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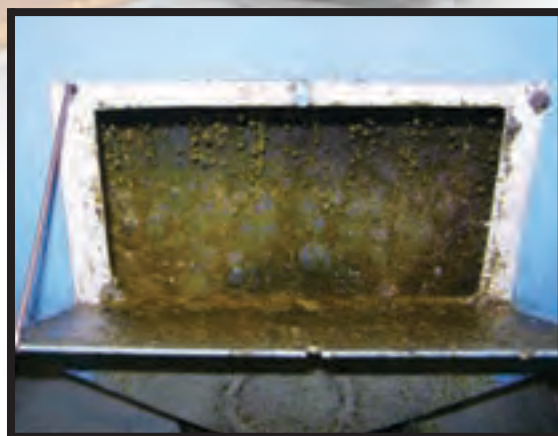
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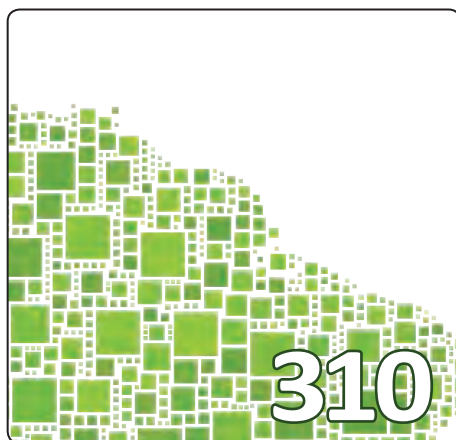
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Erratum

There is an error on page 79 of the February 2015 issue of *Inform*. Figure 1 in the article, Tailoring lipid synthesis in oil crops, by Helen K. Woodfield, John L. Harwood, and Randall J. Weselake, incorrectly lists the percentages each of the world's major oil crops contributes to total oil production. The percentages were correct in the original version submitted by the authors, and the mistake was introduced during editing. The caption should read:

FIG. 1. The world's major oil crops. Clockwise from top left these are oilseed rape (canola), soybean, oil palm, and sunflower, which account for about 12%, 22%, 24%, and 9% of total oil production, respectively.

The editor regrets the error.

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James B.M. Rattray

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Leslie Kleiner
Dave McCall
Robert Moreau

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Ultra oil-producing

Haibo Huang, Stephen Long, and Vijay Singh

The US national objective to achieve national energy independence, together with worldwide efforts to reduce carbon emissions, have spurred the development of biofuel technologies that are based on the use of crops and crop residues as feedstock.

In the United States, ethanol production, primarily from corn, has become a major industry. US ethanol production increased from 6.0 billion liters (1.6 billion gallons) in 2000 to approximately 49.1 billion liters (13.0 billion gallons) in 2014 (RFA, 2015). Cornstarch-based ethanol will no doubt continue to be an important mainstay for US biofuel production. However, its further increase is limited by the revised Renewable Fuel Standard (RFS2) mandate, as well as competition with other uses of corn. The RFS2 required a cap of 56.7 billion liters (15 billion gallons) ethanol production from corn starch. Although much emphasis has been placed on lignocellulosic ethanol, this alternative form of ethanol still faces many technical challenges.

Biodiesel, on the other hand, is easily produced from vegetable oils and requires few inputs. Consequently, it has a very favorable energy output to input ratio. Biodiesel supports an engine performance that is generally comparable to that of conventional diesel fuel, while reducing engine emissions of particulates, hydrocarbons, and carbon monoxide (Haas, *et al.* 2006). Alternatively, hydro-treatment processes allow conversion to jet fuels. With increased demand, US biodiesel production increased substantially from 32 million liters (8.5 million gallons) in 2001 to 5.1 billion liters (1.3 million gallons) in 2013. The major US source of biodiesel has been and continues to be soybean. However, although soybean occupies more land than any other crop except corn, the small amount of oil produced per hectare means that even if the entire US crop were used for biodiesel production it would still fall far short of the RFS2 requirement. Crops that can produce far more oil per hectare of land will be essential to allow further increases in biodiesel production.

PETROSS sugarcane and sweet sorghum

Recently, a group of scientists in the Plants Engineered to Replace Oil with Sugarcane and Sorghum (PETROSS) project began genetically engineering two of the most productive

- This article describes the development of genetically modified sugarcane and sweet sorghum that can accumulate substantial amounts of oil in both stems and leaves.

- Techno-economic models have been developed to simulate oil-producing sugarcane processing on commercial scales that involve using the extracted oil to produce biodiesel and the remaining sugar to produce ethanol.

- Biodiesel production from the oil-producing sugarcane can be more than 10 times higher than that from soybean per hectare of land area.

sugarcane and sweet sorghum

crops in the world—sugarcane and sweet sorghum—into ultra-productive oil crops (PETROSS sugarcane and sweet sorghum). This is an inter-institutional project among the University of Illinois at Urbana-Champaign, the University of Florida, the University of Nebraska-Lincoln and the Brookhaven National Laboratory, and is funded by the Advanced Research Projects Agency (ARPA)—Energy within the US Department of Energy.

There are several important reasons why sugarcane and sweet sorghum were chosen over other crops. First, sugarcane is known to be the most productive crop in terms of converting energy from the sun into chemical energy that is stored in the plant. Sweet sorghum, which can be double- or even triple-cropped in the Southern United States, is also highly productive. Sweet sorghum can also be fed to mills after sugarcane production has ceased, which would keep the mills productive for a longer period of time.

Second, sugarcane and sorghum are less demanding on soil quality and fertilizers, and more drought-tolerant than grain crops. These properties allow sugarcane and sorghum

to be grown in underused and marginal land without competing with food crops.

Third, sugarcane and sweet sorghum are farm and factory ready. The infrastructure for planting, growing, transporting, and crushing the juice—including the oil—is already in place, so only small processing adjustments are needed. Currently, ethanol from sugarcane is the second-largest global source of biofuel after corn ethanol. However, life cycle analysis shows that sugarcane ethanol has a very favorable ratio of carbon output to input compared to other sources of ethanol. Biodiesel from sugarcane would have an even lower carbon footprint than ethanol from sugarcane, since biodiesel production does not require the energy-intensive fermentation and distillation steps of ethanol production.

Research efforts

Produce oil in place of sugar. During photosynthesis, normal sugarcane and sweet sorghum produce sugar that is trans-

