including defining the functional unit, the system boundaries, the assumptions and the limitations of the study, the impact categories, and the methods used to allocate environmental burdens.

## LCA of high-oleic soybean oil used for restaurant frying

DuPont Pioneer used attributional life-cycle methodology to quantify the environmental impacts of and identify supply chain hotspots for high-oleic soybean oil (HOSO), conventional soybean oil (CSO), hydrogenated soybean oil (HSO), high-oleic canola oil (HOCO), and conventional canola oil (CCO). (Attributional LCA describes the environmentally relevant physical flows to and from a product or process, whereas consequential LCA analyzes how relevant environmental flows will change in response to possible decisions.)

Two scenarios were used in the study. The first assumed two days of fryer use with a 4% loss rate of oil to the food per day and the second assumed six days of fryer use and a 10% loss rate of oil to the food per day. The system boundaries included agricultural inputs, farming operations, seed processing, soybean and canola oil refining (and hydrogenation, when applicable), transport along the supply chain and of the refined oil to a warehouse near the restaurant, oil use in the restaurant, washing of the fryers, and spent oil disposal.

Results of the study indicated that across all impact categories, the increased oil stability of the high-oleic



**Oils heated at 150°C for 20 days.**  
Photo courtesy of DuPont Pioneer.

oils and HSO resulted in roughly a 30–45% reduction in impact relative to conventional oils. The impact categories used for this study were climate change potential, eutrophication potential, acidification potential, land use, and nonrenewable energy demand.

“The large majority of food companies understand high-oleic oils and the value they bring in terms of oxidative stability and function,” noted Susan Knowlton, senior research manager at DuPont Pioneer. “They don’t, however, understand the environmental impact. This LCA helped us to educate people downstream about how oils from Plenish high-oleic soybeans

can be used to lower the environmental impact of frying oils in the restaurant setting.”

“Environmental Life-Cycle Impacts of High Oleic Soybean Oil Used for Frying,” Todd M. Krieger and Susan Knowlton, *Proceedings of the 9th International Conference on Life Cycle Assessment in the Agri-Food Sector*, October 2014. This article is available for download at [lcafood2014.org/papers/236.pdf](http://lcafood2014.org/papers/236.pdf).



The *LCI phase* examines all the inputs and outputs in the product's life cycle, beginning with raw material extraction and extending through manufacture to the consumer-use phase and, finally, through to disposal (or recycling) of the product and/or packaging.

Next, the *life-cycle impact assessment* translates the LCI results into environmental impacts. These can be assessed at either the midpoint—for example, water use, eutrophication or land use—or endpoint levels such as the impact on human health, resource depletion or ecosystem quality.

The *final interpretation phase* identifies, quantifies, and evaluates information from the results of the LCI and LCIA. It should result in a set of conclusions and recommendations, and in order to comply with ISO 14040 (2006) and ISO 14044 (2006) standards, the LCA must be critically reviewed by a panel, with the results made available to the public.

"In theory, concepts of LCA can be applied to any value chain that involves any kind of material, energy, packaging, processes, products, technologies, processes, or services," explains Bill Flanagan, director of the Ecoassessment Center of Excellence at GE in Niskayuna, New York, USA. Flanagan also is chair of the board of directors of the American Center for Life Cycle Assessment, a non-for-profit membership organization based in Washington, DC, USA.

Comparing different products or technologies on a functionally equivalent basis can provide strategic insights and potentially even lead to changes in a company's business model, he says. For example, a company that sells music on compact disc determines through an LCA that it is more advantageous to offer music via download or online streaming.

## CAVEATS AND CONUNDRUMS

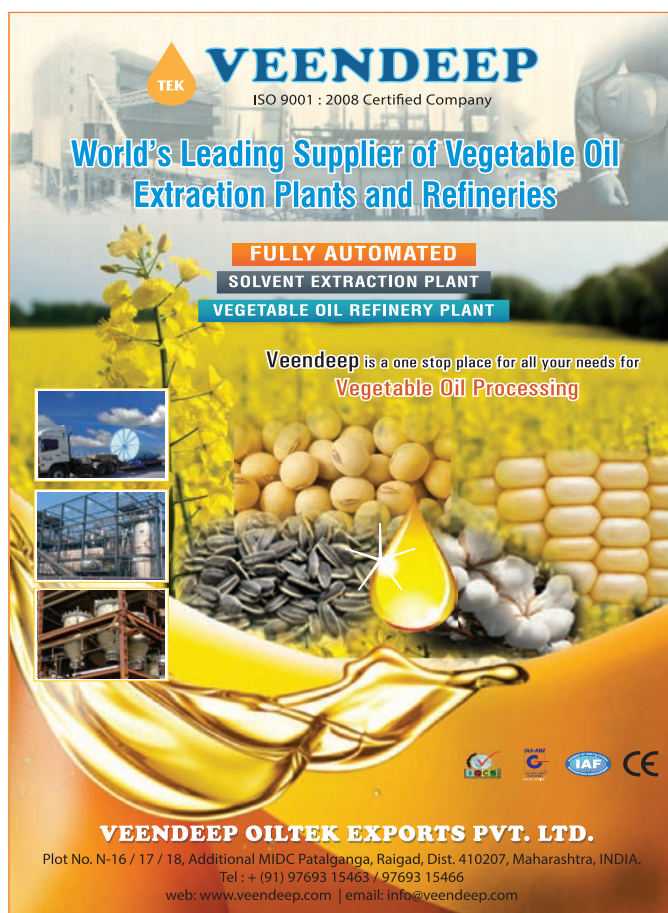
So, just how robust is the LCA methodology?

"Data are crucial to LCAs, and LCAs are not hard science," notes one source, who asked to remain anonymous. "The input and output data need to be selected as consistently and stringently as possible, and that may often be difficult. Nevertheless, LCAs can provide guidelines for policy makers and other interested parties as long as their potential shortcomings are recognized."

Thomas P. Gloria, director of sustainability at the Harvard Extension School (Cambridge, Massachusetts, USA) and managing director of Industrial Ecology Consultants (Newton, USA) agrees with this view.

"Datasets can be small in sample size and may not be truly representative," he says. "If it is a critical item in your analysis, you need to check, recheck, and understand the uncertainties and variability associated with datasets, which generally are not well characterized. LCA is a wonderful tool, but there are other methodologies that might be more appropriate at a regional or site-specific level."

Gloria also counsels that interpretation of LCA results is subjective and, therefore, can lead to difficulties in decision-making. If the results show an increase in environmental impact in one category and a decrease in another, "a value judgment needs to be made." He emphasizes that "the LCA



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methodology provides the awareness and transparency to show where the science ends and a value judgment is made.”

Despite the caveats, LCA “is the way you should think in considering the whole of what you are doing,” says Gloria. “The problems in the world are becoming more and more

complex, which makes holistic, multidimensional tools such as LCA not just preferred, but essential.”

*Catherine Watkins is a freelance writer based in Champaign, Illinois, USA. She may be reached at [c.e.watkins@sbcglobal.net](mailto:c.e.watkins@sbcglobal.net).*

## Resources

### Organizations

The **American Center for Life Cycle Assessment** (ACLCA; Washington, DC, USA; [www.aclca.org](http://www.aclca.org)) is a nonprofit membership organization providing education, awareness, advocacy, and communications to build capacity and knowledge of environmental LCA. ACLCA membership is drawn from industry, academia, government, consulting, and nongovernmental organizations. ACLCA includes both domestic and international members.

The **Life Cycle Initiative** ([www.lifecycleinitiative.org](http://www.lifecycleinitiative.org)) is a joint activity of the United Nations Environment Programs and the Society of Environmental Toxicology and Chemistry (SETAC; [www.setac.org](http://www.setac.org)). The objective of the Life Cycle Initiative is to “facilitate the generation and uptake of science-based life cycle approaches and information for products by business, government, and civil society practice worldwide as a basis for sustainable consumption and production.” As such, the Initiative advocates Life-Cycle Sustainability Assessments (LCSA), which extend LCAs in order to evaluate all environmental, social, and economic impacts of product life cycles. A number of reports on LCSAs are available at [www.lifecycleinitiative.org/resources/reports](http://www.lifecycleinitiative.org/resources/reports).

### Environmental Product Declarations

Customers and consumers alike increasingly demand information about product sustainability before they make buying decisions.

One advantage of conducting an ISO-compliant LCA is that one can then apply for an independently verified and registered document known as an Environmental Product Declaration (EPD; ISO 14025). EPDs are much like nutrition labels; they simply disclose the environmental impacts of a product rather than certifying that the product itself is “green.” Visit [tinyurl.com/EPD-Sample](http://tinyurl.com/EPD-Sample) to view a sample EPD document for Castillo de Canena extra virgin olive oil. Search among all EPDs registered within the International EPD® System, or browse the database by product category at [www.environdec.com/EPD-Search](http://www.environdec.com/EPD-Search).

### LCI databases

The most comprehensive commercially available databases for life-cycle inventories, according to Bill Flanagan of the

ACLCA, are ecoinvent and GaBi. The former is available by license at [www.ecoinvent.org](http://www.ecoinvent.org) from a not-for-profit association founded by the institutes of the ETH Domain and the Swiss Federal Offices. The latter is available from thinkstep (Echterdingen, Germany) at [www.gabi-software.com](http://www.gabi-software.com).

An interactive map of databases is available at <http://tinyurl.com/LCA-DB-Map>.

### Published LCAs

“Comparison of oleo- vs. petro-sourcing of fatty alcohols via cradle-to-gate life-cycle assessment,” *Journal of Surfactant and Detergents* (2016), <http://dx.doi.org/10.1007/s11743-016-1867-y>.

This LCA found petro-based fatty alcohols had overall lower average greenhouse gas emissions compared to palm kernel oil (PKO)-based fatty alcohols. “We found the judicious decisions on land use change, effluent treatment and solid waste treatment are key to making PKO-FA environmentally sustainable,” according to the article’s abstract.

“Life-cycle assessment of five vegetable oils,” *Journal of Cleaner Production* (2015), <http://dx.doi.org/10.1016/j.jclepro.2014.10.011>.

Researchers conducted a consequential LCA of palm, peanut, rapeseed, soybean, and sunflower oils, focusing on global warming, land use, and water consumption. “With respect to global warming, rapeseed oil and sunflower oil are the best performing, followed by soybean oil and palm oil, and with peanut oil as the least good performing. For land use, palm oil and soybean oil are the oils associated with the smallest contribution, followed by rapeseed oil, and with sunflower oil and peanut oil as the oils with the largest net occupation of land. When focusing on water consumption (using the water stress index), sunflower oil had the smallest impact, followed by rapeseed oil, palm oil and soybean oil, and with peanut oil as the oil with the largest contribution.”

“Potential of biofuels from algae: comparison with fossil fuels, ethanol and biodiesel in Europe and Brazil through life-cycle assessment,” *Renewable and Sustainable Energy Reviews* (2017), <http://dx.doi.org/10.1016/j.rser.2017.01.152>.

## ISO LCA standards

ISO—the International Organization for Standardization—has developed a suite of nine standards relating to life-cycle assessment, which are available for purchase at <https://www.iso.org/store>. These standards cover the assessment of impacts on the environment from the extraction of raw materials (cradle) to the final disposal of waste (grave). Note that two additional LCA standards are under development by ISO Technical Committee 207/Subcommittee 5 (ISO 14044:2006/DAMd 1 and ISO PRF TR 14073).

In addition, standards for streamlined LCAs looking only at quantification and validation/verification of greenhouse gas emissions, including calculating the carbon footprint of products, are available (ISO 14064–14067 and ISO 14069).

Following are the standards pertaining to full LCAs.

### ISO 14040:2006 (Ed. 2)

Environmental management—Life cycle assessment—Principles and framework

### ISO 14044:2006 (Ed. 1)

Environmental management—Life cycle assessment—Requirements and guidelines

### ISO 14045:2012 (Ed. 1)

Environmental management—Eco-efficiency assessment of product systems—Principles, requirements and guidelines

### ISO 14046:2014 (Ed. 1)

Environmental management—Water footprint—Principles, requirements and guidelines

### ISO/TR 14047:2012 (Ed. 2)

Environmental management—Life cycle assessment—Illustrative examples on how to apply ISO 14044 to impact assessment situations

### ISO/TS 14048:2002 (Ed. 1)

Environmental management—Life cycle assessment—Data documentation format

### ISO/TR 14049:2012 (Ed. 2)

Environmental management—Life cycle assessment—Illustrative examples on how to apply ISO 14044 to goal and scope definition and inventory analysis

### ISO/TS 14071:2014 (Ed. 1)

Environmental management—Life cycle assessment—Critical review processes and reviewer competencies: Additional requirements and guidelines to ISO 14044:2006

### ISO/TS 14072:2014 (Ed. 1)

Environmental management—Life cycle assessment—Requirements and guidelines for organizational life-cycle assessment

### ISO/DTR 14073

Environmental management—Water footprint—Illustrative examples on how to apply ISO 14046



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# The effect of **monoglyceride incorporation** on the **solvent absorption** and mechanical properties of **glycerol-based** polymer films

Prince G. Boakye, Kerby C. Jones, Nicolas Latona, Cheng-Kung Liu, Samuel A. Besong, Stephen E. Lumor, and Victor T. Wyatt

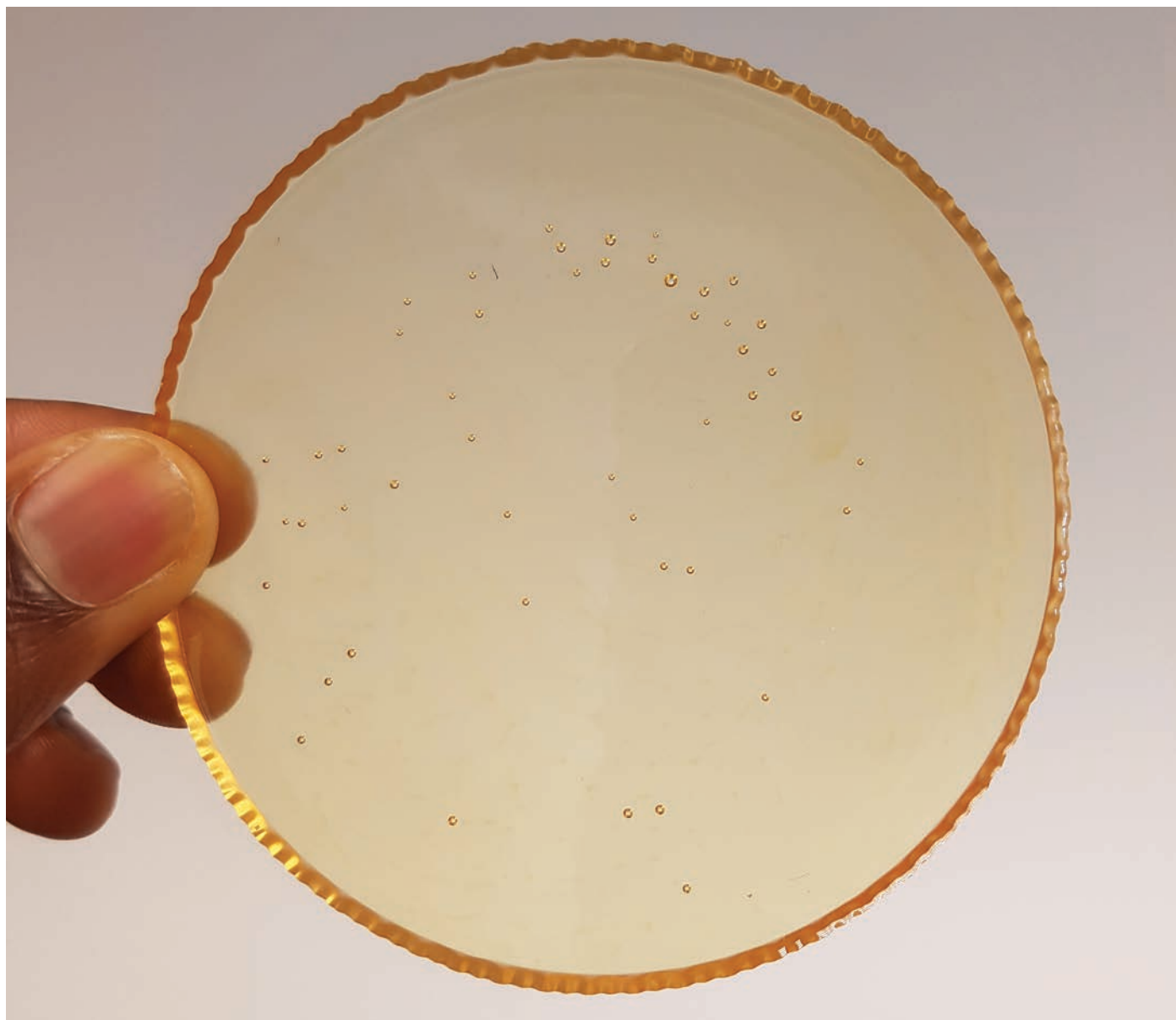
Biodiesel, aside from being renewable, has attracted substantial attention recently because it produces 57–86% fewer greenhouse gas emissions than petroleum diesel does. Consequently, the United States has shifted its attention to biodiesel production, and this has resulted in an exponential increase in the quantity of biodiesel produced. According to the US Environmental Protection Agency (EPA), US biodiesel production set a new record of 2.093 billion gallons in 2015, exceeding the previous record of 1.8 billion gallons produced in 2013. To put that in perspective, just about a decade ago, about 250 million gallons or less of biodiesel were produced per year.

- Glycerol has been used to synthesize absorbent polymeric films.
- Monoglycerides were incorporated into glycerol-co-glutaric acid polymer films.
- Monoglyceride incorporation improved the absorption and elongation properties of the polymer films.

The increased production of biodiesel is positive for the country as it attempts to rely on its own bio-based fuels and reduce its dependency on foreign oil. However, because biodiesel is mainly produced via the transesterification of vegetable oils and fats using an appropriate catalyst to form FAMES, the exponential increase in the production of biodiesel generates a concomitant increase in the production of glycerol, a major co-product of biodiesel production. In fact, about one pound of glycerol is produced for every 10 pounds of biodiesel. Even before biodiesel production levels escalated, the glycerol market was already being saturated via soap production and the hydrolysis of triglycerides to produce free fatty acids, as glycerol is also a byproduct of these reactions. Therefore, the need to find other uses for glycerol has become imperative. Fortunately, glycerol's low price, non-toxicity, and multi-functionality make it an attractive feedstock for the chemical industry. Moreover, finding new uses for glycerol could potentially reduce the cost of biodiesel production and help stabilize the market by creating new revenue streams for the industry.

Toward that end, the US Department of Agriculture (USDA) has been exploring the synthesis of absorbent polymer films using glycerol and glutaric acid (Wyatt 2014; Wyatt and Yadav 2013; Wyatt *et al.*, 2013). These polymeric films were able to act as organogels by absorbing organic solvents. This is important, as organogels have found use in food, agriculture, and medicine. In those studies, the





**FIG. 1.** Curing the gels in an oven at 150°C for 24 h produced solid, transparent films with a yellow hue.

glycerol-co-glutaric acid polymer films easily broke on swelling, and were too stiff due to excessive polymerization. In an effort to address these problems, our group conducted a study aimed at reducing the stiffness of the polymer films by incorporating monoglycerides. Hypothetically, the incorporation of monoglycerides would improve the elongation properties and result in softer, less brittle polymer films. This is because monoglycerides differ from glycerol in the number of free hydroxyl groups available for polymerization: Glycerol contains three hydroxyl groups while monoglycerides contain two hydroxyl groups.

The monoglycerides used for this study were isolated from a glyceride mixture containing glycerol, monoglycerides, and diglycerides. A combination of molecular distillation and solvent extraction techniques was used to isolate the monoglycerides. The monoglycerides were then characterized using gas chromatography-mass spectrometry (GC-MS), and were

found to have a purity level of 99.15%, comprising 1-mono-palmitin (4.46%), 2-monoolein (6.25%), 1-monoolein (86.40%), and 1-monostearin (2.05%). Six formulations were used to synthesize the polymer films. The control film contained glycerol and glutaric acid in the molar ratio 2:3. The five other formulations contained glycerol: monoglyceride: glutaric acid in molar ratios 1.9:0.1:3.0, 1.7:0.3:3.0, 1.5:0.5:3.0, 1.25:0.75:3.0, and 0.0:1.0:1.0. The polymers were synthesized by reacting the respective mixtures for 1 h at 100°C to form pre-polymers, and then further reacted for 4 h at 135°C to form polymeric gels.

The gels were cured in an oven at 150°C for 24 h, which produced solid, transparent films with a yellow hue (Fig. 1). The starting materials (glycerol, monoglycerides, glutaric acid) and their respective films were analyzed using Attenuated Total Reflectance Infrared (ATR-IR) spectroscopy to determine the extent of reaction. The ATR-IR spectra of the films showed that the starting materials had been consumed in the reaction, and all the films were completely cured.

**TABLE 1. Mechanical property measurements of polymer films**

Type of film	Elongation at Break (%)	Young's Modulus (MPa)	Tensile Strength (MPa)	Fracture Energy (J/cm <sup>3</sup> )
A	18.45 ± 1.23	8.29 ± 0.41	1.05 ± 0.03	0.105 ± 0.010
B	17.85 ± 1.74	6.50 ± 0.46	0.83 ± 0.07	0.081 ± 0.010
C	18.04 ± 1.19	5.46 ± 0.22	0.72 ± 0.04	0.071 ± 0.010
D	18.62 ± 1.71	2.84 ± 0.17	0.41 ± 0.04	0.043 ± 0.010
E	24.20 ± 3.12	0.21 ± 0.05	0.04 ± 0.01	0.21 ± 0.050
F	18.85 ± 1.61	0.26 ± 0.04	0.04 ± 0.01	0.04 ± 0.00

A – 2.0:0.0:3.0; B – 1.9:0.1:3.0; C – 1.7:0.3:3.0; D – 1.5:0.5:3.0; E – 1.25:0.75:3.0; F – 0.0:1.0:1.0. The ratios are in the order glycerol: monoglyceride: glutaric acid.

The mechanical properties measured were tensile strength (MPa), elongation at break (%), Young's modulus (MPa), and fracture energy (J/cm<sup>3</sup>). The incorporation of monoglycerides resulted in a reduction of tensile strength of the poly(glycerol-co-glutaric acid) films. The incorporation of monoglycerides also resulted in an almost 39-fold reduction in Young's Modulus and a more than 17-fold reduction in fracture energy of the resulting films. However, the incorporation of monoglycerides increased the elongation at break of the polymer films (Table 1). Correlation analysis revealed a direct linear relationship between Young's Modulus and fracture energy ( $R^2 = 0.996$ ), and between Young's Modulus and tensile strength ( $R^2 = 0.997$ ). On the other hand, there was an inverse linear relationship between Young's Modulus and elongation at break ( $R^2 = 0.643$ ). These findings show that the incorporation of monoglycerides successfully reduced the stiffness and strength of the poly(glycerol-co-glutaric acid) films, while improving their elongation properties. Moreover, it was observed that the concentration of monoglycerides incorporated into the films had an effect on the mechanical properties: The concentration of monoglycerides incorporated was directly proportional to elongation at break, and an inversely proportional to Young's Modulus, tensile strength, and fracture energy.

Solvent absorption studies were performed by first boring the films into smaller circles that were 0.75 inches in diameter. The samples were weighed ( $W_d$ ) and immersed into solvent in capped jars. The samples were removed from the jars after

10 h of incubation at room temperature, blotted with Kimwipe, and then weighed ( $W_s$ ). Weight changes due to solvent absorption was determined by the equation:

$$\% \text{ Weight change} = \frac{W_s - W_d}{W_d} \times 100$$

( $W_s$  and  $W_d$  represent weight of the swelled and dry films, respectively).

Four solvents [methyltertbutyl ether (MTBE), diethyl ether, 1,4-dioxane, and tetrahydrofuran (THF)] were used for the absorption study; they were selected based on previous findings (Wyatt 2014). In that study, MTBE and diethyl ether were found to be poorly absorbed by poly(glycerol-co-glutaric acid) films, while 1,4-dioxane and THF were strongly absorbed. As such, we selected these four solvents to investigate how monoglyceride incorporation would affect their absorption into the polymer films. Our study showed that monoglyceride incorporation improved the absorption of all four solvents into the films (Table 2). Consistent with previous findings (Wyatt 2014), the highest absorbed solvent was found to be 1,4-dioxane. In addition, the amount of solvent absorbed into the matrices of the films was directly proportional to the concentration of monoglycerides incorporated into the films. We also investigated the ability to reuse the films by drying them to a constant weight and repeating the absorption (resorption). A trend similar to that of the absorption was observed in the resorption study. Furthermore, we observed that the films resorbed more solvent than they had originally.

**TABLE 2. Maximum absorption values of MTBE, diethyl ether, THF, and 1,4-dioxane into polymer films**

Type of film	Maximum absorption (%)			
	MTBE	Diethyl ether	THF	1,4-Dioxane
A	0.0 ± 0.0	2.0 ± 0.2	54.9 ± 0.9	59.3 ± 2.0
B	1.3 ± 0.0	5.8 ± 0.3	91.9 ± 0.1	115.5 ± 2.9
C	21.5 ± 0.6	20.9 ± 0.6	100.7 ± 18.1	161.7 ± 10.7
D	32.1 ± 3.2	32.8 ± 2.0	166.2 ± 6.7	185.6 ± 14.5
E	81.9 ± 1.8	65.0 ± 5.9	216.6 ± 13.6	328.0 ± 14.8
F	170.3 ± 17.2	73.8 ± 18.2	452.6 ± 0.5	493.4 ± 19.5

A – 2.0:0.0:3.0; B – 1.9:0.1:3.0; C – 1.7:0.3:3.0; D – 1.5:0.5:3.0; E – 1.25:0.75:3.0; F – 0.0:1.0:1.0. The ratios are in the order glycerol: monoglyceride: glutaric acid.

In summary, we have successfully incorporated mono-glycerides into poly(glycerol-co-glutaric acid) films to produce poly(glycerol-co-glutaric acid-co-mono-glyceride) films with improved absorption and elongation properties.

**Prince G. Boakye** has just completed his M.S. food science and biotechnology degree at Delaware State University (Dover, Delaware). His thesis focused on the synthesis and characterization of glycerol-based polymer films for industrial applications. He worked with the Sustainable Biofuels and Co-products Research Unit at the USDA Eastern Regional Research Center (Wyndmoor, Pennsylvania) for his thesis research. He can be reached at [pgboakye@gmail.com](mailto:pgboakye@gmail.com). **Kerby C. Jones** is a chemist at the Sustainable Biofuels and Co-products Research Unit of the USDA Eastern Regional Research Center (Wyndmoor, Pennsylvania). **Nicolas Latona** is a materials engineer at the Biobased and Animal Co-products Research Unit of the USDA Eastern Regional Research Center (Wyndmoor, Pennsylvania). **Cheng-Kung Liu** is a research leader (lead scientist) and materials engineer at the Biobased and Animal Co-products Research Unit of the USDA Eastern Regional Research Center (Wyndmoor, Pennsylvania). **Samuel A. Besong** is the professor and chair of food science and nutrition at the Department of Human Ecology, Delaware State University (Dover, Delaware). **Stephen E. Lumor**

## Further reading

Wyatt, V.T., M.P. Yadav, N. Latona, and C.K. Liu, Thermal and mechanical properties of glycerol-based polymer films infused with plant cell wall polysaccharides, *J. Biobased Mater. Bioenergy* 7: 348–356, 2013, <http://dx.doi.org/10.1166/jbmb.2013.1368>.

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is a senior scientist - research and development at IOI Loders Crocklaan (Channahon, Illinois). **Victor T. Wyatt** is a research chemist at the Sustainable Biofuels and Co-products Research Unit of the USDA Eastern Regional Research Center (Wyndmoor, Pennsylvania).

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# A bio-renewable surfactant with a trick up its sleeve

Kristeen Esther Joseph and Christoph Krumm

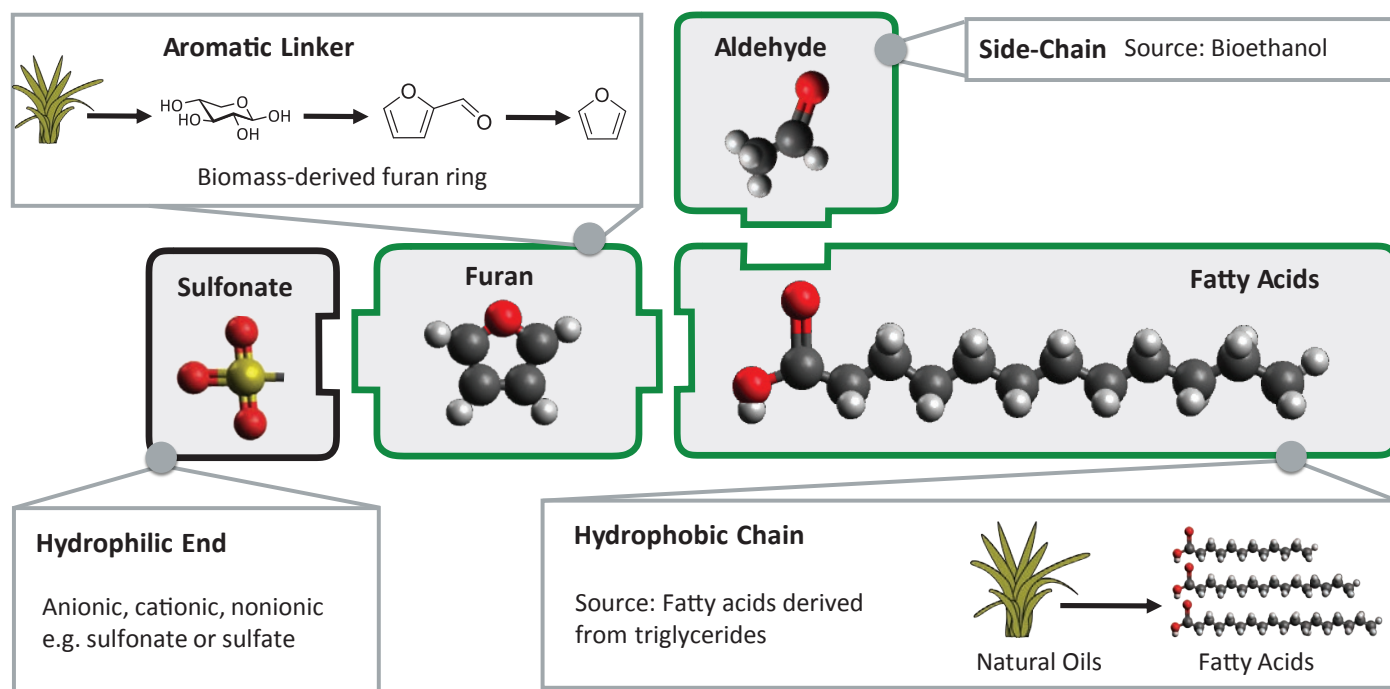
Surfactant chemicals are widely used in cleaning products, oil spill remediation, and even agricultural products, with precise structures tuned for each application. Despite years of technology development, most large-volume surfactants are made from petrochemical sources, while efforts to make renewable surfactants are focused on making existing surfactant structures from renewable sources. More recently, a new surfactant based on the natural structure and chemistry of plant-based oils and sugars has demonstrated superior function and suitability as a replacement to petrochemicals.

- Limited selectivity and tunability of petroleum-derived commercial surfactants, such as linear alkylbenzene sulfonates, result in poor hard water performance, thereby demanding the use of expensive chelants in detergent formulations.
- Oleo-furan sulfonate (OFS) surfactants are synthesized renewably from biomass-derived furans and fatty acids obtained from the hydrolysis of triglycerides found in natural oils such as coconut and soybean oil.
- OFS demonstrates a hundredfold improvement in hard water stability when compared to conventional surfactants, thereby bypassing the need for chelants.