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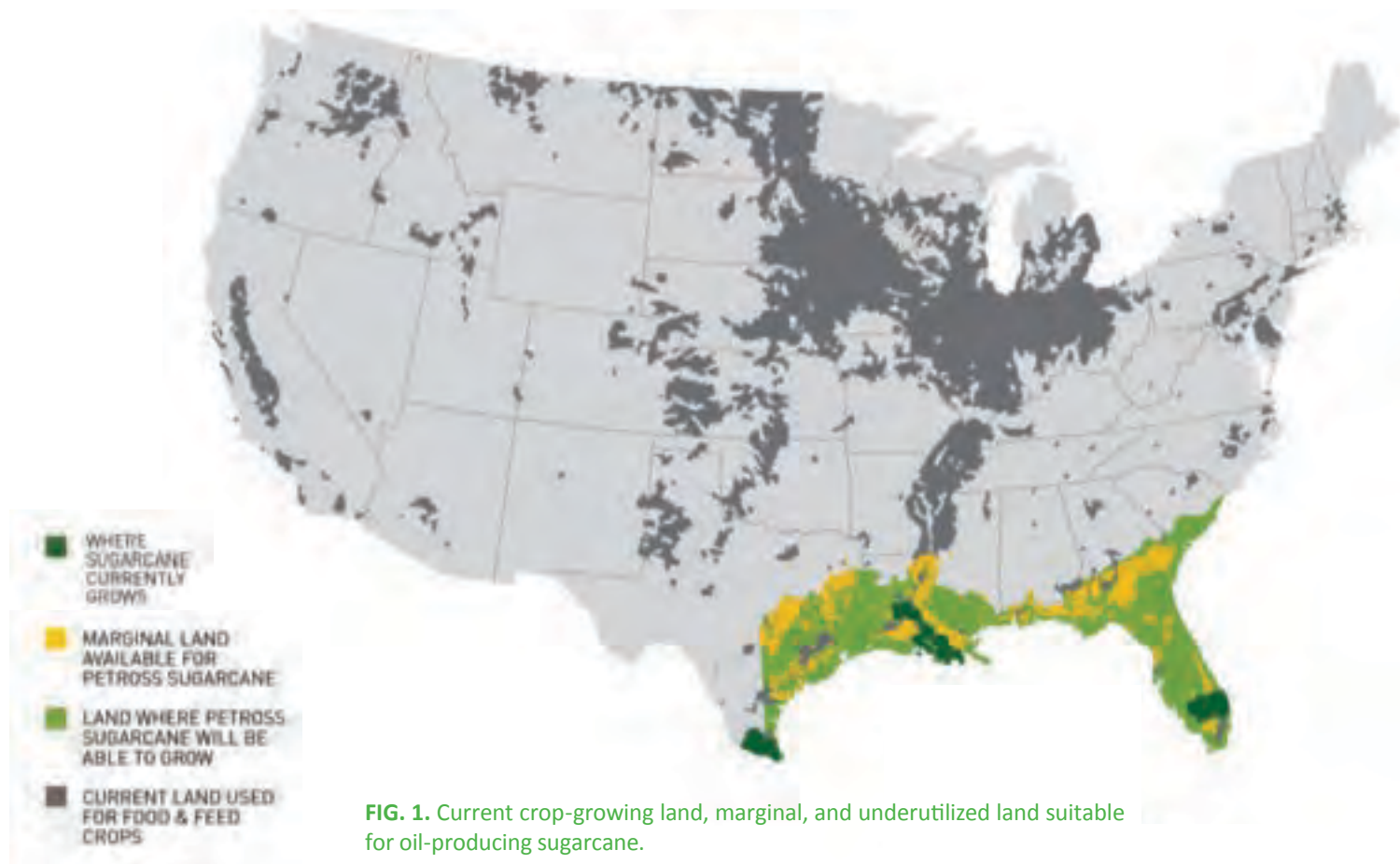


FIG. 1. Current crop-growing land, marginal, and underutilized land suitable for oil-producing sugarcane.

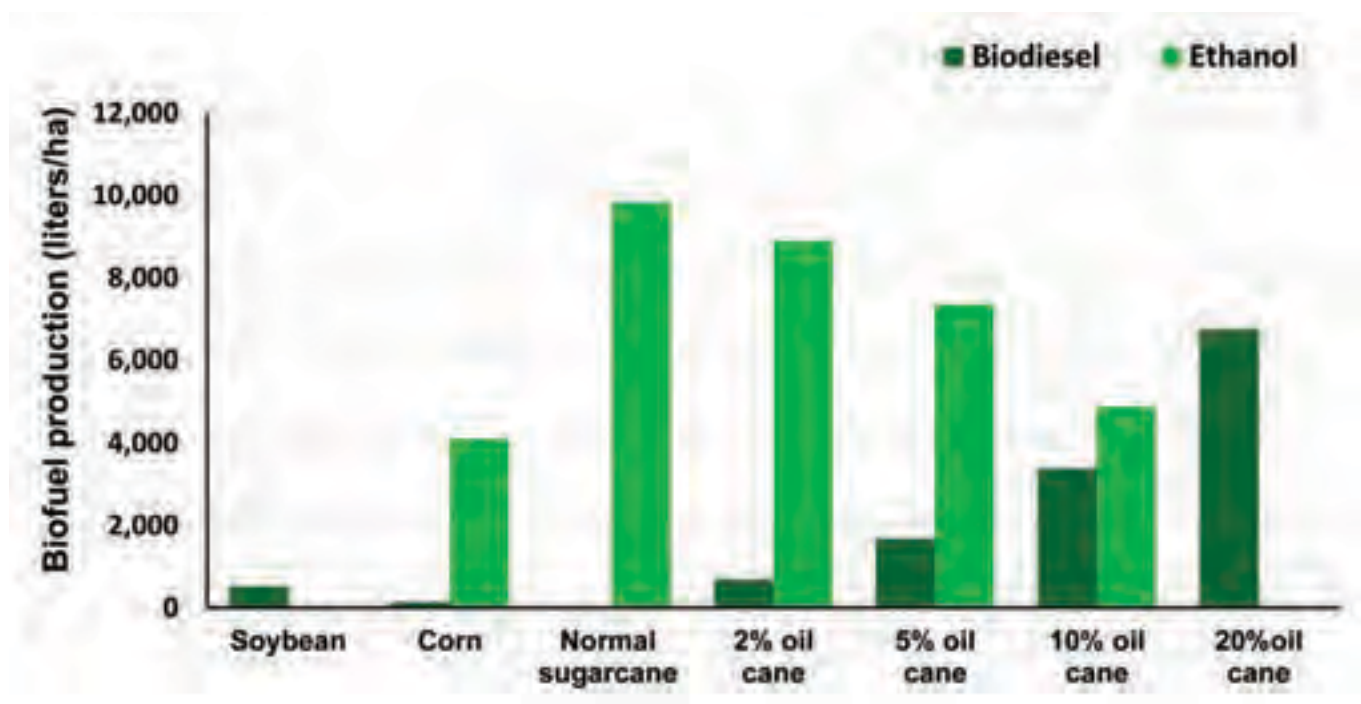


FIG. 2. Biodiesel and ethanol production from each hectare of land planted with soybean, corn, normal sugarcane and PETROSS sugarcane.

ported from the leaves to their stout stems, where it accumulates. This sugar is extracted by crushing and hot washing the stems. The resulting liquor can then be crystalized to produce crystalline sugar or fermented to produce ethanol. Both plants naturally produce triacylglycerides (TAGs) without any assistance from genetic engineering, but this oil is quickly metabolized. To increase TAG accumulation, the PETROSS research group has down-regulated expression of the genes that consume TAGs and up-regulated those that form the TAGs. Progressive additions of genes and gene promoters (pieces of DNA that control expression of genes) have steadily increased oil accumulation in these plants. To date, the group has achieved oil accumulations of up to 5% of dry weight of PETROSS sugarcane—more than 100-fold increase compared to conventional sugarcane. Ultimately, the team expects to achieve an accumulation of 20% oil by dry weight.

Increase photosynthesis. To achieve even more biofuel per unit land area, the PETROSS team has used genetic engineering to improve the photosynthetic efficiency of both PETROSS sugarcane and sweet sorghum. Promising approaches were identified through the development of a digital design platform that optimizes the system *in silico* to identify genes in both metabolism and whole plant design that could increase productivity. By up-regulating or down-regulating these genes, the team has engineered changes that have increased the efficiency with which the leaves use sunlight to assimilate carbon dioxide by 35%. Field trials are underway in Florida, to determine if these changes result in similar yield increases.

Boost cold tolerance. Sugarcane can only be grown in the southern part of the United States. This includes Hawaii, Florida, and the southern edge of the Gulf Coast states. Sweet sorghum also prefers warmer regions, such as Texas and Oklahoma. Genes for cold tolerance are transferred to sugarcane by hybridizing it with miscanthus, a close but cold-tolerant cousin of sugarcane. This makes it possible to grow sugarcane on underused and marginal land in the Southeastern United States (Fig. 1, page 279).