The word “surfactant” is derived from the term “*Surf(ace) act(ive) a(ge)nt*.” The unique molecular structure of a surfactant, which consists of a hydrophobic chain coupled to a hydrophilic moiety, enables reduction in surface or interfacial tension and imparts detergency, wetting, emulsifying, dispersing, and foaming properties to liquids. This makes surfactants a key ingredient in laundry detergents, cleaning products, cosmetics, personal care products, industrial solvents, and agrochemicals. Household detergents primarily comprised of surfactants like linear alkylbenzene sulfonate (LAS) constitute over 50% of the global surfactant market, which is currently estimated at \$30 billion [1].

Current high-volume surfactant molecules are commonly made from petrochemical carbon sources such as benzene, alpha-olefins, and ethylene. Efforts to reduce reliance on fossil fuels have resulted in oleochemical analogues, which primarily use coconut oil, palm kernel oil, or algal oils for surfactant synthesis. The growing emphasis on products made from renewable carbon sources is evidenced by the development of programs such as the US Department of Agriculture *Biopreferred Product* award and the US Environmental Protection Agency *Design for the Environment* certification. Still, cost is a major limitation for plant-based surfactants. Nearly all green products come with a premium price tag. As a result, green cleaners have managed only 3% market penetration, even though over 95% of consumers say they would purchase green products if they were cheaper, according to *Packaged Facts* [2].

The challenge for oleochemical surfactants to compete with petrochemicals is further increased by their performance. Alkylation chemistry used in commercial surfactant production yields a broad distribution of structures, which limits the application-specific engineering of surfactant molecules to achieve desired properties. Large-volume oleochemical surfactants, such as sodium lauryl sulfate (SLS) have limited performance compared with petrochemical analogues, such as LAS, especially in hard water conditions; calcium and magnesium ions in hard water bind to the surfactant causing it to precipitate from the solu-



**FIG. 1. Natural structure.** Oleo-Furan Surfactants are renewably synthesized via tunable reaction chemistries from biomass-derived furans and fatty acids obtained from triglycerides.

tion. As a result, formulators of detergents incorporate costly co-formulated chelating agents (phosphates, ethylenediaminetetraacetic acid, among others) that preferentially bind to the ions to mitigate the effects of hard water and improve performance. Petroleum dependence, limited selectivity, and tunability of commercial surfactants, coupled with the use of expensive and toxic chelating agents, drive the need to find bio-renewable surfactants with improved properties. Superior bio-products will incentivize further development efforts to make sustainable products economically viable and competitive against existing fossil-based commercial counterparts. A recent paper by Park, D.S., *et al.*, "Tunable oleo-furan surfactants by acylation of renewable furans," reveals a new renewable surfactant that boasts 100x greater hard water stability, eliminating the need for chelating agents [3]. The result could make bio-renewable detergent formulations even cheaper than their petrochemical counterparts.

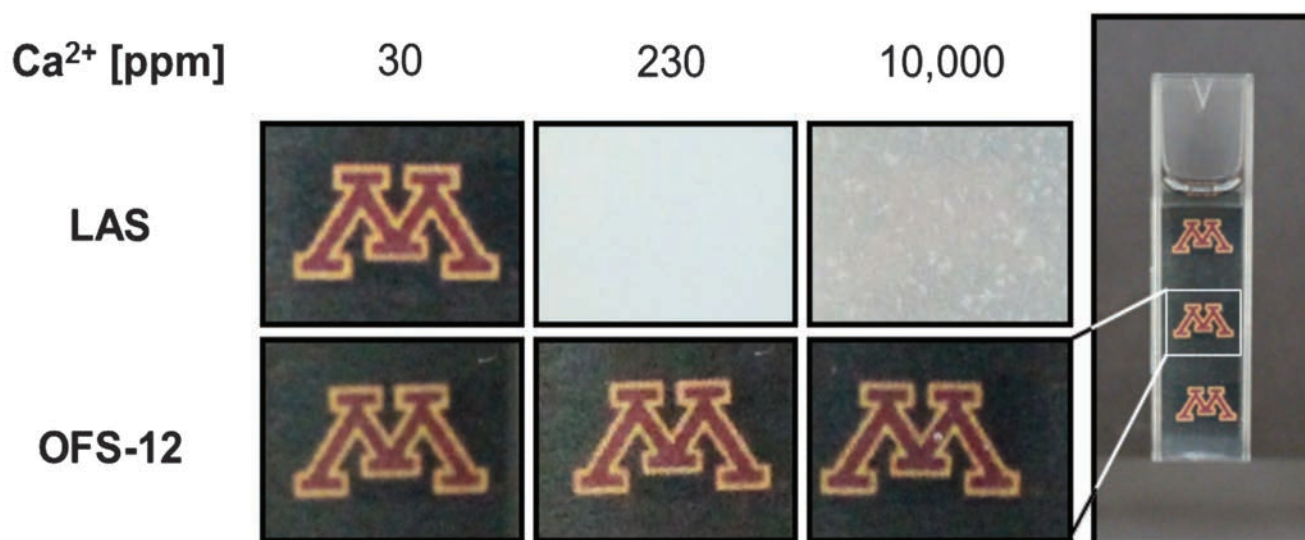
## OLEO-FURAN SURFACTANTS: NATURAL STRUCTURE FOR NATURAL FUNCTION

The natural oil- and sugar-derived structures of oleo-furan surfactants harness their inherent function for improved detergency, solubility, and exceptional stability in hard water conditions. OFS precursors are synthesized through the acylation of sugar-derived furan with natural oil-derived fatty acids. Furan is synthesized renewably from xylose, a sugar found in lignocellulosic biomass, while fatty acids are obtained from hydrolysis of triglycerides found in various natural oils, such as coconut and soybean oil (Fig. 1).

The synthesis of OFS molecules is highly tunable. Surfactant properties can be selected by using different sources of triglycerides and by coupling various chemistries to obtain a wide range of surfactants incorporating linear or branched structure—or different chemical functionalities. Fatty acids with varying chain lengths, from 7 to 18 carbon atoms, were used to synthesize a wide range of surfactants with different hydrophobic tail lengths. The coupling of chemistries such as acylation, hydrogenation, aldol condensation, and sulfonation aid in the synthesis of linear, branched, and functionalized surfactants exhibiting high control over the tunability of reactions and surfactant structure. High yield and selectivity (>90%) toward the desired OFS precursor can be achieved in the presence of a zeolite catalyst. Using the aforementioned chemistry, 10 different OFS surfactant prototypes were synthesized in high purity with variation in the chain length and functionality of the hydrophobic group.

## EVALUATION OF OFS PERFORMANCE: ENHANCED HARD WATER STABILITY

Surfactant performance was evaluated by testing standard properties, such as critical micelle concentration (CMC), Krafft point, foaming, wetting, and stability in hard water. Ideally, surfactants should possess low CMC and Krafft points to ensure a wide range of operating conditions, especially in detergency applications. In comparison with LAS, OFS surfactants were found to have similar values of CMC and lower Krafft points, enabling application in dilute conditions and cold water. More importantly, OFS were found to possess superior detergency in



**FIG. 2. Hard water performance of oleo-furan surfactants.** Comparison of performance in hard water for OFS surfactants with LAS reveals that most oleo-furan surfactants remain clear and functional in hard water conditions when viewed through a cuvette, while conventional surfactants such as LAS become cloudy (230 ppm) and precipitate (10,000 ppm).

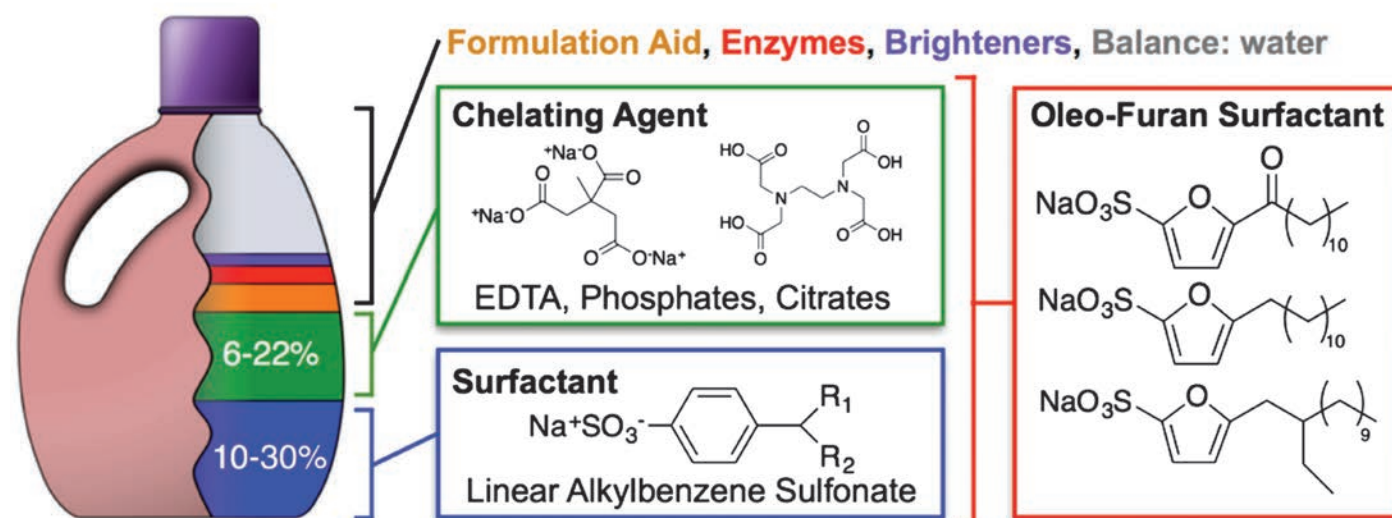
(Image Source: Park, D. S., *et al.*, ACS Central Science 2: 820–824, 2016, <http://pubs.acs.org/doi/pdfplus/10.1021/acscentsci.6b00208>)

hard water conditions. While LAS precipitates at low ion concentrations (230 ppm) and forms a cloudy solution rendering it unusable, OFS remains stable in solution even at hundred-fold ion concentration (Fig. 2). About 85% of the water in the United States is hard (>200 ppm of Ca<sup>2+</sup>) and unsuitable for direct use with surfactants [4].

This drawback necessitates the use of chelants as additives when using LAS in a formulation, as the chelants trap ions in hard water to ensure minimal interference of Ca<sup>2+</sup> in surfactant performance. Such chelants are expensive, and many are banned due to their detrimental impact on the environment. OFS molecules, on the other hand, eliminate the use of a chelants, which significantly simplifies the ingredient list of a formulation and reduces toxicity and cost (Fig. 3). They were also

found to produce equal amounts of foam and possess fast fabric-wetting characteristics.

Major drawbacks associated with commercial surfactants are dependence on fossil-based resources, lack of tunability, and the need for hazardous additives in detergent formulation. The driving force for synthetic, petroleum-derived surfactants is their ready availability and low price, whereas the market for bio-based surfactants is driven by increasing consumer awareness of ecological benefits, and the availability of a wide variety of raw materials. OFS surfactants have the added benefit of superior performance when compared with synthetic surfactants like LAS. Apart from being bio-renewable, OFS molecules have revealed high tunability during synthesis, good surfactant properties, and superior stability in hard water—all



**FIG. 3. Dual functionality of oleo-furan surfactants.** Water-based linear alkylbenzene sulfonate (LAS = blue) surfactants require metal chelating agents (green), both of which are replaced by a single oleo-furan sulfonate surfactant (OFS = red).

(Image Source: Park, D. S., *et al.*, ACS Central Science 2: 820–824, 2016, <http://pubs.acs.org/doi/pdfplus/10.1021/acscentsci.6b00208>)



of which are expected to be of high importance for detergents and other applications, where the water used for formulations or cleaning do not necessarily undergo pretreatment. The ability to tailor furans with desired functionality in the final surfactant molecule enables the expansion of this chemistry to make a wide variety of OFS and opens the doors to explore the potential of furan-based surfactants for other applications.

*Kristeen Esther Joseph earned her Bachelor's Degree in Chemical Engineering from the Institute of Chemical Technology, Mumbai (India) in 2013. She is currently pursuing her Ph.D. in Chemical Engineering at the University of Minnesota. Her research is focused on developing renewable surfactants that exhibit superior performance. She can be reached at josep370@umn.edu.*



*Christoph Krumm is the founder of Sironix Renewables, a University of Minnesota-based start-up company developing bio-renewable surfactants for laundry detergent applications. Christoph earned a BS in Chemical Engineering from the University of Washington, and his PhD in Chemical Engineering from the University of Minnesota. He can be reached at ckrumm@sironixrenewables.com.*

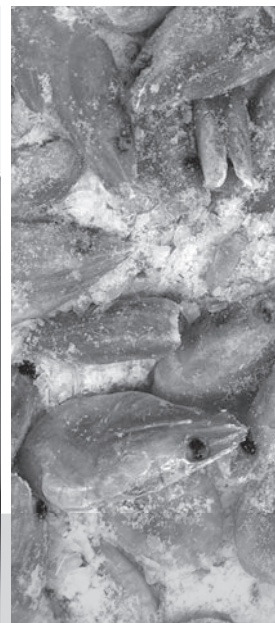


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# Sophorolipids from a winterization oil cake

Pedro Jiménez-Peñalver, Xavier Font, and Teresa Gea

Sophorolipids (SLs) are glycolipid biosurfactants that can serve as “environmentally friendly” alternatives to chemically produced surfactants in household cleaning products, cosmetics, and other commercial products. SLs are considered to be the most promising biosurfactants because production yields are high and substrate conversion can be achieved by fermentation of non-pathogenic yeast strains such as *Starmerella bombicola*. SL molecules are glycolipids comprised of a sophorose moiety coupled with a hydroxylated fatty acid. They are produced as a mixture of different molecules with two major points of variation: 1. acetylation in the sophorose and 2. lactonization (Fig. 1). SLs are conventionally produced under submerged fermentation (SmF) using glucose as the hydrophilic carbon source of the synthesis, and a high-oleic-acid feedstock as the hydrophobic carbon source (Van Bogaert et al., 2011).

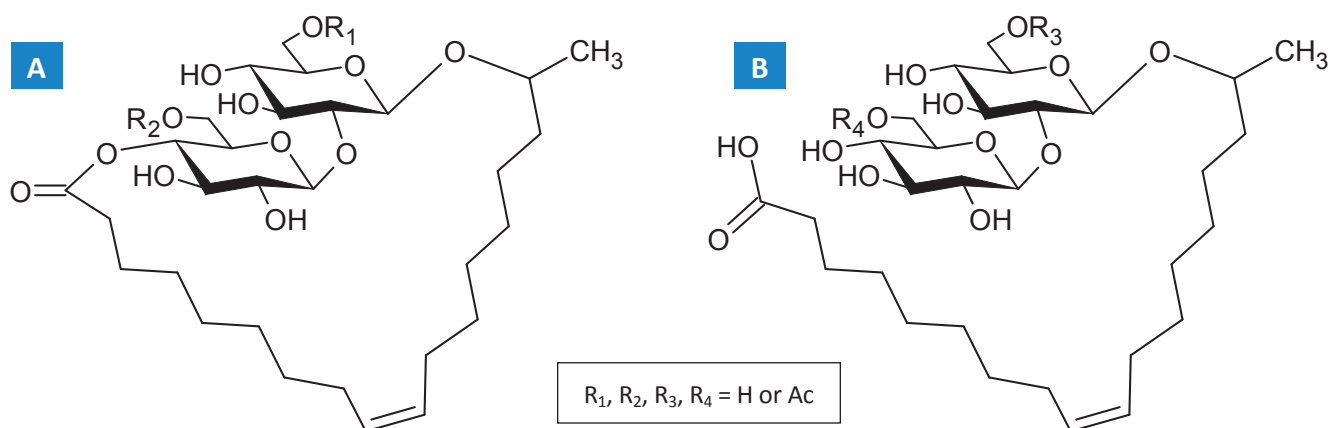
- Residual oil cakes from vegetable oil refining are suitable substrates to produce sophorolipids by solid-state fermentation.
- Promising yields of 0.235 g of crude sophorolipid per g of dry substrate were obtained, and the scale-up of the process is being assessed.
- A successful scale-up would present new opportunities to valorize residual oil cakes from a variety of oils by using them as substrates for biotechnological processes.