DNA assembly. While improving each individual target (oil production, increased photosynthesis, and cold tolerance) has the potential to make a huge impact, the synergistic benefits of improving them all could make the plants truly revolutionary. Toward this end, DNA-assembler technology is being used to assemble the engineered improvements into a single large piece of DNA that is added to increase oil production, improve photosynthesis, and boost cold tolerance.

Economic potential of oil-producing sugarcane and sweet sorghum

To evaluate the economic potential of oil-producing sugarcane and sweet sorghum, the PETROSS crops were compared with corn and soybean, the two main crops used to produce ethanol and biodiesel in the United States. Oil concentrations in the PETROSS sugarcane were assumed to be 2%, 5%, 10%, and 20%, and techno-economic models were developed to simulate the PETROSS sugarcane processing on commercial scales using SuperPro Designer Software. In the processing models, the extracted oil was

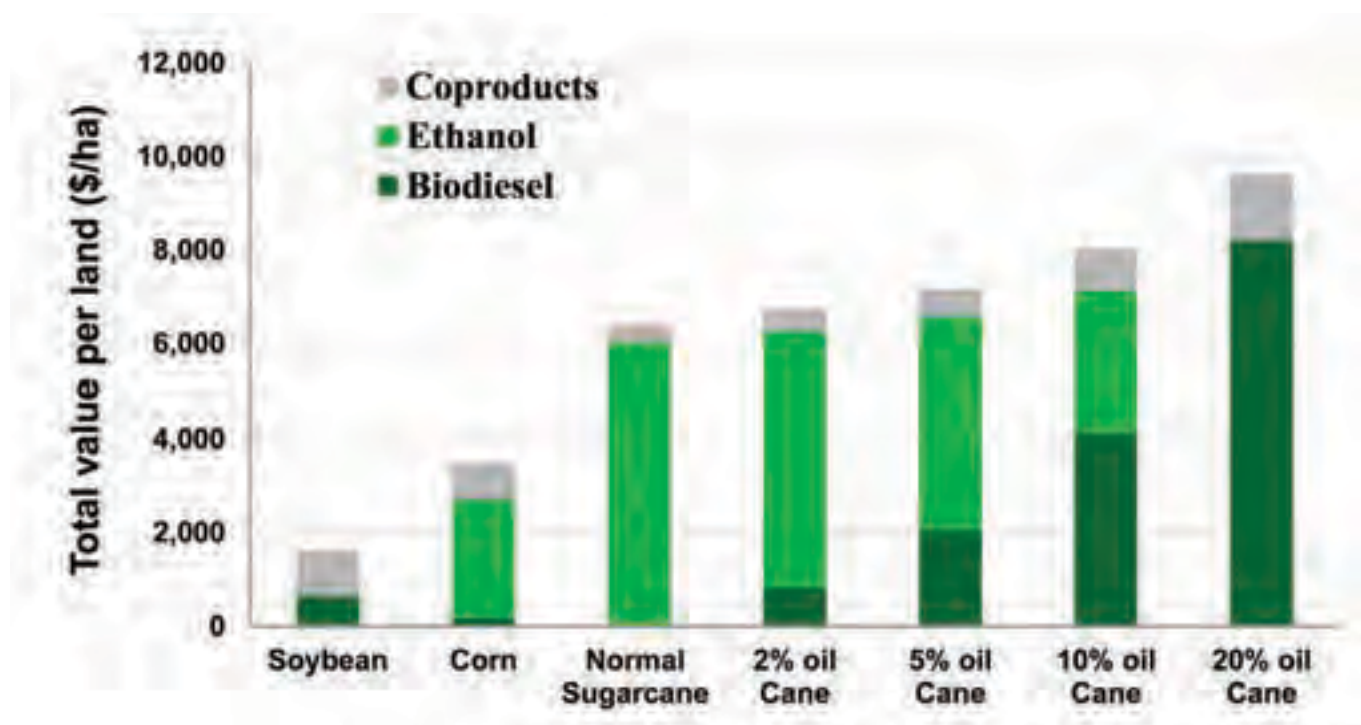


FIG. 3. Total values of products from each hectare of land planted with soybean, corn, normal sugarcane, and oil-producing sugarcane. The numbers were based on the product marketing price in 2013, when ethanol was \$0.61/liter, biodiesel was \$1.19/liter, soy meal was \$480/MT, DDGS was \$260/MT, and electricity was \$0.065/kwh.

used to produce biodiesel by transesterification, and the remaining sugar was used to produce ethanol by fermentation. Bagasse, a byproduct in sugarcane processing, was burned to produce steam to generate electricity for the mills, with the excess sold to the grid.

Usually, one hectare of soybean can provide enough oil to produce about 500 liters of biodiesel. One hectare of corn, on the other hand, can provide enough starch to produce about 4,000 liters of ethanol and an additional 100–150 liters of biodiesel—provided the partial corn oil is extracted at the back-end of the dry-grind fermentation process. The results of our modeling studies showed that even at an oil concentration of 2%, the PETROSS sugarcane produced about 700 liters of biodiesel per hectare of land area— 40% more biodiesel per hectare than a soybean crop (Fig. 2).

Meanwhile, the remaining sugar can be used to produce more than 8,500 liters of ethanol. These potential gains in biodiesel and ethanol production for PETROSS sugarcane are primarily due to its having a higher productivity than either soybean or corn. In the United States, the average yields of soybean and corn are 2.8 and 10.0 MT/ha, respectively (USDA, 2013). The sugarcane dry matter yield in the most productive zone (Hawaii) can be as high as 50–70 MT/ha, while this value is 20–30 MT/ha in the southern states of Florida, Louisiana, and Texas). Increasing the oil concentration in the PETROSS sugarcane caused biodiesel production to increase and ethanol production to decrease. This makes sense, since more sugars were diverted to produce

INFORMATION

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oil. If the oil concentration could be increased to 20%, our calculations indicate that the potential biodiesel production would be about 6,700 liters/ha, or 13 times more than that from soybean.

The total value of all products, including biodiesel, ethanol, and coproducts, from each hectare of land area was also calculated for each crop. We used soybean meal as the coproduct for soybean, distillers dried grain with solubles (DDGS) as the coproduct for corn, and surplus electricity as

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the coproduct for sugarcane processing. Our calculations showed that the total value of all products produced from PETROSS sugarcane was greater than the total value of all products produced from soybean or corn (Fig. 3). This was expected due to higher biofuel productions of PETROSS sugarcane per hectare of land.

Further modeling showed that the total value of the PETROSS sugarcane products increased as oil concentrations increased. Such results indicate that PETROSS sugarcane offers a huge economic benefit and market potential in the United States. Additionally, since the sugarcane harvesting season in the United States is only 3–5 months long, processing PETROSS sorghum in the same mills during the inter-harvest season could even further increase the economic potential of a biorefinery. More information is available at <http://petross.illinois.edu/>.

Vijay Singh is a professor of agricultural and biological engineering at the University of Illinois at Urbana-Champaign, USA. He can be contacted at vsingh@illinois.edu.

The coauthors are also at the University of Illinois at Urbana-Champaign. Haibo Huang is a postdoctoral research associate in agricultural and biological engineering, and Stephen Long is a professor in crop science and plant biology, and a director of the PETROSS project.

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Use of spent bleaching earth for economic and environmental benefit

Norman J. Smallwood

Since the advent of using clay to adsorb color pigments and impurities from edible oil, processors have been confronted with the problem of spent earth disposal. Typically, spent bleaching earth contains entrained oil in the range of 30 to 50% by weight. The entrained oil is in the form of a thin film spread over the immense surface area provided by the clay particles. Upon exposure to air, rapid oxidation of the oil film occurs and sufficient heat is generated to cause spontaneous combustion. Consequently, the most common method of spent earth disposal is to haul and bury it at a solid waste disposal site. Handling and disposing of spent earth is a fire risk, an operating expense, and a source of environmental regulatory concerns. In addition, the value of the oil that is lost is a significant cost.