Solid-state fermentation (SSF) is an alternative technology for SL production. SSF has become more feasible in biotech industries during the last decade due to its potential applications in the production of enzymes and other bioproducts, and a growing knowledge of bioreactor design (Mitchell *et al.*, 2006). Using SSF to produce SLs presents two important advantages over SmF (Krieger *et al.*, 2010). First, SSF avoids potential problems associated with foaming. Second, SSF allows the use of inexpensive solid substrates, such as the huge quantities of oil cakes that are generated during vegetable oil refining.

Our research group recently studied the feasibility of using SSF to produce SLs from a winterization oil cake (WOC) obtained from a local vegetable oil refinery (Lípidos Santiga S.A., Barcelona, Spain). Process dynamics of the SSF process were studied through respirometry.

## SUBSTRATES AND SSF MIXTURE

The WOC we used is an agro-industrial waste obtained by cooling sunflower oil to 5°C for 24 h, and then filtering it with diatomaceous earth to remove the waxes. The chemical composition of the WOC depends on the efficiency of the winterization process and the quality of the raw oil. WOC has a high



**FIG. 1. Structure of natural sophorolipids (A) lactonic form and (B) free acid form**

fat content, about 60–70% of dry mass. Sugar beet molasses (MOL)—a by-product of the sugar industry that was supplied by the sugar company AB Azucarera Iberia S.L. (Madrid, Spain)—was used as a co-substrate of the SSF. These two substrates were mixed in a ratio 1:4 MOL:WOC (w/w), and blended with wheat straw, which acts as inert support for the SSF (Jiménez-Peñalver *et al.*, 2016).

The main characteristics of the substrates and the SSF mixture are summarized in Table 1.

WOC provides fats to the mixture, which act as the hydrophobic carbon source for the synthesis of SLs. MOL provides sugars, which act as the hydrophilic carbon source, and also provides nitrogen and micronutrients such as Mg, K, or Na, which decreases or even eliminates the need to supplement the yeast with minerals. (Van Bogaert *et al.*, 2011). The physical appearance of the substrates and the SSF mixture are depicted in Fig. 2, page 22.

Fermentations were carried out at 100-g scale, at 30°C, with an air supply of 0.30 L air kg<sup>-1</sup> min<sup>-1</sup>. More information about the experimental set-up can be found at Jiménez-Peñalver *et al.*, 2016.

## PROCESS DYNAMICS

The SSF process was carried out over 10 days to determine the time course of SL production (Fig. 3, page 22). SLs production was detected since the first sampling on day 3.

The maximum yield was reached on day 10, with a total amount of 0.179 g per g DM, which corresponds to 19.1 g per



**TABLE 1. Main characteristics of the substrates and the initial SSF mixture**

	Winterization oil cake	Sugar beet molasses	SSF mixture
pH	4.75 ± 0.01	6.79 ± 0.06	6.54 ± 0.03
Moisture (%)	0	16.9 ± 0.8	44.7 ± 0.9
Organic matter (% db)	67.5 ± 2.5	87.3 ± 2.1	74.7 ± 0.4
Fat content (% db)	<b>67.5 ± 2.5</b>	n.d.	39.0 ± 0.2
Carbohydrate content (% db)	n.d.	<b>78.4 ± 2.8</b>	10.6 ± 1.1

Abbreviations: db, dry basis; n.d., not detected. Data presented as mean ± standard deviation of the sample.



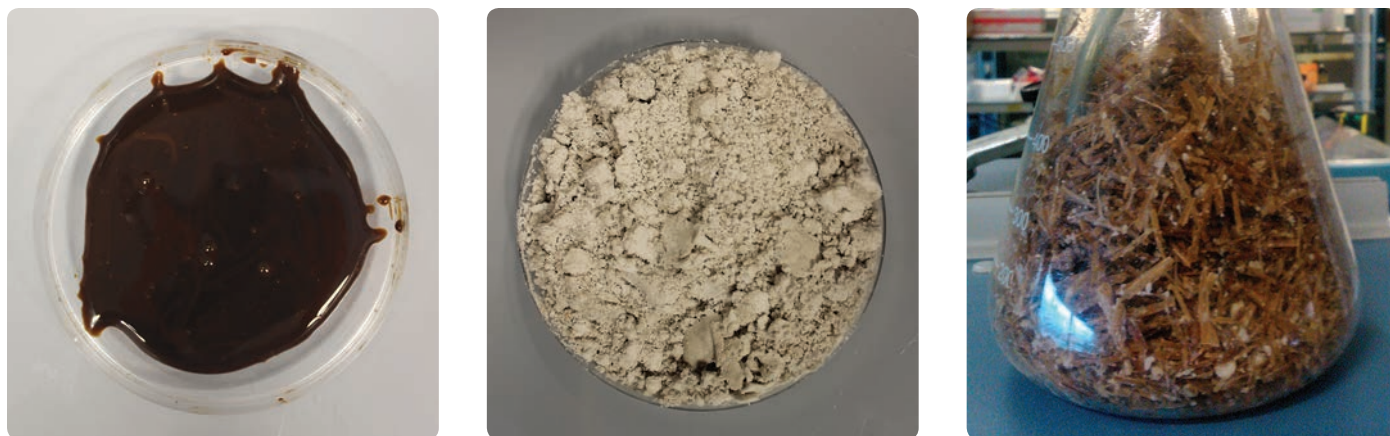


FIG. 2. Pictures of the sugar beet molasses (left), the winterization oil cake (center), and the SSF mixture (right)

100 g of wet substrates. SLs were amber colored, honey like semi-crystalline molecules as seen in Fig. 4.

Respirometry was used as an indirect way to study the biological activity during the SSF processes. The oxygen concentration in the exhaust gases was recorded and used to calculate the Oxygen Uptake Rate (OUR) and the Cumulative Oxygen Consumption (COC) as described by Ponsá *et al.* (2010). Fig. 3 shows OUR and COC profiles over the 10 days. OUR reached its maximum value at 66 h ( $1.92 \pm 0.21$  mg O<sub>2</sub> g<sup>-1</sup> DM h<sup>-1</sup>, average  $\pm$  standard deviation for 4 fermentations), and the oxygen consumed at the end of the fermentation was 0.330 g O<sub>2</sub> per g DM. Further experiments performed in our laboratory demonstrated that there is a linear correlation between the SL yield and the COC, i.e.; the oxygen consumed during the fermentation (Jiménez-Peñalver *et al.*, 2016). This correlation suggests that respirometry can be a useful tool for monitor-

ing SL production by *S. bombicola* in SSF. Monitoring of SSF processes was identified as one of the challenges needing to be addressed before this technology can be used to produce biosurfactants at commercial scale (Krieger *et al.*, 2010). In this sense, our research group brought considerable experience to the table—not only with respect to monitoring SSF processes for the production of biosurfactants, but also in the production of enzymes, aromatic compounds, or biopesticides.

Fig. 3 also shows the profile of fat content expressed as grams of fat per gram of total dry matter in the fermentation medium. Approximately half of the initial fats were consumed at the end of the fermentation. Unused fats remaining in the reactor may have been composed of waxes not degraded by *S. bombicola* or fats that are not accessible to the yeast (such as fats contained within the innermost portions of the bigger particles). Further experiments performed with intermit-

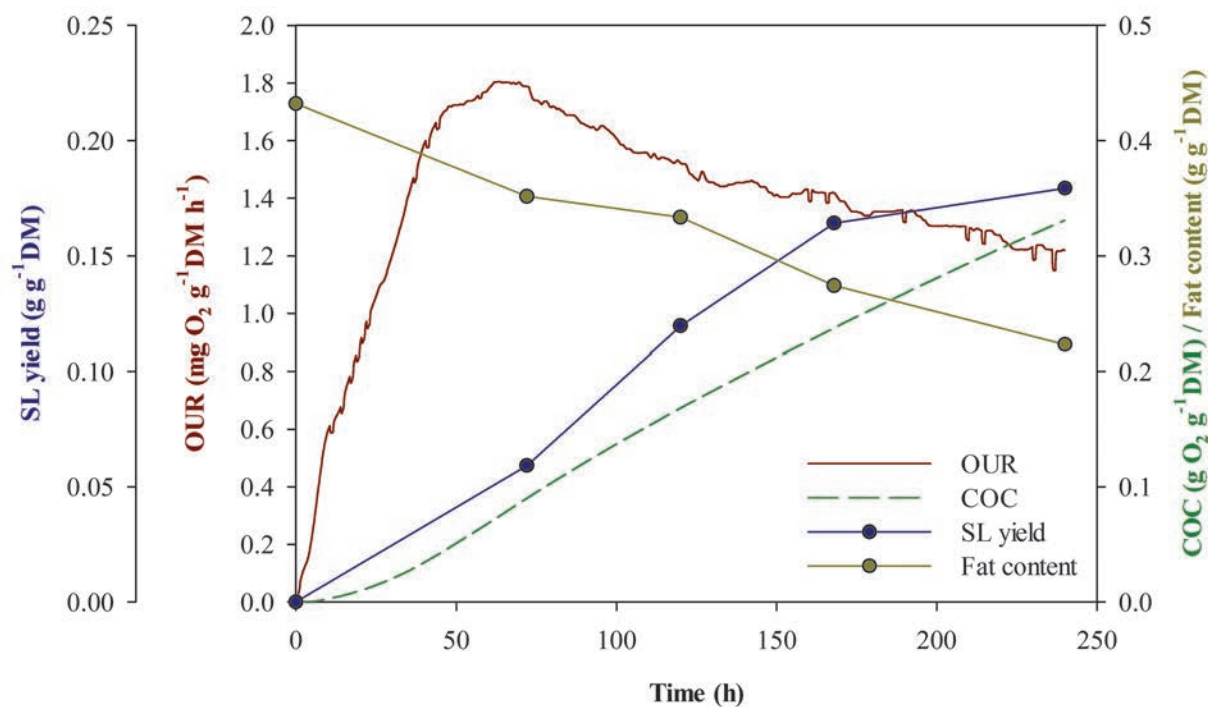


FIG. 3. Solid-state fermentation profile of the process: OUR and COC profiles and time course of SL yield and fat content

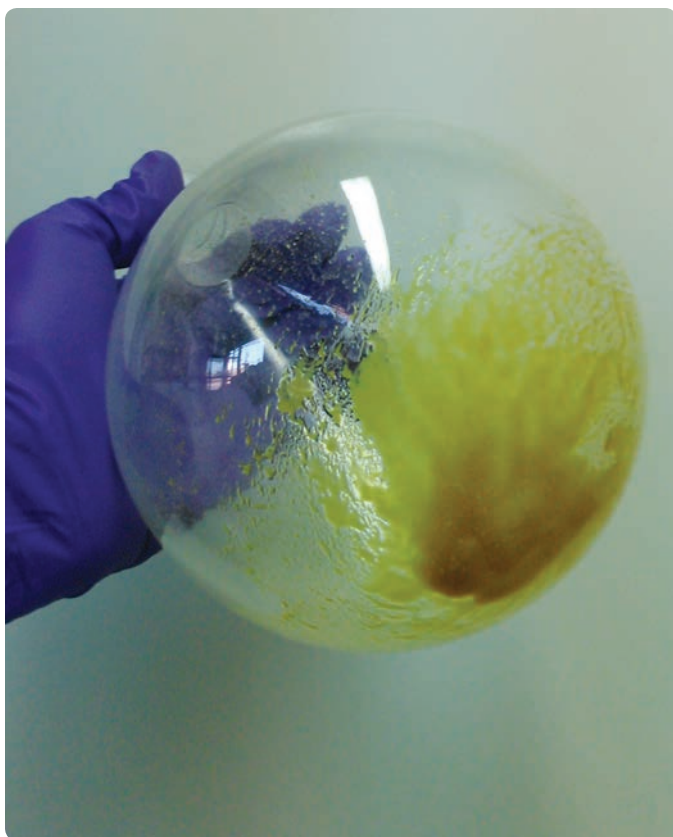


FIG. 4. Amber colored, honey-like semi-crystalline sophorolipids

tent mixing of the SSF mixture showed a 22% increment in the fat consumption and a 31% increase in the SL yield, which was 0.235 g per g of DM or 25.1 g per 100 g of wet substrates (Jiménez-Peñalver *et al.*, 2016). From these observations, we concluded that intermittent mixing increases the bioavailability of substrates to the yeast; therefore, more fats are consumed and more SLs produced.

## A PROMISING PROCESS

As our studies demonstrated, WOC is a feasible substrate for producing SLs by SSF using sugar beet molasses as the co-substrate and *Starmerella bombicola* as the microorganism. The SL yield at 10 days of fermentation was 0.179 g per g of DM, and increased to 0.235 g per g of DM when intermittent mixing was performed to the SSF mixture. The use of WOC to produce high-value products such as SLs could valorize vegetable oil refining by providing new revenue streams for a waste product that would otherwise be composted. Scale-up experiments using a 200-L rotating reactor are being conducted to evaluate the economic feasibility of the process. Moreover, oil cakes from other types of oil are being considered as substrates for SL production.

## Further reading

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Xavier Font and Teresa Gea are professors in the Department of Chemical, Biological and Environmental Engineering at UAB, and researchers in the Composting Research Group (GICOM, [www.gicom.cat](http://www.gicom.cat), [info@gicom.cat](mailto:info@gicom.cat)). The group has conducted research on the composting and anaerobic digestion of industrial and urban wastes since 1999. More recently, the group's research has shifted to increasing the valorization of wastes beyond compost and biogas by using solid state fermentation to obtain valuable products and change the paradigm from waste to feedstock to from waste to product.

The authors thank the Spanish Ministerio de Economía y Competitividad (Project CTM2015-69513-R) for their financial support. Pedro Jiménez has a doctoral scholarship from the Spanish Ministerio de Economía y Competitividad (BES-2013-064852).

# Separating **lipids** from a **fermentation** **broth** can dramatically improve **bioprocess** **performance**

Ben Dolman and James Winterburn

- Integrated separation can be used to remove fermentation products from a bioreactor before they can inhibit production or create other processing challenges.
- This article describes the development of a gravity-based separation system that can be used to separate insoluble bio-products from a fermentation broth as they are produced.
- The separation process was successfully applied to sophorolipid biosurfactants and dramatically improved sophorolipid production.

The products of many fermentative biochemical processes inhibit continued production and create other bioprocessing challenges that drastically reduce production rates and titers. Consequently, finding new ways to remove biological products from bioreactors as such products are produced—a process known as integrated product recovery—has become a major focus of research. The techniques typically used to recover bulk biochemicals, such as distillation and solvent extraction, often cause irreversible damage to the producing microorganisms. To enable continued production, the producing cells must first be removed (often by centrifugation) before the product can be separated. Such separation procedures are usually cost-prohibitive on an industrial scale; hence, few bioprocesses employ integrated separation.

Meanwhile, interest in a number of oleochemicals that could potentially serve as alternatives to petroleum-derived fuels and chemicals continues to grow. Until recently, these oleochemical alternatives have primarily been produced intracellularly, with an expensive cell disruption required to separate the product. A number of companies and institutions have since developed organisms which secrete the product directly



into the media, a trend that is likely to continue with further advances in genetic engineering.

Sophorolipids, along with mannosylerythritol lipids, are in a class of naturally occurring, microbially produced glycolipids that are frequently used as biosurfactants. Sophorolipids are composed of a sophorose sugar joined to a fatty acid chain. A number of different molecular structures are possible, making it possible to customize the characteristics of sophorolipids somewhat by tuning their structure. A common sophorolipid structure is shown in Fig. 1.

During fermentation, an insoluble product phase is formed once a certain glycolipid concentration is reached. This enables product titers of up to  $400 \text{ g l}^{-1}$  and  $165 \text{ g l}^{-1}$  to be reached for sophorolipids and mannosylerythritol lipids, respectively. Glycolipid biosurfactants are highly surface-active, making them ideal for cleaning applications. They also have antimicrobial properties, are biodegradable, and non-toxic—properties that, combined with high product titers, have facilitated their commercial use in a number of high-end personal care, cosmetic, and cleaning products.

Due to the high product titers that can be achieved, and the lack of product inhibition, little effort has been devoted to integrating the production and separation of glycolipid biosurfactants. However, increasing interest in using glycolipid biosurfactants for lower-value, bulk applications, where cost is one of the most important factors, such as bioremediation and enhanced oil recovery, highlights the importance of improving fermentation efficiency.

In sophorolipid production, the yeast *Candida bombicola* converts a sugar and fatty acid into sophorolipid. Because *Candida bombicola* has a high oxygen demand, oxygen must be transferred to the system by bubbling air through the bioreactor and stirring the system vigorously. However, after 8–12 days of fermentation, the fermentation broth becomes viscous due to higher quantities of sophorolipid in the bioreactor (about  $300\text{--}400 \text{ g l}^{-1}$ ). The viscosity of the sophorolipid phase makes it difficult to provide sufficient agitation to the bioreactor. At this point, the cells' biological oxygen demand can no longer be met, meaning the fermentation must be stopped, the sophorolipid recovered, the system cleaned, and the process started again—at significant expense and loss of production time. Further processing challenges are caused by the use of high cell densities in sophorolipid-producing fermentations which can facilitate high productivity of over  $3 \text{ g l}^{-1}\text{h}^{-1}$ , but require high substrate feeding rates. This leads to significant volume increases in the bioreactor, and the bioreactor volume capacity can soon be reached. The fermentation must therefore be stopped, limiting production and requiring expensive cleaning and start up procedures.

To overcome these challenges, our research group has developed a gravity-based separation system that can be used to recover the sophorolipid-rich phase from the bioreactor (Fig. 2. and Fig. 3, page 26.) The sophorolipid-rich fermentation broth is pumped from the bioreactor to the separator, where the flow conditions are laminar, creating an environment that allows the sophorolipid droplets within the broth to coalesce and form a continuous, separate phase. The sophorolipid-rich

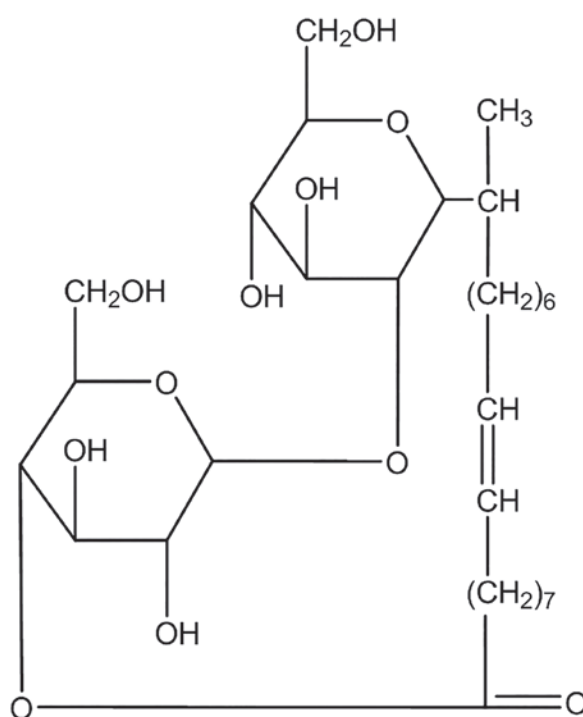
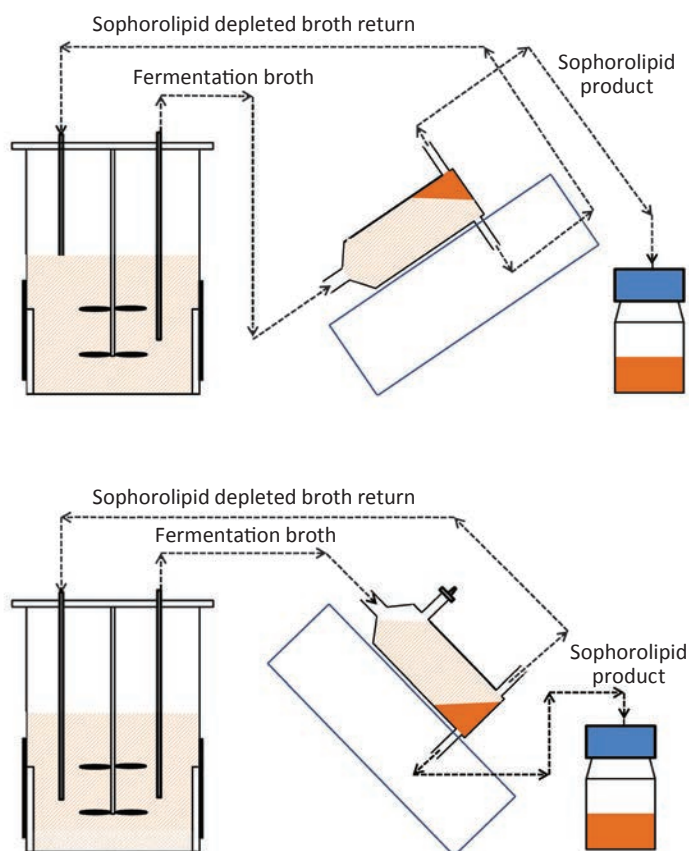


FIG 1. Common structure of a sophorolipid molecule

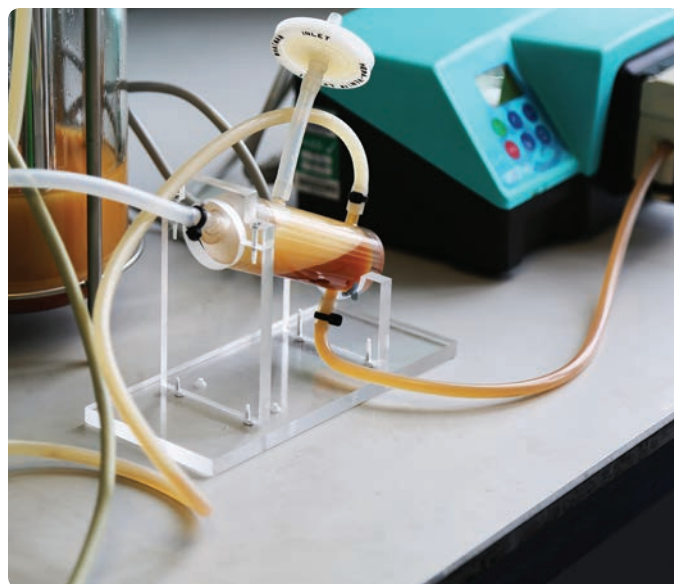


**FIG. 2. Diagram of integrated separation system in use with sophorolipids**

phase either settles to the bottom of the separator or rises to the surface, depending on the relative density of the product phase and the media. This relative density is determined by the fermentation conditions, and can be effectively controlled. The product phase is pumped out of the separator into a collection vessel, and the cells, substrate, and media are returned to the bioreactor to continue sophorolipid production.

We have successfully applied this system to a number of sophorolipid producing fermentations, recovering up to 98% of the sophorolipids produced. By removing the sophorolipid phase, we are able to continue fermentation past the point at which it would normally have to be stopped due to excessive broth viscosity and the bioreactor volume capacity being reached. This has enabled us to produce almost 800 g sophorolipid per liter of initial broth, and to reduce the power input for stirring by 34 % by the end of a fermentation. These results suggest that this system could dramatically reduce the cost of sophorolipid production by reducing the bioreactor volume required, reducing the number of batches (and therefore expensive inoculum preparation and cleaning cycles), and providing a simple and inexpensive separation technique. The separation technology has been successfully trialed at pilot scale.

What's more, the system could similarly be applied to other insoluble biomolecules. Complete phase separation of many lipid products is difficult to achieve during fermentation. This is because the surface-active species in the broth tend to stabilize the lipid-water emulsion that forms. While this gravity-based system could not separate the lipids from an emul-



**FIG. 3. The separation system being used for integrated separation**

sion, it could separate the emulsion from the fermentation broth and reduce the effects of product inhibition and other processing challenges. Finally, this crude separation could greatly reduce the volume passed to subsequent downstream processing, and dramatically lower the cost of this often economically prohibitive step.

Working with the University of Manchester Intellectual Property team, our research group has filed a patent for our technology, and we are interested in working with collaborators both to demonstrate the technology on other insoluble products and to commercialize the process.

**Ben Dolman** is a Chemical Engineering PhD student at The University of Manchester. He has developed the technology described in this article as part of his doctoral program, which is supervised by **James Winterburn**, a Lecturer in Chemical Engineering at The University of Manchester. Much of the work described here has been carried out with funding from a BioProNET BIV, entitled "Novel bioprocessing approaches for the separation of product phases." Dolman can be contacted at [ben\\_dolman@hotmail.co.uk](mailto:ben_dolman@hotmail.co.uk).

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Conference presentation video at: <https://www.youtube.com/watch?v=eqiX90k018g>.



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welcome

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# A process design to increase the efficiency of batch oil deodorizers

Daniela S. Laoretani and Oscar A. Iribarren

- Batch deodorization is used for small production capacities (e.g., 50 to 100 tons/day) of edible oil or, due to its flexibility, in plants where the type of oil to be processed is frequently changed. However, the times required to process a batch are quite long, with high stripping steam consumption per ton of treated oil.
- A processing alternative that strongly improves the efficiency, is to couple the batch deodorizer to a continuous desorption column: The steam exiting the batch deodorizer is fed to the bottom of the column, while the oil contained in the batch vessel is recycled through the top of the column and then returned to the vessel.
- An efficient (and practical) mechanical design is in progress to implement this alternative, in which a small packed column section is placed inside the batch deodorizer vessel, and the oil from the bulk batch holdup is raised to the top of the column section by steam lifting.

## BATCH DEODORIZATION

Batch deodorization treats the oil in a closed container with direct steam injection through a distributor, to strip the undesirable components. This is attractive in several respects: It is easy to design and to operate, has a low investment cost, and is very flexible for product changeovers. However, it also has disadvantages: The times required to process a batch are quite long (e.g., 8 to 12 h, which implies a low productivity) due to poor vapor-liquid contacting efficiency, and the stripping steam consumption per ton of treated oil is high (Gavin, 1981; O'Brien, 2009). At industrial scale, the stripping steam requirement for batch deodorizers is about 2 to 4% of the oil to be treated, whereas continuous and semi-continuous deodorizers need 0.75 to 1.5%, and continuous thin-film systems can operate with as little as 0.3 to 0.6% steam (Carlson, 1996).

## SEMI-BATCH PROCESSING

Laoretani and Iribarren (2017) explored a processing alternative aimed at improving the efficiency of batch deodorizers, coupling them to a small continuous desorption packed column: The steam exiting the batch deodorizer was fed to the bottom of the column, while the oil contained in the batch vessel was recycled through the top of the column and then returned to the vessel. This strongly increased the efficiency of separation, reducing stripping steam consumption and processing time. This processing alternative is depicted in Figure 1, and was assessed through a case study of soybean oil with an initial content of 5% free fatty acids (FFA) to be stripped up to a product specification of 0.05% FFA, at 210°C, and a pressure of 5 mmHg. Batch sizes of 10,000 kg were considered, and a steaming rate of 3 kg/h steam per 100 kg of oil being treated was applied, as recommended by Gavin (1981), to get an appropriate contact between the phases.

The process was optimized using Douglas (1988) "profit" as objective function, which takes into account revenues, labor, operating (stripping steam, heating and cooling utilities, and electricity), and annualized capital (the deodorizer, the column, and a pump) costs. The optimization variables were



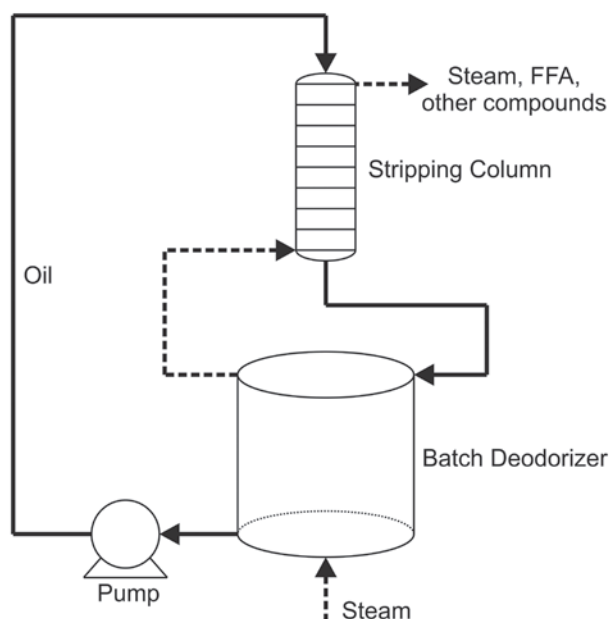


FIG. 1. Semi-batch process proposed in Laoretani and Iribarren (2017)

the number of equilibrium stages in the column and the flow rate of oil recycled. To illustrate the effect of the number of stages, Figure 2 depicts depletion of FFA vs. stripping time for 0 (no column at all), 1, 2, 3, 4, and 5 equilibrium stages in the column. It can be seen that just one stage improves the performance of the deodorizer significantly, after which the impact is more modest; the optimal is at about 4 stages.

The optimal semi-batch design reduces the stripping time by about 40% (batch conventional 3 h, semi-batch 1.8 h). This leads to an increase in productivity: The original 1,720 batches per year can be increased to 1,845. The batch deodorizer is the same for both alternatives, with a diameter  $D = 2.5$  m and a height  $H = 5$  m (half full with oil); the semi-batch alternative adds a pump of 1 K Watt, and the desorption column has  $D = 1$  m and a height that can contain 4 equilibrium stages (which, with structured packing, is about 2 m).

## ECONOMIC ASSESSMENT

The capital investment is about 25% larger in the semi-batch; the operating costs also increase (mainly because the number of batches increase) by 10%, but revenues increase 40% (due to a larger production), which leads to a net increased benefit of \$75,000/yr with the semi-batch. This processing alternative should be attractive for facilities that have already adopted batch technology, and even more so if the raw material to be deodorized is palm oil or bait, where the initial content of FFA is high. In existing plants where deodorization is a bottleneck, the benefit will be more noticeable. In plants where deodorization is not a bottleneck, semi-batch processing may

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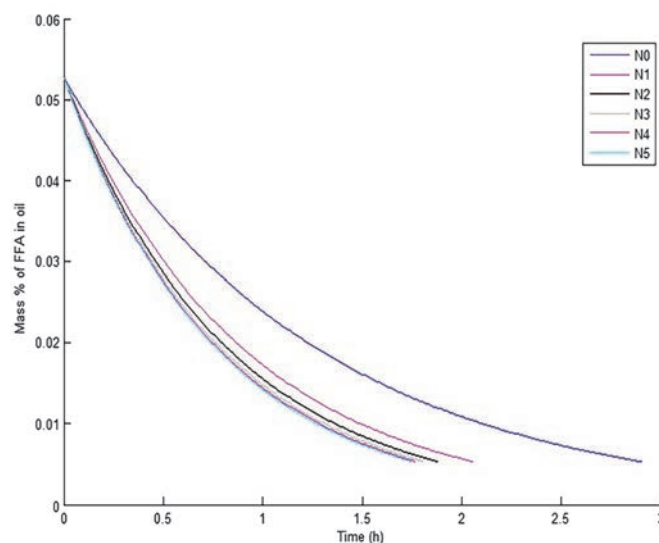


FIG. 2. Content of free fatty acid (FFA) in oil as a function of deodorization time

lead to a reduction of labor costs (reducing the number of shifts required), and in the case of a new plant construction project, the cost impact will be mainly due to a lower size of deodorizers.

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# Where's the (omega-3) beef?

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

Laura Cassiday

To help stave off heart disease and reap other health benefits, the American Heart Association recommends consuming two servings of fish per week. Oily fish such as salmon, tuna, and mackerel are rich in omega-3 fatty acids, which have been shown in some studies to reduce the risk of cardiovascular disease, ease depression, and promote the neurological development of infants. Yet many people do not like the taste of fish, and scientists are concerned about the long-term sustainability of increased fish consumption. Therefore, researchers are exploring techniques to incorporate omega-3 fatty acids into beef, a more popular and abundant protein source than fish in the United States and some other countries.

"As a society, Americans' consumption of fish, especially fish that contributes to these omega-3 fats, is quite low compared to other proteins," said Jim Drouillard, professor of animal sciences and industry at Kansas State University, in Manhattan, Kansas, USA, in a news release dated February 4, 2013 (<http://tinyurl.com/greatobeef>). "Reasons for this include cost, access to fish, and personal preference. Americans do, however, like hamburgers. So if we can give people a hamburger that is rich in omega-3s, it's an alternative form of a product that they already eat and does not require a lifestyle change, which is difficult to make."