For more than 100 years, ideas about practical uses for spent bleaching earth have been originated and tried without much success. Integrated oilseed processors and edible oil refiners have had limited success in adding some spent bleaching earth to meal by taking advantage of the allowable use of clay as a flow aid. Due to problems involving safe handling and uniform blending, the addition-to-meal practice is limited. There is some adding of spent bleaching earth to livestock feed in several countries, but the risk of spontaneous combustion is typically a barrier.

- Bleaching earth that has been used to remove the color pigments and impurities from edible oil typically contains entrained oil in the range of 30 to 50% by weight.
- This spent bleaching earth is susceptible to spontaneous combustion; consequently, there are few practical uses for it, and handling and disposing of spent bleaching earth is a fire risk, an operating expense, and a source of environmental regulatory concerns—not to mention a significant cost due to the value of the oil that is lost.
- This article describes how mixing the spent bleaching earth with salt can eliminate the spontaneous combustion problem while providing a high-demand salt-lick product for livestock.



Fig. 1. Experimental work was conducted by the author using spent bleaching earth that came directly from an edible oil processing plant.

CONTINUED ON NEXT PAGE



Fig. 2. The author mixes the products produced for the trial.

A NEW SOLUTION WITH POSITIVE FEEDBACK

In 2011, the author originated the idea that salt could be added and mixed with spent bleaching earth to eliminate the spontaneous combustion problem and provide a high-demand salt-lick product for livestock. A provisional patent was filed to protect the idea and to provide the time for validation. During the summer of 2012, experimental work was conducted to verify that the hygroscopic properties of salt, together with the dilution effect, would eliminate the spontaneous combustion problem with spent bleaching earth. The experimental work was carried out over a week using spent bleaching earth that came directly from an edible oil processing plant (Fig. 1, page 283). It was found that salt content as low as 35% would eliminate

spontaneous combustion. To provide an additional safety factor, 45% salt addition was established as the minimum.

After the experimental work was completed, filing was initiated for a regular patent under the protocol of the international patent agreement. Following successful review of the patent application by the international board, applications have subsequently been filed in most of the livestock-producing countries of the world.

In June 2014, salt-lick products in block and granular form were produced for trial by five cattle producers in the US States of Colorado, Missouri, and Louisiana (Figs. 2 and 3). The feedback from these trials was universally positive. The inventor had no doubt about the expected outcome given his experience:

- as the son of a Colorado cattle rancher who used salt-lick products for cattle;
- working for 50 years in all aspects of the edible oil processing industry;
- monitoring the attraction of cattle to lick spent bleaching earth at a pasture site used for spent bleaching earth disposal. (While anecdotal, 38 years of observation revealed that the cattle involved thrived in terms of health, weight gain, and glistening coats.); and
- interacting in recent years with livestock nutritionists focused on the significance of metabolizable energy.

PRODUCT FORMULATION, FORM, BENEFITS, AND MARKET

The formulation of salt-lick products that incorporates spent bleaching earth has a broad range of possibilities. An array of minerals can be added on the basis of consumer preference.

Some amount of protein meal and/or other feed ingredients can be added if that is the consumer preference.

For livestock, salt is supplied in solid blocks, pellets, and fine particles. From the perspective of supply, it would be desirable to supply the spent bleaching earth and salt mixture for livestock-lick products in all three forms. A literature search was made to determine what bonding agents have been commonly used to make solid blocks for livestock feed. It was found that Type I/II Portland cement has been used for that purpose in making a variety of livestock feed blocks in several countries. Given that Type I/II Portland cement contains about 75% calcium minerals, it is beneficial for livestock ingestion. For the solid block and pellet product, Portland cement was added at levels of 5, 10, and 15%. For the fine particle or granular product, a mixture of spent bleaching earth and salt is



Fig. 3. The trial product was formulated to meet each user's specifications with respect to the inclusion of minerals and/or protein meal.

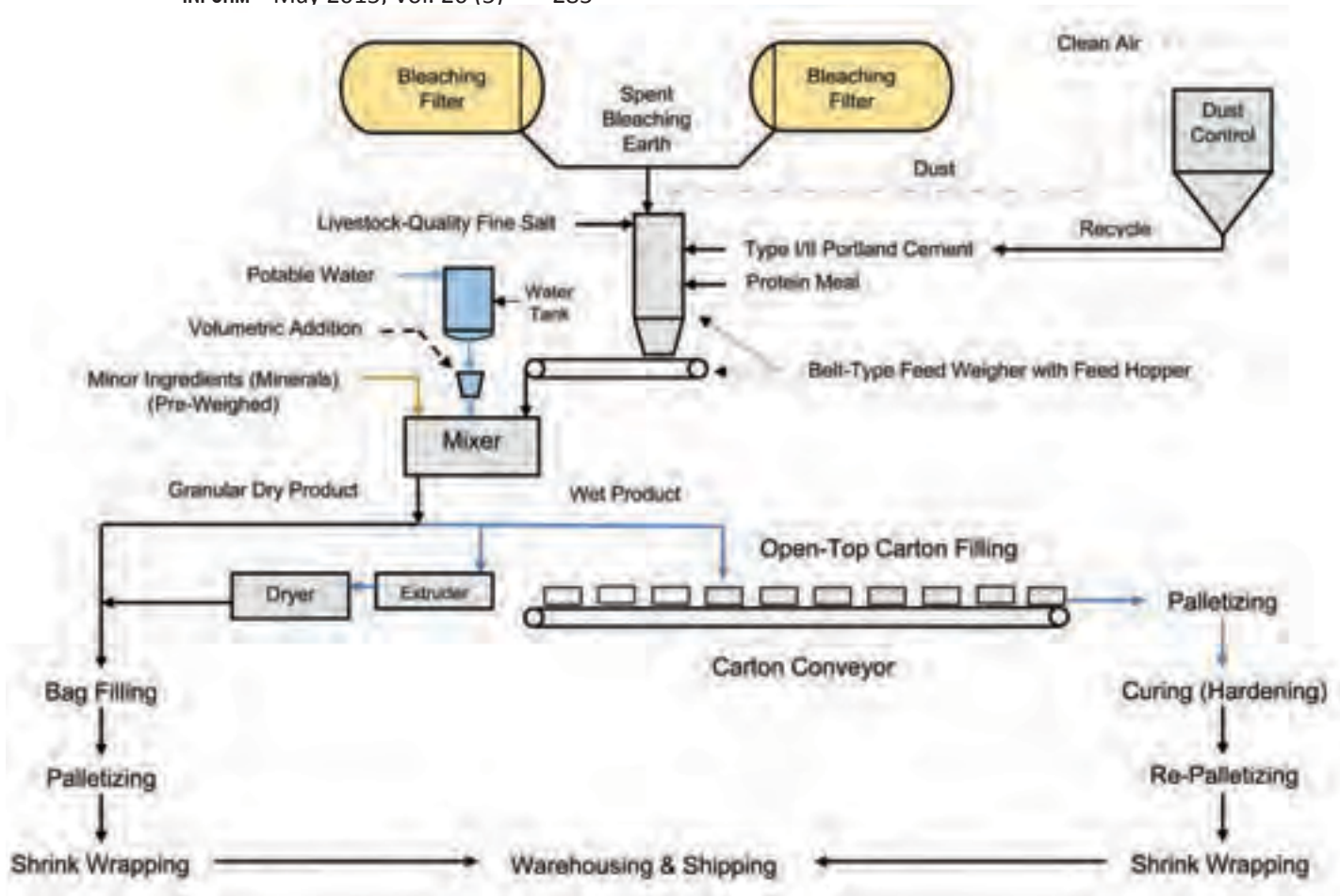


Fig. 4. Production of salt-lick products using spent bleaching earth

acceptable. However, adding 5% Portland cement as a desiccant and mineral agent prevents lumping during long-term storage.