Drouillard and his colleagues found that feeding cattle flaxseed, which is rich in omega-3 fatty acids, produced omega-3-enriched beef. To maximize the transfer of omega-3s from flaxseed to beef, the researchers encapsulated the flaxseed in a dolomitic lime matrix (Alvarado-Gilis, C. A., *et al.*, <http://dx.doi.org/10.2527/jas.2015-9171>, 2015). This encapsulation protected omega-3s in the flaxseed from conversion to saturated fats by microorganisms in the cow's rumen.

A quarter-pound hamburger made with the enriched ground beef provides about 200 mg of omega-3 fatty acids, mainly in the form of alpha-linolenic acid (ALA). For comparison, a 4-ounce serving of salmon contains about 1,600 mg of omega-3s. A similar serving of grass-fed beef contains about 20–30 mg of omega-3s. According to Drouillard, sensory panel tests have not detected any significant differences in the flavor profiles of omega-3 beef and conventional beef.

Omega-3-rich ground beef produced by Drouillard's approach has been sold on a test basis in supermarket chains

in Texas and the northeastern United States under the name GreatO Premium Ground Beef (<http://greatofoods.com>). "Currently, GreatO beef is not being sold anywhere," says Todd Hansen, chief executive officer of NBO3 Technologies ([www.nbo3.com](http://www.nbo3.com)), the company that markets GreatO. "We did some consumer research, and we're repositioning it for a relaunch later in 2017. There's certainly a consumer desire for it. We just have to figure out the best way to communicate to the consumer through our packaging."

Although ALA is an essential fatty acid found in seeds, nuts, and soybeans, it may not be the best choice for omega-3 fortification of beef. Most of the cardiovascular and other health benefits of omega-3 fatty acids have been attributed to docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA), the omega-3 fatty acids abundant in oily fish. In the human body, ingested ALA can be converted to EPA and DHA, but the efficiency is very low, on the order of a few percent for EPA and even less for DHA. However, Drouillard notes that flaxseed contains anti-inflammatory and other beneficial compounds, in addition to ALA, that are likely also transferred to beef.

To enrich beef with the same omega-3s found in oily fish, researchers led by Shawn Archibeque, associate professor of animal sciences at Colorado State University, in Fort Collins, USA, and Clint Krehbiel, professor of animal science at Oklahoma State University, in Stillwater, USA, fed cattle algae that produce DHA and EPA. Microalgae are the source of DHA and EPA in fish, as the omega-3s accumulate in the marine food chain. The challenge was finding the right amount of



algae to add to cattle feed to supply sufficient DHA and EPA without affecting the cow's appetite.

The researchers had consulting agreements with the company Omega3Beef (Oklahoma City, USA; <http://www.omega3beef.com>), which seeks to commercialize the omega-3-enriched beef. According to Don Van Pelt Smith, chief executive officer and founder of Omega3Beef, the type of algae is also important. Smith and his colleagues tested different types of algae and found a genus, *Schizochytrium*, that maximized the transfer of omega-3s from feed to beef.

Fortuitously, these algae are already produced commercially in large quantities for the harvesting of their omega-3-rich oil.

A serving of Omega3Beef provides about 190 mg total DHA and EPA. This compares to about 7 mg total DHA and EPA in a burger made from grain-fed cattle, and about 14 mg total DHA and EPA in a burger from grass-fed cattle. In a flavor-rich food like beef, these amounts do not impart a fishy odor or aftertaste, says Smith. Because of the cost of the ultra-pure algae feed supplement, Omega3Beef is about \$0.35 per pound more costly to produce than conventional beef. According to the company's website, researchers plan to do large-scale tests with more cattle before seeking the approval of the Center for Veterinary Medicine of the US Food and Drug Administration (FDA) for feeding the *Schizochytrium* algae to beef cattle. Smith says that these feeding trials will begin soon, with results expected later this year. But European consumers may have the first opportunity to purchase Omega3Beef. Smith notes that *Schizochytrium* algae have already been approved as a safe feed ingredient for animals in Europe. He estimates that Omega3Beef will be commercially available in Europe by the end of 2017.

In 2015, a team of scientists led by Gong Cheng at Northwest A&F University, in Yangling, China, took a transgenic approach to producing omega-3-enriched beef (Cheng, G., *et al.*, <http://dx.doi.org/10.1007/s10529-015-1827-z>). The researchers introduced a gene from nematodes (*Caenorhabditis elegans*) into cattle fetal cells using somatic cell nuclear transfer. The gene, called *fat1*, encodes a desaturase enzyme that converts omega-6 fatty acids to omega-3s, including ALA, DHA, and EPA.

## Soap Manufacturing Technology, Second Edition

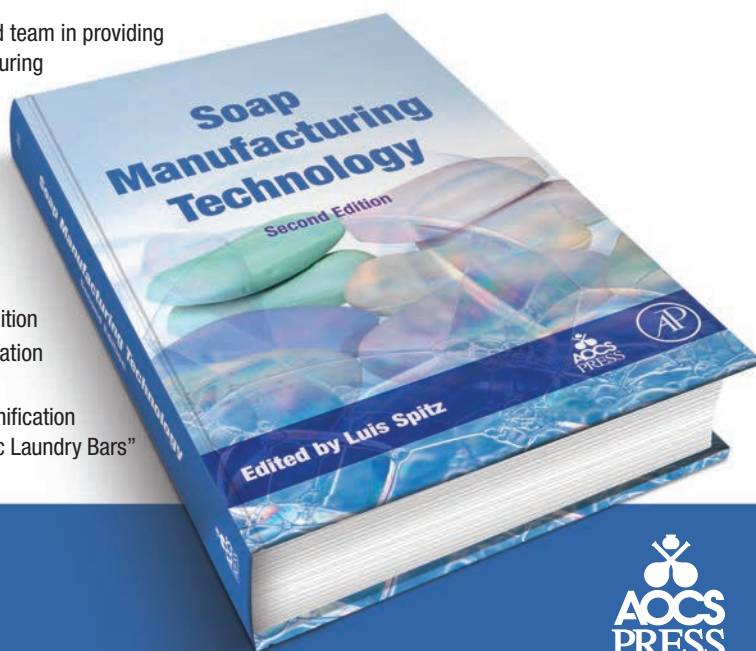
Edited by Luis Spitz

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The transgenic cattle produced beef with about 5 times the normal level of omega-3 fatty acids.

Unfortunately, 11 of the 14 cattle died before the age of 4 months from inflammation and hemorrhagic septicemia, a bacterial disease of cattle. According to the researchers, the abnormalities were probably due to imperfections in the procedure for somatic cell nuclear transfer. The transferred somatic cell nucleus may not have been completely reprogrammed by the egg cell cytoplasm, and genes may have been inadvertently turned on or off during the procedure. "There is much to learn about the best scientific techniques and the best husbandry required to make beef a rich animal source of omega-3 oils for human nutrition, but we have taken the first step," Cheng told Food Navigator (<http://tinyurl.com/FN-omega3-beef>).

If researchers could establish a breed of cattle that produces omega-3-rich beef, this could be a more direct and, over the long term, cost-effective approach than feeding strategies. However, Drouillard notes that consumer acceptance may present a problem. "The people to whom omega-3-enriched beef would appeal—in other words, the health-conscious consumer, is not somebody that's generally going to be really open to the idea of anything that's genetically modified," says Drouillard. "I'm not convinced we could make the same progress there as we could through feeding, and we wouldn't have the same kind of resistance with the feeding."

Although each approach to omega-3 beef has unique advantages and disadvantages, none can currently compete with the high levels of omega-3s found in oily fish such as salmon. However, Smith believes that as more foods such as milk, eggs, and beef are fortified with omega-3s, the cumulative level a person consumes throughout the day will approach clinically meaningful values. "Harvard Medical School determined a couple years back that if a healthy older person ingested a total of 400 milligrams of DHA and EPA from fish, it extended their life expectancy 2.2 years due to reduced cardiovascular disease," he says (Mozaffarian, D., *et al.*, <http://dx.doi.org/10.7326/0003-4819-158-7-201304020-00003>, 2013).

In contrast, the American Heart Association recently issued a scientific advisory report concluding that omega-3 fish oil supplements likely do not prevent cardiovascular disease in healthy adults, although they may reduce the risk of death from cardiovascular disease in those who have already experienced a heart attack or heart failure (Siscovick, D., *et al.*, <https://doi.org/10.1161/CIR.0000000000000482>, 2017). "There are two things that may be going on here," says Smith. "Number one, people who eat fish may have healthier diets in general. And number two, there are a dozen key ingredients in fish other than omega-3s, but when people purify fish oil, they usually remove these ingredients." Smith notes that Omega3Beef has the same source of omega-3s and other ingredients that fish do: algae. "We traced these other beneficial fatty acids, like palmitoleic acid, right through into our beef to make sure that they were there," he says.

Only time will tell if omega-3-fortified beef is a hit with consumers. According to Drouillard, because of regulatory hurdles in the United States and elsewhere, the biggest challenge

lies in even making consumers aware of omega-3-fortified beef. "The USDA [US Department of Agriculture] prohibits companies from putting anything on beef labels that identifies them as having higher amounts of omega-3 fatty acids," he says. "So we have a major regulatory limitation today on labeling that puts these beef companies at a huge disadvantage." Drouillard notes that fish, because its labeling is regulated by the FDA instead of the USDA, is not subject to the same omega-3 labeling limitations. "On beef, you can have a line on the label that says it has x grams of omega-3 content, but there's no way to alert the consumer that the product was intentionally modified to be more healthful or contain more omega-3s," says Drouillard. "In my opinion, that's been the largest impediment to progress."

*Olio is produced by Inform's associate editor, Laura Cassiday. She can be contacted at [laura.cassiday@aocs.org](mailto:laura.cassiday@aocs.org).*

## Information

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# New chemical review process slows, while states remain active

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

**Kelly Franklin**

The US Environmental Protection Agency's (EPA's) apparent policy to stop using "non-5(e) Snurs" under the new Toxic Substances Control Act (TSCA) is delaying products from coming to market and stifling innovation of safer alternatives, according to some chemical manufacturers and industry consultants.

Under the reformed TSCA, the new chemicals review process has slowed, and the percentage of substances subject to consent orders has risen sharply. With these changes, it appears that the EPA has also discontinued its use of so-called non-5(e) Snurs to manage potentially concerning uses outside of those enumerated in a pre-manufacture notice (PMN).

But the agency's website indicates that "a small subset of PMNs that previously would have been "dropped" from review and for which a non-section 5(e) Snur would have been issued ... will now be subject to [5(e)] orders."

## LIFE HAS CHANGED

At an American Chemistry Council GlobalChem conference held earlier in the year, Marcia Levinson, regulatory affairs specialist at chemicals firm Covestro, said that "life has changed for industry" as far as new chemicals are concerned.

The advent of the EPA no longer using non-5(e) Snurs means that now new substance manufacturers must frequently enter into a consent order for "something that they did not even foresee in their PMN application," she said.

And while the order is being written, the manufacturer must wait beyond the 90-day PMN review period before their new substance can go to market—a process she says takes a minimum of 60 days. The resulting consent order may also contain testing requirements for foreseeable uses.

"This definitely has an impact on our innovative spirit," she said.

Further, she said that it is more challenging to sell a regulated product than a non-regulated one.



"The perception is that a regulated chemical is much more hazardous than a non-regulated chemical," said Levinson. "Therefore, we've got a discrepancy in terms of the perception of the impact of this regulation."

Even if companies are able to work with customers to explain that this is simply the new way of doing business, there is still the added burden of managing a regulated chemical, such as recordkeeping requirements, export notifications, lower chemical data reporting (CDR) rule reporting thresholds, or changes to existing hazcom practices.

For these reasons, she said, "it's hard to get customers to go toward these new chemicals."

Lisa Marie Nespoli, also of Covestro, agreed: "We're creating safer models that are more sustainable, and nobody wants to buy them because they have the recordkeeping [requirements]."

"That is our biggest concern for our innovation pipeline—we are creating things that are better, this is what everybody wants—but yet we can't get them through, and I don't know how we fix that."

## US STATE LEGISLATIVE ACTIVITY REMAINS UNRESTRAINED

Meanwhile, state legislative activity on chemicals remains as high as ever despite the passage of the Lautenberg Chemical Safety Act, according to industry groups.

Many in industry had looked to the Toxic Substances Control Act (TSCA) reform to stem the growth of state-level legislation on chemicals in products, but state activity appears



not to have been diminished by increased federal oversight of chemicals.

Perennial legislation on flame retardants, ingredient disclosure (particularly for cleaning products), and establishing lists of reportable chemicals of concern remain in play this year across the country. But new issues—and states that do not frequently consider chemicals policies—are also coming to the fore.

Mississippi and West Virginia, for instance, have introduced measures to ban certain flame retardants in children's products and upholstered furniture. And Iowa has proposed a bill to ban triclosan in cleaning products.

Meanwhile, in Hawaii the legislature has proposed more than 10 separate bills to address oxybenzone, a common sunscreen ingredient linked to concerns for coral organisms. There has also been an uptick in legislation focused on fluorochemistries, particularly in food packaging.

## EXPANDING POLICIES

Other states are seeking to expand on existing chemicals policies.

In Vermont, a measure was introduced in the House and Senate (HB 268/S103) that comprises the “wish list of every nonprofit that wants to regulate the chemicals industry,” according to the Toy Industry Association's director of state affairs, Owen Caine.

At around 80 pages in length, the measure covers “every aspect of the economy,” Caine said. Among a number of hazardous waste requirements, it would also “dramatically expand” Vermont's children's product reporting requirements to cover all consumer products, with some exceptions.

The measure also seeks to:

- require the removal of reportable substances from children's products within six years of a manufacturer's first report to the state;
- ban the manufacture or sale of dental floss or food contact materials containing perfluoroalkyl substances (PFAS); and
- establish an interagency committee to identify substances of concern and propose measures that address risks they may pose, in consultation with a citizen advisory panel.

“There is literally no part of the economy or supply chain that isn't affected by this legislation—it is incredibly damaging and poses huge regulatory burdens on anyone who does business in Vermont,” said Caine.

Meanwhile, he pointed out that the New York Department of Environmental Conservation announced it will require cleaning product ingredient disclosure under a decades-old law, and has labelled these new guidelines as a test case for other kinds of products.

“This is an example of ... ignoring the legislature,” he said. With bills slow to pass, it appears New York has moved to a regulatory authority that “loosely has the authority” to act.

Now that TSCA has been reformed, said Caine, stakeholders are looking for “other ways to get at the information—and ultimately restrictions”—on chemicals.

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*Kelly Franklin is Editor, North America, for Chemical Watch.*

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## Changes to consent orders, Snurs under new TSCA

TSCA section 5(e) orders—including consent orders—are only binding to the original pre-manufacture notice (PMN) submitter. As a consequence, the EPA typically promulgates a significant new use rule (Snur) that mimics the 5(e) order so that all subsequent manufacturers and processors of the substance will be bound to the same requirements.

The agency has continued the practice of these so-called 5(e) Snurs under the new law.

This does not appear to be the case, however, for non-5(e) Snurs, or Snurs issued in the absence of a consent order. Before TSCA reform, the EPA would often use these when it determined that the specific conditions of use and precautions described in a PMN would not pose an unreasonable risk, but for which additional uses may be concerning.

In these cases, the agency would “drop” the PMN—that is, allow the substance to come to market—and subsequently issue a non-5(e) Snur that defined additional uses or conditions that may be problematic. Any future engagement in such activities by a manufacturer or processor would thereby require notifying the agency before doing so.

But under the new TSCA, the agency may no longer drop a PMN; it must instead make an affirmative finding that the substance is not likely to present an unreasonable risk. And in order to meet this bar, it has apparently interpreted the statute as requiring it to impose a consent order on the original submitter to block potentially foreseen uses, even if they are not named in the PMN. The use of non-5(e) Snurs has correspondingly dropped off.

This change has resulted in real consequences for the PMN submitter. Not only is that company likely to experience delays in coming to market while a consent order is negotiated, but it also may face additional testing requirements for uses it has not indicated an intent to pursue.