Grass-fed (late spring, summer, and early fall periods) and hay-fed (late fall, winter, and early spring periods) livestock typically receive low levels of metabolizable energy (fat) in their diet. As a consequence, animals have to metabolize some muscle mass to provide sufficient energy to survive. Thus, during the grass- and hay-feeding periods, there is no significant weight gain. By providing salt-lick products that contain metabolizable energy, less to no muscle mass will be metabolized for energy, and the growth rate for livestock will be improved.

While there is no nutritional or mineral benefit, the adsorptive property of the clay does have value. Livestock feeding on grass and hay have the possibility of ingesting some heavy metals. The clay adsorbs these heavy metals and is discharged as waste.

Considering cattle alone, if all of the spent bleaching produced per year in the United States was used to make salt-lick products, it would satisfy only about 10% of the demand for salt based on 20 pounds of salt per animal. Taking into account the salt needs of cattle, sheep, goats, hogs, and horses worldwide, the available market overwhelms the potential supply.

ECONOMIC ADVANTAGES

On a global basis, the conservative numbers are:

- annual global consumption of vegetable oil: 220 billion pounds,

- average bleaching earth usage: 0.3%,
- annual use of bleaching earth: 660 million pounds,
- average retention of vegetable oil in spent bleaching earth: 40%,
- annual generation of spent bleaching earth: 1.1 billion, and
- value of the vegetable oil in spent bleaching earth discounted to yellow grease price: (1.1 billion pounds) (0.4 retention) (\$0.30/pound) = \$132 million/year.

For the United States, the conservative numbers are:

- annual U.S. consumption of vegetable oil: 17.8 billion pounds,
- average bleaching earth usage: 0.4%,
- annual use of bleaching earth: 71.2 million pounds,
- average retention of vegetable oil in spent bleaching earth: 40%,
- annual generation of spent bleaching earth: 118.7 million pounds, and
- value of the vegetable oil in spent bleaching earth discounted to yellow grease price: (118.7 million pounds) (0.4 retention) (\$0.30/pound) = \$14.2 million/year.

These numbers are deliberately conservative.

On the basis of producing product in 50-pound units that include 35% spent bleaching earth by weight, 35% of the

CONTINUED ON NEXT PAGE

product can be produced at no cost to an edible oil processor. If the spent bleaching earth is 40% edible oil by weight, 7.0 pounds of edible oil is contained in each unit. Using a five-year average price for yellow grease (fat applicable to animal feed) of \$0.30 per pound, the value of the edible oil in each 50-pound unit is \$2.10. If the all the other ingredients were priced at purchase cost plus the cost of each unit production, the value (profit potential) of each pound of spent bleaching earth would be \$2.10 per 50-pound unit.

Those interested in exploiting this opportunity are advised to make their own calculations based on local conditions.

PRODUCTION SCHEME

The scheme for producing an array of salt-lick products from spent bleaching earth is quite simple and involves standard equipment used in the livestock feed industry. Diagramed in Figure 4, the batch production process involves the following sequence for adding ingredients to the product mixer:

1. spent bleaching earth acquired directly or indirectly from the bleach process filters. (A weigh-belt feeder is used to weigh both the quantity of the bleaching earth and the quantities of subsequent major ingredients as prescribed by the selected formula.)
2. fine stock salt.
3. type I/II Portland cement.
4. other dry ingredients if specified in the formulation. (Livestock minerals should be pre-weighed on a highly accurate scale in view of the relatively small quantity involved and the much higher unit cost.)
5. water, if prescribed.

Selection of the mixer type should be based on achieving product homogeneity and eliminating any ingredient lumps. On the initial production run to make product for livestock

trial, a simple concrete mixer was used with acceptable results. The type, mixing time, size, and material of construction of the mixer should be determined on the basis of economics and the capacity needed.

To make the lick blocks, product is discharged from the mixer into open-top containers (either milk carton-like paper board or plastic) positioned on a conveyor. Product is received and distributed by placing a multiple-container funneling device over the open-container tops before filling them.

To make pellets, the product from the mixer is fed to an extruder. Then, the extruded, pelletized, product is fed through a hot air dryer before bagging.

Dry granular product is fed from the mixer to the bagging equipment.

PRODUCTION EQUIPMENT LOCATION

Maximum use of gravity transport and lowest-cost production can be achieved by including the capability to make salt-lick products in the design and construction of future edible oil processing facilities. At existing edible oil refineries, space is unlikely to be available for incorporating salt-lick product making at the bleaching process location. In this situation, the closest possible production site should be selected to minimize transportation costs and enable use of the existing infrastructure.

Given that using spent bleaching earth to make salt-lick products is a new business venture possibility, production-facility-location decisions can be made based on the lowest total cost. Cost considerations include transportation of ingredients to the production site, economy of scale for production, and transportation of finished products to distribution system locations. For enterprises with multiple edible oil refineries, this method would likely be advantageous. A business enterprise with no edible oil refineries would likely employ a similar approach.

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Because exposure to air increases the risk of spontaneous combustion, US Department of Transportation (DOT) regulations require spent bleaching earth to be hauled within 24 hours after discharge from a bleaching system. However, mixing the spent bleaching earth with the prescribed quantity of fine stock salt at the edible oil refinery site eliminates this risk, and the restriction on haul time is negated.

MARKETING AND SALES

Salt-lick products can be readily incorporated into large, vertically integrated agriproducts enterprises that produce livestock feed products. In this case, production can be launched at one production site and expanded as market demand develops.

Smaller enterprises with only one refinery would need to develop a market. This could be accomplished by negotiating agreements with livestock feed retailers, such as Tractor Supply Company, and promoting the product aggressively with independent livestock feed stores, farmer cooperatives, and cattle-men's associations in the region of the production site.

The internet offers many sales possibilities, including private internet sales sites and online sales enterprises like amazon.com. The sales capabilities of the internet are particularly attractive and profitable for novelty and niche product offerings. Feeding trials conducted to date reflect that salt-lick products formulated with spent bleaching earth attract deer, elk, squirrels, and chipmunks.

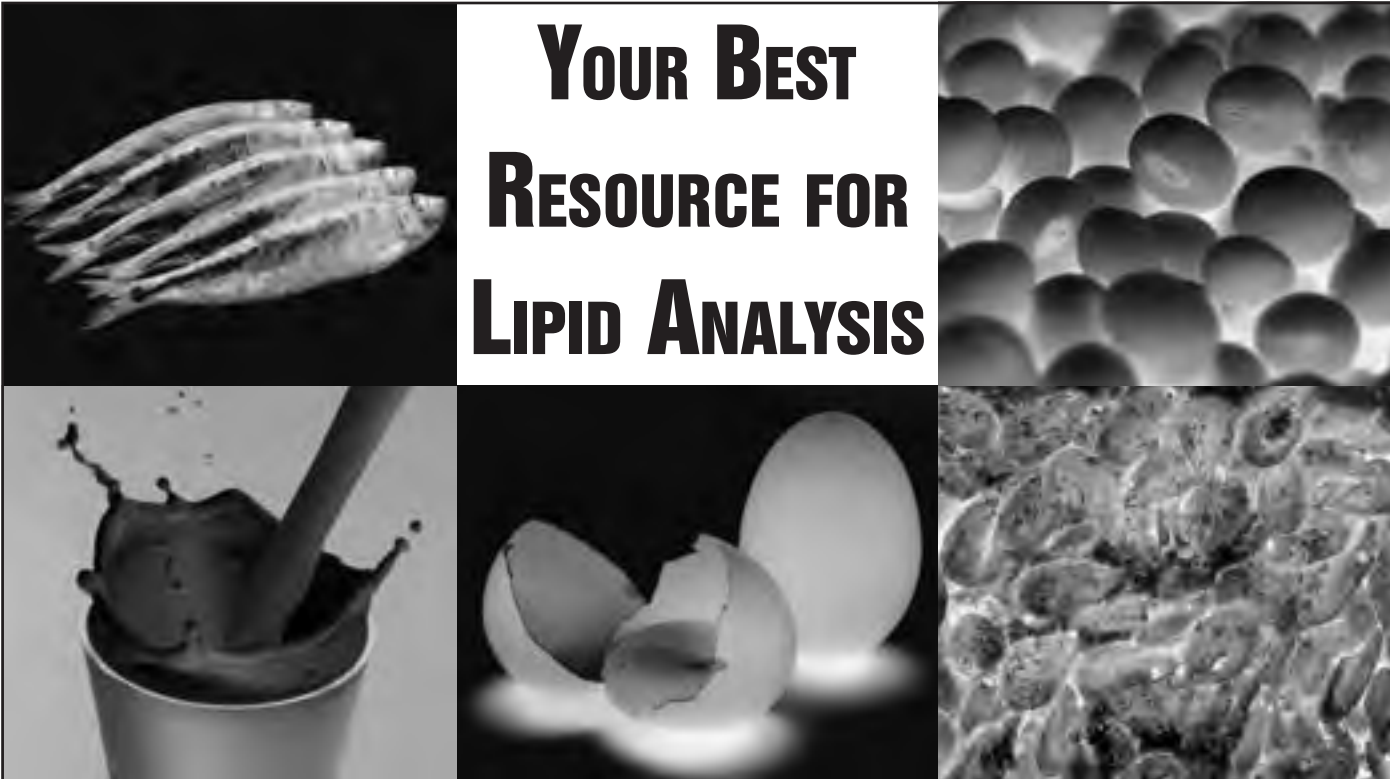
ENVIRONMENTAL IMPLICATIONS AND CURRENT USE STATUS

Aside from the economic benefits gained from product sales and eliminating the expense for waste disposal, producing salt-lick products could be a solution for companies that are seeking a more environmentally sustainable method for the disposition of spent bleaching earth. Since the publication of the patent application, the inventory has received inquiries from potential users who are particularly concerned about their environmental vulnerability with the present disposal methods.

The inventor has applied for the registered trademark "Grow Max Salt Lick" for use with salt-like products containing spent bleaching earth. This trademark provides a brand name that is functionally descriptive. The trademark could be available for use by enterprises that obtain the right to exploit the patent.

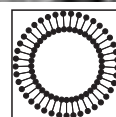
To date, inquiries have been received from edible oil processors, livestock feed and mineral suppliers, and other interested enterprises. Discussions are ongoing. To date, no commitments have been made by the inventor to authorize use. Viable options will be identified and fully explored before any decisions are made.

Norman J. Smallwood is owner of The Core Team®. He can be contacted at Norm.thecoreteam@verizon.net.



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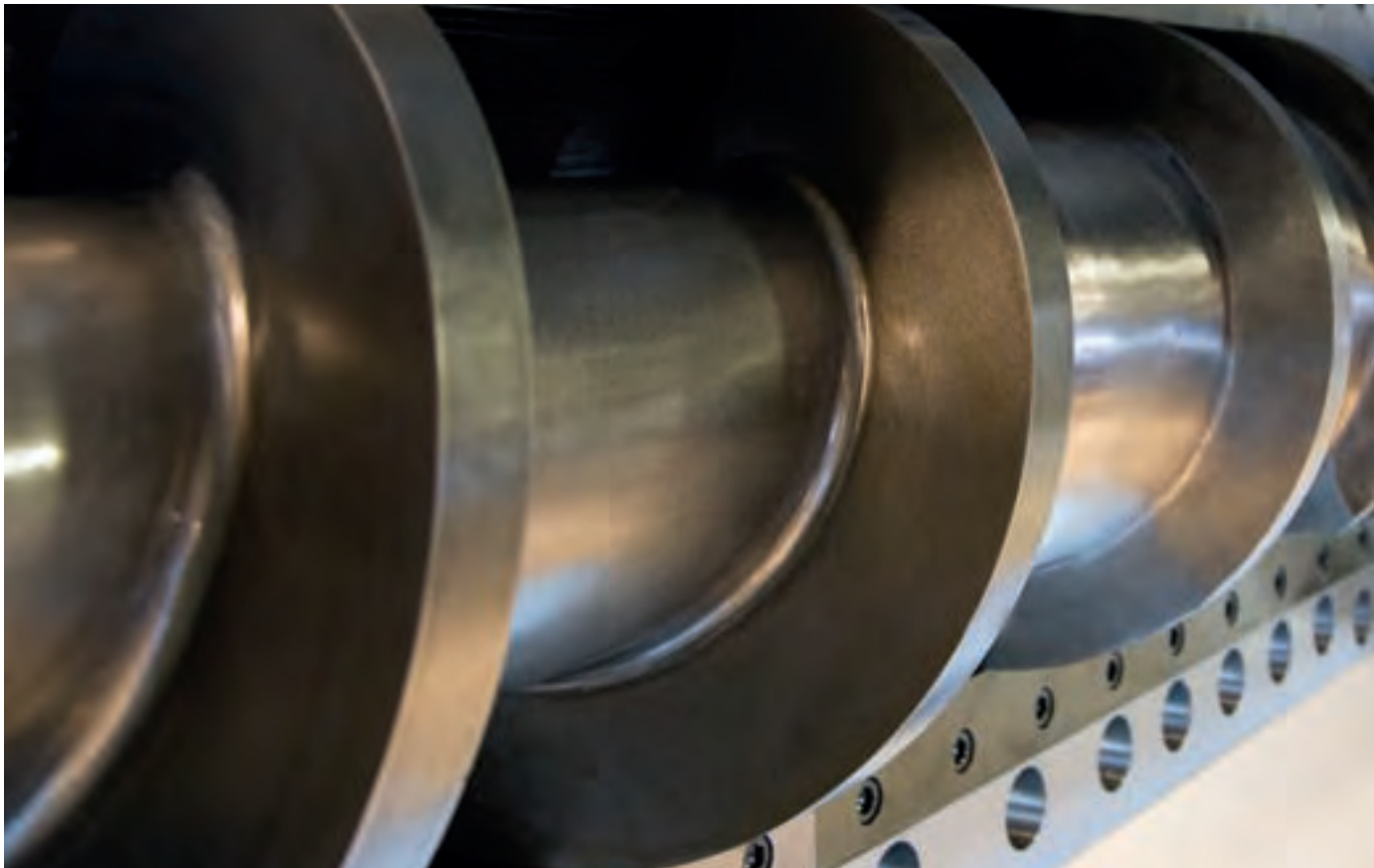
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The *Enid News* reports that Northstar Agri Industries has postponed the construction of a canola processing plant in Enid, Oklahoma, USA, owing to a drought that reduced canola yield by 50% last year. The plant requires 750,000 to 1 million acres of canola to support it, which is expected to be available in 2016.

■■■

DuPont Nutrition & Health has opened an Innovation Application Center in Gurgaon, India. The Innovation Application Center, which can be expanded at a later date, will employ 15 to 20 people by the end of 2015. DuPont Nutrition & Health also has an existing blending facility for specialty ingredients in Sohna, Haryana, India.

■■■

The US Food and Drug Administration (FDA) and the Environmental Protection Agency (EPA) have signed a Memorandum of Understanding (MoU) to share data on pesticides and toxic substances. The MoU establishes a process of disclosure and sharing of their respective databases that will facilitate the decisions of each agency related to food safety, veterinary medicine, and cosmetics. This data sharing will provide the most up-to-date information and assist in coordinating reviews between the agencies when a given substance is regulated by both agencies. For example, a manufacturer of an antimicrobial food wash is required to demonstrate to the FDA that its use is safe and does not contaminate food. The manufacturer may also be required to demonstrate to EPA that use of the same substance (labeled as a pesticide) will not hurt the environment.