Previously, this testing burden had fallen to those manufacturers or processors submitting significant new use notifications (Snuns) for any potentially concerning uses identified in the Snur.

# Argentina: process simulations for process optimization

Leslie Kleiner


During product development, R&D scale-ups, plant trials, and plant runs, food scientists and engineers could benefit from methodologies that help them predict outcomes without performing numerous experimental runs. To understand how process simulation can reduce the number of test runs, and how process simulation is implemented in Mercosur and other regions, I interviewed Marcelo Usseglio, process simulation manager and founder of ProSimTechs, San Fernando, Buenos Aires, Argentina.

**Q: What are process simulations for industrial processes, and how are they performed?**

Process optimization tasks refer to selecting/implementing the best process conditions to improve oil yields, minimize energy costs, reduce wastewater, maximize the quality of co-products, and ultimately, to improve profit margins. There are different methods to perform such optimization tasks.

Usually, industrial tests are performed in the plant, using historical information based on process conditions and lab results. Typically, only one process variable is changed at a time, all the results are recorded, and then the impact on the process parameters is analyzed. There is nothing wrong with this approach, but it involves a relative high cost, time, uncertainty of results and a certain risk to the operation, and it clearly limits the scope and scale of the optimization.

Process modeling and simulation techniques make it possible to take a more systemic approach to these optimization tasks. An advanced process simulation model integrates all processing details, such as particular compounds in the vegetable oil inlet, processing variables, key equipment characteristics, and other parameters. Using this model, an unlimited number of process simulation runs can be performed, making it possible to consider all the process variables— even different qualities of inlet streams— to identify optimal process conditions. Once optimized conditions have been met, then industrial test can be performed, reducing the time, cost, and degree of uncertainty of industrial plant tests. Therefore, a process simulation study not only can be performed to optimize particular tasks, but to acquire in-depth knowledge of



*Latin America Update is a regular Inform column that features information about fats, oils, and related materials in that region.*

an entire process. It would be impossible to acquire the same knowledge level using industrial trials alone.

**Q: Can partial processes within a larger process be optimized (i.e., deodorization or tocopherol recovery)?**

Any of the process units within the refining process can be optimized through process simulation; one of the key applications of process modeling and simulation in vegetable refining is deodorization optimization.

Deodorization is a steam stripping process in which different and complex phenomena occur (mass transfer, liquid-vapor phase equilibrium, side reactions and hydraulic effects, as well as the chemistry of a complex mix of lipids). A process simulation model can take all these phenomena into consideration, making it possible to predict changes in the composition of outlet streams (deodorized oil and distillates) under different process conditions, or with composition changes in bleached oil, with a high degree of certainty.

For instance, the procedure typically used to study the possibility of reducing the stripping steam consumed in the deodorizer is to reduce the total stripping steam flowrate injected into the column. However, in some cases it is better to understand the effect of closing the injection of stripping steam in the last stage (last tray). For this analysis, you must know the free fatty acid (FFA) concentration in the oil at each stage. Although this thought process is not feasible in a real deodorizer, a simulation model can do these studies quickly due to its stage-to-stage column design.

The process simulation of tocopherol concentration is another clear application of processing optimization. For example, in a process performed in a double scrubber, the tocopherol concentration and drawn mass are sensitive to the condensing temperature of the so-called "hot scrubber." However, due to the nature of phase equilibrium of the compounds present in the distillate, there is a peak in the tocopherol concentration curve related to this temperature. This peak is not easy to find during industrial tests, but it is easily detected with process simulation. Locating this peak and matching it with the amount of drawn distillate, allows us to find the process point that maximizes concentration and therefore the revenue of tocopherol distillate.

**Q: How has process simulation been implemented in the Mercosur region?**

Within Mercosur, and in fact in all of South America, soft oils (soybean, canola, sunflower), tallow, and tropical oils (palm oil and its fractions) are used for specific final applications. This makes it necessary to obtain the same final product from variable stock, sometimes with changes in processing conditions. Processors in Mercosur using process simulation now have a great tool to anticipate processing scenarios using variable stock.

**Q: Can process simulation increase safety at the plant?**

The application of process simulation to process optimization is more straightforward than it is for industrial safety. Also, there is software in the market that is more specific for industrial safety analysis, for instance for relief systems, thermal stress analysis, and so on.

**Q: On a practical level, how do simulation models for vegetable oil refining work?**

Using a rigorous and complex simulation platform embedded in MS Excel, but at the same time a simple to use tool, you can run any kind of processing scenario to assess a new scenario or to diagnose a current one. The key is to build the model and to validate it against plant conditions and lab data, so you will have a high degree of certainty of results. These are the steps we follow when we build a given process simulation model. The process engineer can then apply it to any kind of processing scenario and select which one should be tested at the industrial scale.

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



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# PATENTS

## Acetylated polyglycerine fatty acid ester and a PVC insulator plasticised therewith

Chaudhary, B.I., *et al.*, Dow Global Technologies LLC, US9481633, November 1, 2016

The present disclosure is directed to acetylated polyglyceride fatty acid ester and compositions containing the same. The acetylated polyglyceride fatty acid ester may be blended with an epoxidized fatty acid ester. The present acetylated polyglyceride fatty acid ester and blends find advantageous application as a plasticizer.

## Biodegradable putty compositions and implant devices, methods, and kits relating to the same

Kunjachan, V., Globus Medical, Inc., US9498558, November 22, 2016

Disclosed are phospholipid based compositions and implant devices, as well as methods and kits that include such compositions or components thereof. In particular, the present compositions include a polymer component such as a poloxamer or PEG component and a phospholipid component, such as a Phosal. The present compositions may include at least one additional component, such as granules, powder, and/or particulates. The present compositions may further include one or more bone graft materials and/or active ingredients. The compositions may be used on their own or incorporated on or in a surgical implant.

## Epoxidized fatty acid alkyl ester plasticizers and methods for making epoxidized fatty acid alkyl ester plasticizers

Ghosh-Dastidar, A., *et al.*, Dow Global Technologies LLC, US9499681, November 22, 2016

Epoxidized fatty acid alkyl esters and methods for making epoxidized fatty acid alkyl esters. Such epoxidized fatty acid alkyl esters can be prepared by epoxidizing fatty acid alkyl esters with an acid and a peroxide. Epoxidation can be performed under controlled reaction conditions to provide epoxidized fatty acid alkyl esters having an iodine value in the range of from 4–15g I<sub>2</sub>/100g of epoxidized fatty acid alkyl esters. Epoxidized fatty acid alkyl esters can be employed in plasticizer compositions, either alone or in combination with other plasticizers, such as epoxidized natural oils. Such plasticizers in turn may be used in the formation of polymeric compositions.

## Betaine esters and process for making and using

Burk, C.H., *et al.*, Eastman Chemical Co., US9487805, November 8, 2016

A variety of betaine esters, including dialkylaminoalkyl cocoate betaines and dialkylaminoalkyl hydrogenated cocoate betaines, are disclosed. These betaines can be advantageously prepared in high yield and purity by a three-step transesterification chemoenzymatic process or a two-step direct esterification chemoenzymatic process. These betaine esters have excellent surfactant properties.

## Heat-curing reactive systems

Sauer, R., *et al.*, Henkel AG & Co. KGaA, US9493687, November 15, 2016

The present application provides heat-curing preparations containing (a) a triglyceride fraction, the fatty acid distribution of which has a proportion of at least 5 wt%, in particular at least 60 wt%, of one or more  $\Omega$ -3 fatty acids and/or one or more  $\Omega$ -6 fatty acids, (b) at least one vulcanizing agent selected from the group consisting of (b1) sulfur, (b2) peroxidic vulcanizing agents, (b3) quinones and/or quinone dioximes and/or (b4) dinitrosobenzenes, and (c) at least one inorganic filler.

## Soy adhesives and composites made from the adhesives

Li, K., Oregon State University, US9493693, November 15, 2016

A formaldehyde-free aqueous adhesive composition comprising (a) soy protein and (b) magnesium oxide or a mixture of magnesium oxide and magnesium hydroxide, wherein components (a) and (b) together constitute at least 50 wt% of the composition, excluding the weight of the water.

## Fuel composition with enhanced low temperature properties

Burgazli, J., *et al.*, Innospec Ltd., US9493716, November 15, 2016

Disclosed herein is a fuel oil composition comprising a renewable fuel or a blend of petroleum based fuels with renewable fuels, also containing an additives composition to enhance the base and combined fuel's resistance to forming insoluble particulates upon storage at diminished operating temperatures. Further described is the additive package used to inhibit particulate formation.

## Compositions and methods for making and modifying oils

Lam, D., *et al.*, DSM IP Assets B.V., US9499844, November 22, 2016

The invention provides novel methods for making or modifying oils, e.g., plant animal or microbial oils, such as vegetable oils or related compounds, that are low in a particular fatty acid(s), for example, low linoleic oils, linolenic oils, low palmitic oils, low stearic oils or oils low in a combination thereof.

## Filling composition comprising an encapsulated oil

Arfsten, J., *et al.*, Nestec S.A., US9504265, November 29, 2016

The present invention relates to filling compositions comprising an encapsulated oil, and optionally a free liquid oil. The encapsulated oil comprises at least 40% w/w of a liquid oil encapsulated in a matrix material. The invention also relates to processes for the preparation of the filling composition and its use in composite food products.

## Functional food additives

Dutta, P., US9505738, December 6, 2016

Novel compounds, and in particular to diacylglycerol (1, 3-DAG) and alpha-lipoic acid (LA) and/or dihydro-alpha-lipoic acid (DHHA) derivatives. In particular these novel compounds are used as functional food additives. These may for example be used as nutraceuticals and/or pharmaceuticals in the prevention and treatment of obesity, diabetes, atherosclerosis, oxidative stress, and other lifestyle-related diseases.

## Fungicidal compositions

Wurms, K.V., *et al.*, The New Zealand Institute for Plant And Food Research Ltd., US9510600, December 6, 2016

The present invention relates to fungicidal compositions and particularly fungicidal compositions comprising anhydrous milk fat (AMF), soybean oil and/or coconut fat. The invention also relates to a fungicidal method employing a composition of the invention. The compositions and methods of the invention have applications in the management (prevention and control) of fungal growth in commercial and small scale crop production.

## Amino lipids, their synthesis, and uses thereof

Davidson, G., *et al.*, Incella GMBH, US9511024, December 6, 2016

The present invention provides new amino lipids and a convenient method for synthesizing these compounds. These (cationic) amino lipids have good properties as transfection agents. The method is an economic versatile two step synthesis allowing the preparation of various amino lipids thus leading to the assembly of a combinatorial library of transfection agents. Moreover, the present invention provides lipid particles (liposomes) containing said amino lipids and their use for delivering bioactive agents into cells. The invention encompasses also the use of lipid particles containing the cationic amino lipids as medicament.

## Compacted rumen protected nutrient pellets and method of manufacture

Garrett, J.E., *et al.*, Qualitech, Inc., US9511062, December 6, 2016

A process for producing a compacted rumen-protected nutrient pellet and the resultant pellet. The process includes the steps of (i) blending a C4-24 fatty acid and a biologically active ingredient to

form solid central core particles, (ii) compacting the core particles to form pellets, and (iii) sequentially coating the pellets with an organic solvent and a fatty acid alkali metal or alkaline earth metal salt.

## Process of producing purified $\gamma$ - and $\Delta$ -tocotrienols from tocol-rich oils or distillates

Howard, L., Board of Trustees of the University of Arkansas, US9512098, December 6

The invention is generally directed to a process of producing purified  $\gamma$ - and/or  $\Delta$ -tocotrienols from tocol-rich oils or distillates, such as rice bran oil deodorizer distillate or palm oil. The process produces a  $\gamma$ - and/or  $\Delta$ -tocotrienol-rich fraction in a high proportion of  $\gamma$ -T3 and/or  $\Delta$ -T3 while minimizing the presence of alpha isomers from the tocol-rich oil or distillate. The  $\gamma$ -T3- and/or  $\Delta$ -T3-rich fraction comprises about 95% of total tocopherols, with the process yielding  $\gamma$ -T3 in approximately 10% and  $\Delta$ -T3 in about 3%, with each having purity in excess of approximately 95%. The process utilizes flash or other low pressure chromatography to provide rapid isolation of  $\gamma$ - and/or  $\Delta$ -tocotrienol from the tocol-rich oil or distillate.

## Bio-based block polymers derived from lignin and fatty acids

Holmberg, A.L., *et al.*, University of Delaware, US9512249, December 6, 2016

The present invention pertains to bio-based block polymers synthesized from functionalized lignin-based molecules (A-monomer) and functionalized fatty acids or fatty alcohols (B-monomer) derived from plant or animal oils, waxes or fats. The block polymers can be synthesized via numerous polymerization techniques, such as reversible addition-fragmentation chain transfer (RAFT). Most importantly, this class of bio-based block polymers shows promise as providing sustainable yet scalable and tunable thermoplastic elastomers and pressure-sensitive adhesives, among other applications.

## Dispersants having biobased compounds

Baseeth, S.S., *et al.*, Archer Daniels Midland Co., US9517442, December 13, 2016

The present disclosure is directed to compositions having lecithin and an organic acid and related methods. The disclosed compositions may also include one or more co-surfactants such as anionic surfactants and/or non-ionic surfactants, and may be used as a dispersant.

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



# Top-cited 2016 JAOCS papers

## Journal of the American Oil Chemists' Society

1. Mohd Noor, M.A., *et al*, Molecular weight determination of palm olein polyols by gel permeation chromatography using polyether polyols calibration
2. Hazer, B. and E. Akyol, Efficiency of gold nano particles on the autoxidized soybean oil polymer: fractionation and structural analysis
3. Jin, J., *et al*, Characteristics of mango kernel fats extracted from 11 china-specific varieties and their typically fractionated fractions
4. Matos, Â.P., *et al*, Chemical characterization of six microalgae with potential utility for food application
5. Babadi, F.E., *et al*, Sulfonated beet pulp as solid catalyst in one-step esterification of industrial palm fatty acid distillate
6. Wang, X., Z. Han, Y. Chen, Q. Jin, and X Wang, Scalable synthesis of oleoyl ethanolamide by chemical amidation in a mixed solvent



7. Biswas, N., Y.L. Cheow, C.P. Tan, and L.F. Siow, Blending of palm mid-fraction, refined bleached deodorized palm kernel oil or palm stearin for cocoa butter alternative
8. Nielsen, P.M., A. Rancke-Madsen, H.C. Holm, and R. Burton, Production of biodiesel using liquid lipase formulations
9. Jana, S. and S. Martini, Phase behavior of binary blends of four different waxes
10. Del Coco, L., *et al*, Protected designation of origin extra virgin olive oils assessment by nuclear magnetic resonance and multivariate statistical analysis: "Terra di Bari," an Apulian (Southeast Italy) case study
11. Lin, J.-T., C.K. Fagerquist, and G.Q. Chen, Ratios of regioisomers of the molecular species of triacylglycerols in lesquerella (*Physaria fendleri*) oil estimated by mass spectrometry
12. McKeon, T.A., S.A. Patfield, and X. He, Evaluation of castor oil samples for potential toxin contamination
13. Jin, J., *et al*, Production of rice bran oil with light color and high oryzanol content by multi-stage molecular distillation
14. Ifeduba, E.A., S. Martini, and C.C. Akoh, Enzymatic interesterification of high oleic sunflower oil and tripalmitin or tristearin
15. Arslan, F.N. and H. Kara, Fully automated three-dimensional column-switching SPE-FIA-HPLC system for the characterization of lipids by a single injection: Part I. instrumental design and chemometric approach to assess the effect of experimental settings on the response of ELSD