■■■

The Roundtable on Sustainable Palm Oil (RSPO) has purged 15 members for failing to submit mandatory annual reports for three straight years. An additional 62 members were suspended for failing to report for two consecutive years. The reports are meant to measure progress toward certifying palm oil operations or purchasing certified sustainable palm oil, according to the World Wildlife

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NEWS & NOTEWORTHY



GHS implementation continues

Few would argue with the objective of GHS, which proposes to protect workers, consumers, and the environment by globally harmonizing how hazardous substances and mixtures are classified and labeled. This voluntary effort—formally known as the Globally Harmonized System of Classification and Labeling of Chemicals—has been decades in the making and is in varying stages of implementation in the 60+ countries that currently participate in the effort (see Table 1 on page 290).

The project began in 1990 when the International Labor Organization developed and adopted a convention and recommendation on Safety in the Use of Chemicals at Work. In 1992, the United Nations (UN) Conference on Environment and Development echoed the call for global harmonization of safety data and labeling, saying “a globally harmonized hazard classification and compatible labeling system, including material safety data sheets and easily understand-

able symbols, should be available, if feasible, by the year 2000.”

It was not feasible. In fact, it took 10 years before the first version of GHS was adopted in December 2002 by the UN’s Sub-Committee of Experts on the Globally Harmonized System of Classification and Labeling of Chemicals. That document, which was published in 2003, provided countries with the regulatory building blocks to develop or modify existing national chemical safety programs in accordance with the internationally agreed-upon provisions of the GHS. The fifth revision appeared in 2013; the sixth is expected in 2015.

June 1 brings with it an important GHS deadline in several areas of the world. It marks the final implementation date of the European Union’s (EU) CLP (for classification, labelling and packaging) regulations. It is also the date on which US manufacturers, importers, distributors, and employers must comply with OSHA’s revised Hazard Communication Standard (HazCom) of 2012. OSHA, or the Occupational Safety and Health

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Table 1. GHS implementation in the industrial triad, Canada, and the BRIC countries

Country or region	Final implementation Substances	Mixtures	Enabling regulation(s)	Regulatory authority or authorities
Brazil	Fully in effect	June 1, 2015	Standard ABNT NRB 14725:2009; based on UN GHS 3rd Rev. (2009). See http://tinyurl.com/GHS-Brazil .	Ministry of Labor and Employment, Ministry of Transport, Ministry of Health
Canada	June 1, 2017	June 1, 2017	Final Hazardous Product Regulation published in February 2015; based on GHS 5th Rev. (2013). See http://tinyurl.com/GHS-Canada .	Health Canada, Canadian Centre for Occupational Health and Safety
China	Fully in effect	Fully in effect	See http://tinyurl.com/GHS-China-Regs .	Ministry of Industry and Information Technology
European Union	Fully in effect	June 1, 2015	Classification, Labelling and Packaging regulations of 2008 (CLP-Regulation (EC) No 1272/2008). See http://tinyurl.com/GHS-EU-Regs .	Directorate-General for Enterprise and Industry; Directorate-General for Mobility and Transport; Environment Directorate-General; Directorate-General for Health and Consumers; European Chemicals Agency
India	Not yet implemented	Not yet implemented	Draft rules were released in late 2011. Final rules have yet to be issued.	Ministry of Environment, Forests, and Climate Change
Japan	Fully in effect	Fully in effect	Industrial Safety and Health Law, the Poisonous and Deleterious Substances Control Law and the Chemical Substances Control Law, based on GHS Rev. 4 (2011). See http://tinyurl.com/GHS-Japan .	Ministry of Health, Labour and Welfare; Ministry of Economy, Trade and Industry; Ministry of the Environment; Ministry of Land, Infrastructure, Transport and Tourism
Russian Federation	?	?	Currently, the use of GHS is voluntary. GHS classification and labeling will become mandatory when the Technical Regulations on the safety of chemical products come into force (expected in 2015). Five new standards, implementing GHS Rev. 4 (2011), have been on the books since August 1, 2014. See http://tinyurl.com/GHS-RuFed .	Ministry of Industry and Trade of the Russian Federation; Federal Agency on Technical Regulating and Metrology; Russian Research Center on Standardization, Certification and Testing of Raw Materials; Coordinating Informational Service Center for CIS enterprises
United States	June 1, 2015	June 1, 2015	Revised OSHA Hazard Communication Standard, based on GHS Rev. 3 (2009), published in March 2012. OSHA has implemented three additional hazards: pyrophoric gases, simple asphyxiants, combustible dust, and hazards not otherwise classified. See http://tinyurl.com/GHS-USA .	Occupational Safety and Health Administration; Environmental Protection Agency; Department of Transportation; Consumer Product Safety Commission

Sources: DHI Group (<http://ghs.dhigroup.com/PagesGHS/GHS.aspx>); www.ghslegislation.com; United Nations Economic Commission for Europe (<http://tinyurl.com/GHS-UNECE>)

Administration, is the lead regulatory agency, although three other US agencies also have input and, sometimes, conflicting regulations. These are the Environmental Protection Agency, the Department of Transportation, and the Consumer Product Safety Commission.

The most obvious changes called for by GHS are changes to chemical classification, safety labels, and safety data sheets (SDSs; formerly called material safety data sheets or MSDSs). GHS organizes SDS content and formatting into 16 sections with a prescribed order. Labels have six standardized components, including specific language contingent upon chemical classification. Pictograms are required on the labels to alert users about the chemical hazards to which they may be exposed. Each pictogram consists of a symbol on a white background framed within a red border and represents a distinct hazard.

The hazard class designates the nature of the hazard (for example, flammability). The hazard category indicates the degree of severity within the class. In the United States, the hazard evaluation of mixtures is based on data for the mixture as a whole; if those data are not available, importers and manufacturers may generalize from data on individual ingredients and/or similar mixtures.

BUILDING BLOCK APPROACH TO CLASSIFICATION

GHS is structured in “building blocks” that correspond to the different hazard classes and categories used to describe the nature of the dangerous substances or mixtures.

Competent authorities in each country are free to choose which hazard classes and categories, or building blocks, they adopt. To complicate things, however, the United States has chosen to regulate four hazard classes not found in the UN’s GHS or as implemented in other countries or regions, such as the European Union’s CLP (for classification, labelling and packaging) regulations. This means that companies cannot use European SDS or labels in the United States.

The first three new classes added the OSHA HazCom 2012 are comparatively straightforward: pyrophoric gases (or gases that ignite spontaneously in air at 130°F or below), simple asphyxiants (substances or mixtures that displace oxygen in the ambient atmosphere), and combustible dusts. The fourth class is a catchall category: “hazards not otherwise classified” or HNOCs. HNOCs do not have to be noted on product labels but do have to appear on SDSs.

Because combustible dust (CD) was classified individually by OSHA as a hazard—rather than as an HNOC—CD hazards must be dealt with on labels and SDSs under HazCom 2012. However, OSHA failed to define CD in the revised standard. Specifically, shipments of products that could produce CDs—such as whole grain (including oilseeds)—when used in processing or where dust is produced, is subject to the new rule. A coalition of industry groups, including the National Grain and Feed Association (NGFA) and National Oilseed Processors Association filed a legal challenge in 2013 against OSHA, asserting that the agency had violated the rule-making process in promulgating HazCom 2012. The legal review was pursued on the grounds that the agency violated several federal laws for, among other things, not seeking public comment on including CD during its rulemaking process. The District of Columbia Circuit of the US Court of Appeals denied the petition in October 2014.