## TD NMR Sample Tubes

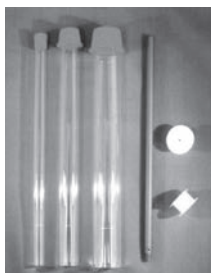
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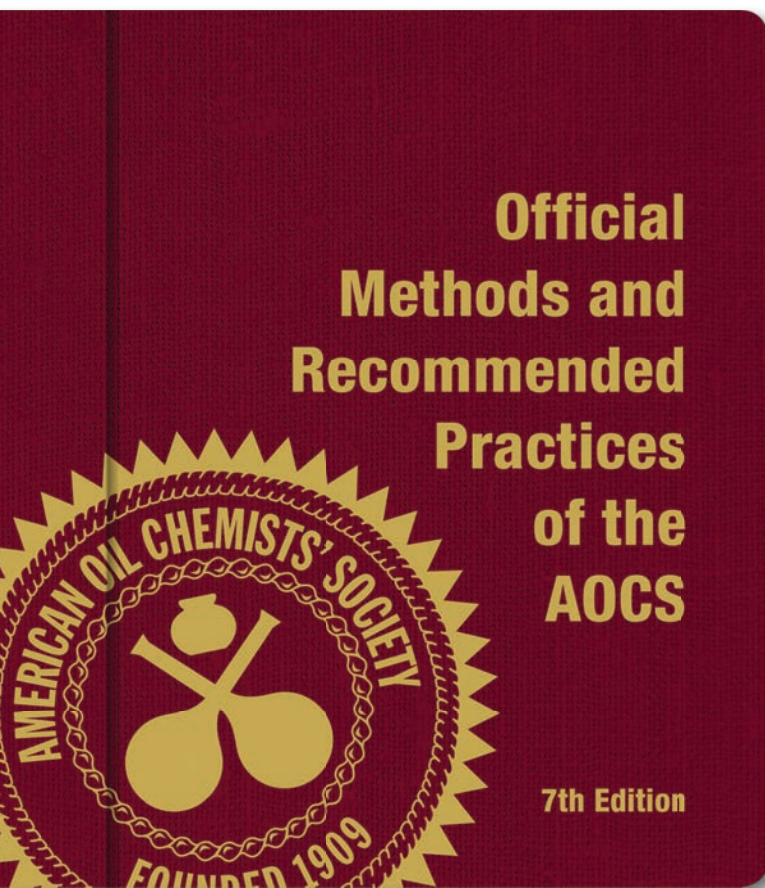
**September 11–14, 2017.** 17th AOCS Latin American Congress and Exhibition on Fats, Oils, and Lipids, Grand Fiesta Americana Coral Beach Hotel, Cancun, Mexico.  
<http://lacongress.aocs.org>

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

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- ▶ Cd 12c-16 (Standard Procedure) Accelerated Oxidation Test for the Determination of Oxidation Stability
- ▶ Cd 30-15 (Official Method) Analysis of 2- and 3-MCPD Fatty Acid Esters and Glycidyl Fatty Acid Esters in Oil-Based Emulsions
- ▶ Ce 12-16 (Official Method) Sterols and Stanols in Foods and Dietary Supplements Containing Added Phytosterols
- ▶ Ce 13-16 (Recommended Practice) Determination of Cyclopropenoic and Nutritional Fatty Acids in Cottonseed and Cottonseed Oil by Gas Chromatography

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# EXTRACTS & DISTILLATES

## Industrial Applications

### Coupling hydrothermal liquefaction and anaerobic digestion for energy valorization from model biomass feedstocks

Posmanik, R., *et al.*, *Bioresour. Technol.* 233: 134–143, 2017, <http://dx.doi.org/10.1016/j.biortech.2017.02.095>.

Hydrothermal liquefaction converts food waste into oil and a carbon-rich hydrothermal aqueous phase. The hydrothermal aqueous phase may be converted to biomethane *via* anaerobic digestion. Here, the feasibility of coupling hydrothermal liquefaction and anaerobic digestion for the conversion of food waste into energy products was examined. A mixture of polysaccharides, proteins, and lipids, representing food waste, underwent hydrothermal processing at temperatures ranging from 200 to 350 °C. The anaerobic biodegradability of the hydrothermal aqueous phase was examined through conducting biochemical methane potential assays. The results demonstrate that the anaerobic biodegradability of the hydrothermal aqueous phase was lower when the temperature of hydrothermal processing increased. The chemical composition of the hydrothermal aqueous phase affected the anaerobic biodegradability. However, no inhibition of biodegradation was observed for most samples. Combining hydrothermal and anaerobic digestion may, therefore, yield a higher energetic return by converting the feedstock into oil and biomethane.

### Optimization of an effective method for the conversion of crude algal lipids into biodiesel

Sha Wu, S., *et al.*, *Fuel* 197: 467–473, 2017, <http://dx.doi.org/10.1016/j.fuel.2017.02.040>.

Although microalgae represent a promising alternative feedstock for biofuels, the current solvent-based lipid conversion systems and processes are neither cost-effective nor energy-efficient due to the bad quality of algal crude lipids. In the present study, a novel lipase-catalyzed transesterification method with crude

*Chlorella* lipids was systematically investigated and optimized. A highly efficient conversion protocol with 97% of fatty acid methyl esters (FAME) yield was obtained after the introduction of co-solvent. Then several crucial parameters including the reaction time, molar ratio of algae oils to methanol, ratio of algae oils to co-solvent, ratio of algae oils to lipase and reaction temperature and intensively studied and selected at 12 h, 1:12, 1:1 (m/v), 20:1 (m/m), and 40 °C, respectively. In addition, the reusability of the immobilized lipase was also investigated with crude algal lipids. The immobilized lipase could maintain 84% of its original activities even after five recycles. Polar lipid standards along with the polar lipids isolated from crude algal lipids were also tested and results indicated that they could also be converted into biodiesel very efficiently. This study provides an important insight into further decreasing the downstream processing costs in algae-based biofuel production.

### A novel integrated biorefinery process for diesel fuel blendstock production using lipids from the methanotroph, *Methylobacterium buryatense*

Dong, T., *et al.*, *Energ. Convers. Manage.* 140: Pages 62–70, 2017, <http://dx.doi.org/10.1016/j.enconman.2017.02.075>.

In light of the availability of low-cost methane (CH<sub>4</sub>) derived from natural gas and biogas along with increasing concerns of the greenhouse gas emissions, the production of alternative liquid biofuels directly from CH<sub>4</sub> is a promising approach to capturing wasted energy. A novel biorefinery concept integrating biological conversion of CH<sub>4</sub> to microbial lipids together with lipid extraction and generation of hydrocarbon fuels is demonstrated in this study for the first time. An aerobic methanotrophic bacterium, *Methylobacterium buryatense* capable of using CH<sub>4</sub> as the sole carbon source was selected on the basis of genetic tractability, cultivation robustness, and ability to accumulate phospholipids in membranes. A maximum fatty acid content of 10% of dry cell weight was obtained in batch cultures grown in a continuous gas sparging fermentation system. Although phospholipids are not typically considered as a good feedstock for upgrading to hydrocarbon fuels, we set out to demonstrate that using a combination of novel lipid extraction methodology with advanced catalyst design could prove the feasibility of this approach. Up to 95% of the total fatty acids from membrane-bound phospholipids were recovered by a two-stage pretreatment method followed by hexane extraction of the aqueous hydrolysate. The upgrading of extracted lipids was then demonstrated in a hydrodeoxygenation process using palladium on silica as a catalyst. Lipid conversion in excess of 99% was achieved, with a full selectivity to hydrocarbons. The final hydrocarbon mixture is dominated by 88% pentadecane (C<sub>15</sub>H<sub>32</sub>) based on decarbonylation/decarboxylation and hydrogenation of C16 fatty acids, indicating that a biological gas-to-liquid fuel (Bio-GTL) process is technically feasible.

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# Lipids from genetically-engineered bacteria

*Lipid Snippets is a regular Inform column that features select content from The AOCS Lipid Library (<http://lipidlibrary.aocs.org/>).*

Annika Röttig and Alexander Steinbüchel

Text excerpted from <http://lipidlibrary.aocs.org/Biochemistry/content.cfm?ItemNumber=41495>; full version includes tables and references.

Remarkable process has been made during the last decade regarding genetic optimization of *E. coli* as well as cyanobacteria to overproduce and secrete free fatty acids (FFAs).

The secretion of fatty acids into the cultivation medium would eliminate the effort of harvesting, drying and solvent-extracting lipid-containing cells and, thus, greatly reduce the overall production costs of bacterial lipids. Although not naturally capable of accumulating lipids, *E. coli* is an attractive production organism as its lipid metabolism has been examined in the most detail amongst prokaryotes and numerous tools have been well established for its genetic modification. Thus, by overexpression of genes involved in fatty acid *de novo* synthesis and/or blocking genes of fatty acid degradation, significant fatty acid overproduction can be achieved. The overexpression of (native or recombinant) thioesterases then releases the acyl residue from acyl binding protein (ACP) and FFA accumulate. Up to 4.8 g FFA/L has already been obtained by engineered *E. coli* strains using glycerol or glucose in fed-batch fermentation, or even 3.8 g/L from woody biomass hydrolysate.

The cyanobacterium, *Synechocystis* sp. SD277, was extensively modified by deleting several genes involved in competing processes (such as the undesirable synthesis of PHB or cyanophycin instead of fatty acids) or to weaken the cell wall integrity to facilitate FFA release into the medium. After overexpressing several thioesterase genes, this engineered strain produced up to 200 mg FFA/L. Although the yield is far below the values obtained with *E. coli*, this process would nevertheless be much more sustainable because CO<sub>2</sub> can serve as the sole carbon source.



## TRIACYLGLYCEROL SYNTHESIS

*R. opacus* can accumulate large amounts of TAG. Thus, most genetic engineering approaches have been aimed at extending its substrate range to enable a more sustainable lipid production from glycerol, (hemi)cellulose-derived sugars or hydrolyzed plant material. By means of targeted modifications and an adaptive evolution strategy, more than 50% (w/w) TAG (corresponding to ca. 16 g/L) was achieved using corn stover hydrolysate as sole carbon source. These yields are significantly above the amounts obtained by wild-type *R. opacus* with different hydrolyzed plant materials (of approx. 28% fatty acids) which clearly underlines the importance of strain optimization for a competitive bacterial lipid production from a challenging feedstock.

A number of studies also focused on establishing TAG accumulation in *E. coli*; for example by combining increased fatty acid and diacylglycerol synthesis with a recombinant expression of essential wax ester synthase/acyl coenzyme A

(acyl-CoA):diacylglycerol acyltransferase (WS/DGAT) genes. However, the achievable yields still need to be significantly improved in order to compete with natural lipid accumulators.

## FATTY ACID ETHYL ESTER SYNTHESIS

The vast majority of biodiesel (fatty acid methyl ester, FAME) is currently produced with methanol derived from fossil sources, although ethanol can alternatively be utilized to gain biodiesel made from fatty acid ethyl esters (FAEE) which can show superior physicochemical properties compared to FAME. In 2006, a strategy was developed to completely synthesize FAEE *de novo* ("microdiesel") from unrelated carbon sources in *E. coli*. In this first approach and follow-up studies, the overexpression of a WS/DGAT gene was usually combined with the ethanol synthesis pathway from *Zymomonas mobilis* (*adhB* encoding alcohol dehydrogenase B, and *pdc* for pyruvate decarboxylase). Similar to strategies employed for FFA overproduction, increasing fatty acid *de novo* synthesis and blocking the beta-oxidation of fatty acids, could further increase FAEE levels in more recent studies.

So far, highest FAEE amounts of 1.5 g/L were only achieved with glucose and in small-scale cultivations. A major disadvantage of *E. coli* is its inability to metabolize lignocellu-

lose, although a first proof-of-principle could be achieved by constructing an optimized strain which utilizes switchgrass hydrolyzate.

Another promising approach is the synthesis of branched-chain fatty acid alkyl esters from (branched) fatty acids and branched short-chain alcohols (e.g. isoamyl alcohol or isobutanol), exploiting the biosynthesis pathway for branched-chain amino acid synthesis. These esters show improved properties at low temperatures, under which conventional biodiesel usually has a rather poor performance. However, the synthesis yield of branched FAEE still needs to be considerably increased. Alternatively, bacteria which naturally incorporate branched fatty acids into TAGs, such as *Streptomyces* isolate G25, could be advantageous for the production of biodiesel with a mixture of straight- and branched-chain fatty acid residues.

Annika Röttig is with the Institut für Molekulare Mikrobiologie und Biotechnologie, Westfälische Wilhelms-Universität Münster, D-48149 Münster, Germany.

Alexander Steinbüchel is faculty in environmental sciences at King Abdulaziz University, Jeddah, Saudi Arabia.

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# Tips from inform|connect

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## HIGHLIGHTS FROM THE TOP 5 DISCUSSIONS IN 2017

### Does anyone know the concentration of citric acid used in the oil industry, and how it is added to the oil?

25–50 ppm of citric acid is added to oil to remove or inactivate metals which could lead to oxidation. The lowest possible amount should be used because excess citric acid raises the acidity of the oil, adding to the apparent amount of free fatty acid present. Citric acid also degrades at high temperatures, so it is usually added to the oil just before it leaves the deodorizer or after deodorization once the oil has cooled.

### What is the maximum storage temperature and time for soybean oil to maintain quality?

Refined soybean oil should be stored at 30°C with nitrogen blanketing. It should be stored in CS/SS storage tanks with positive pressure of N<sub>2</sub> @ 70 mm H<sub>2</sub>O, so that when you pump out the oil, nitrogen will move into the tank. This will prevent an increase in peroxide value, and lead to better stability and control color/flavor reversion.

### How do you calculate the calories per gram in an oil?

Multiply the number of grams of fat by 9. So, 10g of fat/oil x 9 = 90 calories.

### Does anybody have any ideas or suggestions to improve crude oil content analysis on whole canola seed?

Oil extraction efficiency is a function of particle size. AOCS Official Methods define the particle size for these extraction methods. Am 2-93 is the internationally recognized method for exhaustive oil extraction from oilseeds. Other oil extraction methods give lower values, though empirical in nature, these may be better aligned expected values from oilseed processing. Due to the difference in particle size, changing grinders or the type of grinders is likely to cause a difference in results. Although measuring particle size is ideal, a possible alternative would be to devote a single grinder to the oil content test, and ensure that the duration of grinding is consistent.

### What is the importance of Stigmasta-3,5-diene analysis when determining if an extra virgin or pure olive oil is adulterated?

The Stigmastadiene test will tell you if a cold-pressed oil has been adulterated with a refined oil. Stigmasta-3,5-diene is a hydrocarbon derived from sterols during refining (by means of the dehydration of beta-sitosterol). The process is thermally activated, which is why the method explicitly states not to heat the sample solution during testing. Since no heat is to be applied during the pressing of EVOO, little to no stigmastadiene should be present in a virgin olive oil.

It is theoretically possible to remove the sterols of an oil with a similar fatty acid profile—particularly sunflower oil—using bleaching clay during the refining process. This would eliminate the ability to detect adulteration via differences in the sterols profiles. If this were done, the sterols would convert to sterenes during the bleaching process, which *can* be detected because the biggest sterol peak is the betasitosterol, the biggest sterene peak would be the stigmasta-3,5-diene. Consequently, a Stigmasta-3,5-diene analysis is a useful method of detecting adulteration. See <http://lib3.dss.go.th/fulltext/Journal/J.AOCS/J.AOCS/2001/no.3/2001v78n3p305-310.pdf>.

The ratio of the degradation products of sitosterol (stigmastadienes) and campesterol (campestadienes) is also good marker for desterolized sunflower, soybean, palm, or grape seed oil.

## MOST-VIEWED AND DOWNLOADED RESOURCES

- Future Impact of Technological Changes in Oilseed Processing & Oil Refining <https://tinyurl.com/hfpfomp>
- Application of Gas Chromatography Mass Spectrometry (GC MS) in Food Science and Biotechnology <https://tinyurl.com/jjxyxb>
- Natural fats and oils in cosmetics <https://tinyurl.com/ha9eh2a>





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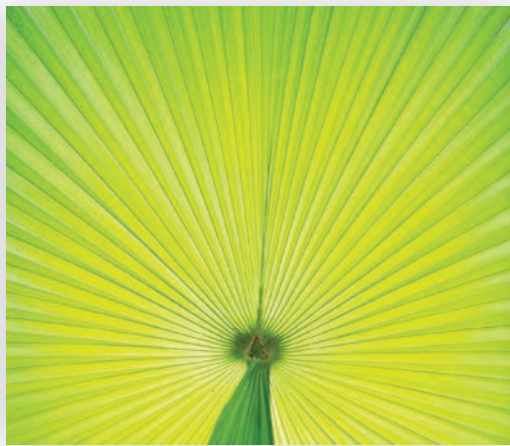
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