“For the grain, feed and processing industry, the biggest practical, bottom-line implication of the court ruling is that, starting June 1, 2015 . . . raw grains and oilseeds shipped to downstream customers will be required to comply with OSHA’s requirement to issue a safety data sheet . . .,” said

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Fund, a founding member of RSPO. Most of the terminated members are small palm oil processors or traders. Three are consumer goods manufacturers in France, according to a report on Mongabay.com.

IN MEMORIAM

Long-time AOCS member Willem van Nieuwenhuyzen, 70, died on March 8, 2015, of cancer.

After various international jobs at Unilever, Eridania Beghin-Say, Central Soya, and Solae-DuPont, he founded Lecipro Consulting in Limmen, Netherlands, in 2004. He was also a founding member and executive director of the International Lecithin and Phospholipid Society (ILPS).

Within AOCS, Mr. van Nieuwenhuyzen was active in the Phospholipids Division, served on the Membership Development Committee, and was an associate editor of *Inform* for more than a decade. As such, he contributed many meeting reports and articles to the magazine. Indeed, his service to AOCS continued to the very end of his life when he wrote and edited a chapter for the new AOCS Press book on polar lipids during his final illness.

“We all lost a friend, a companion, a supervisor, and an exhaustless operator for the dissemination of knowledge and for networking [with] people sharing his interests in the fields of lecithin,” noted current ILPS President Bernd Diehl. “He was a charismatic, pleasant, and warmhearted person.”

Mr. van Nieuwenhuyzen is survived by his wife Jacobijn.



Inform has learned that Mat (Matěj) Schwitzer, an AOCS member, died at his home in London on October 27, 2014, at the age of 97.

A chemical engineer by training, Dr. Schwitzer was at one point a sales director for Akzo. “He was particularly well known for his work on fatty amines and became managing director of a manufacturing division based in Holland,” writes fellow AOCS member Kurt Berger in an obituary published on the Society of Chemical Industry website. (See www.soci.org/News/SCI/Matthew-Schwitzer.) Among other assignments after leaving Akzo and becoming a consultant, Berger notes, Dr. Schwitzer worked with the Malaysian government on the long-term development of that country’s oleochemical industry.

Dr. Schwitzer is survived by a son, three daughters, and six grandchildren. ■

OSHA offers HazCom 2012 enforcement relief and guidance

OSHA has recognized a problem with the June 1 enforcement date of the revised Hazard Communication Standards of 2012. A number of companies report they have been unable to produce SDSs for formulations because many suppliers have not yet provided updated materials for raw materials down the supply chain. Therefore, OSHA offered a solution, which is covered in depth in *Inform's* new Regulatory Update column on page 309. OSHA's Enforcement Guidance document is available at <http://tinyurl.com/OSHA-HCS-Guidance>.

NGFA President Randy Gordon in that group's October 31, 2014, newsletter.

NGFA, along with several other trade groups, is developing a guidance document for both members and nonmembers that will include templates for SDSs for combustible dust. "Our goal is to complete the document by mid-April," said Director of Safety and Regulatory Affairs Jess McCluer in an interview conducted shortly at the end of March. More information about the guidance document is available at www.ngfa.org.

FURTHER CD COMPLICATIONS

The National Fire Protection Association (NFPA; Quincy, Massachusetts, USA) is expected to release its new overarching standard, NFPA 652, Fundamentals of Combustible Dust, by the end of 2015. Meanwhile, NFPA 61—an agricultural industry-specific CD standard—is in its regular cyclical revision process. OSHA has been working on its own CD rule since 2009. However, the release of this proposed rule has been delayed continually; recently the rule was reclassified as a long-term project. Industry observers suggest that reclassification may be because the agency is waiting for NFPA 652 to be made final in order to incorporate it into the OSHA standard by reference.



1.2



Whereas the US HazCom 2012 standard states that whole grain, when processed, produces combustible dust, Canada's GHS-aligned Hazardous Products Regulations (HPR)—released in February 2015—exempts food. "Notably, the HPR do not regulate products that are shipped in a nondust form but which, when processed, would present the hazard of combustible dust," the Canadian regulations state. "However, the HazCom 2012 requires such products to be accompanied by a label and SDS. This difference is not an impediment to harmonization because the voluntary provision of a label or SDS for such products in Canada will not be viewed as noncompliant with the requirements." (See <http://tinyurl.com/GHS-Canada-Gazette> for the complete HPR entry.)

The UN's Sub-Committee of Experts on the Globally Harmonized System of Classification and Labeling of Chemicals is also working on a definition of combustible dust that could be used as a building block in countries' implementation regulations. The United States—which is to say OSHA—chairs both the sub-committee and the correspondence group working on the GHS CD component.

NEITHER GLOBAL NOR HARMONIZED?

"The new [GHS] classification process has actually become so complex that it is confusing and no longer understandable to the average worker, including chemists, toxicologists, chemical hygienists, and chemical workers," said one industry observer based in the United States, who asked to remain anonymous. "Our burden should not be capturing threshold weighted values but focusing on how to protect our . . . workers and customers."

A further difficulty for multinational companies is that OSHA did not adopt all of the UN's GHS recommendations (or the EU's, for that matter). Missing are marine pollutants, category 5 materials, and numerical cross-references on labels, according to Transportation Skills Programs Inc. (TSP), a consultancy based in Kutztown, Pennsylvania, USA. "If you were to buy a copy of the UN's Hazard Communication Recommendations, as we did, you would be shocked at the differences between the final OSHA GHS and the original international UN GHS," writes TSP's Robert Keegan on his blog at www.transportationskills.com. In addition, implementing countries have based their regulations on different GHS revision documents (see Table 1, page 290).

As so often is the case, it is the small- and medium-sized companies that are having the most difficulty with regulatory compliance, in all areas of the world. "Not every company has the manpower," notes a second industry observer, who also asked to remain anonymous. "Regulatory compliance is a cost of doing business without a revenue stream to offset it. That said, at least everybody's in the same boat." ■

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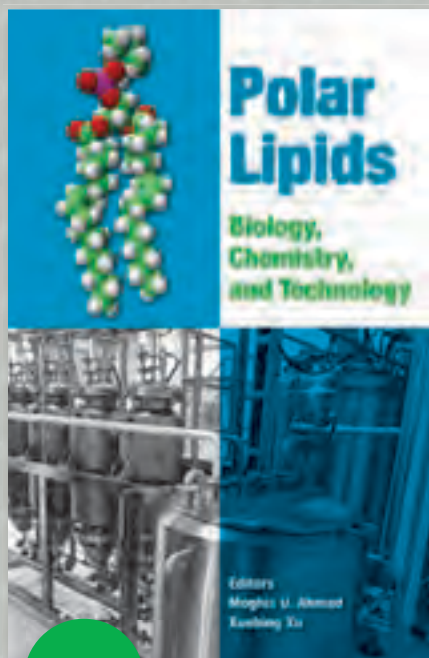
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NEW



Polar Lipids

Biology, Chemistry, and Technology

Edited by Moghis U. Ahmad and Xuebing Xu
2015. Hardback. TBD pages.

ISBN 978-1-630670-44-3. Product code 275

List: \$155 • AOCS Member: \$120

Polar Lipids will be a valuable reference resource providing thorough and comprehensive coverage of the application and utilization of polar lipids in food and nutrition, and health and disease. Chapters cover chemistry and chemical synthesis, biosynthesis and biological effects, functional and nutritional properties, applications, processing technologies, and future trends of a variety of polar lipids—including glycolipids, ether lipids, phenol lipids, serine phospholipids, omega-3 phospholipids, rice lecithin, palm lecithin, sunflower lecithin, sugar- and protein-based lipids, lysophospholipids, and more.



NEW



Sunflower

Chemistry, Production, Processing, and Utilization



AOCS MONOGRAPH SERIES ON OILSEEDS, VOLUME 7

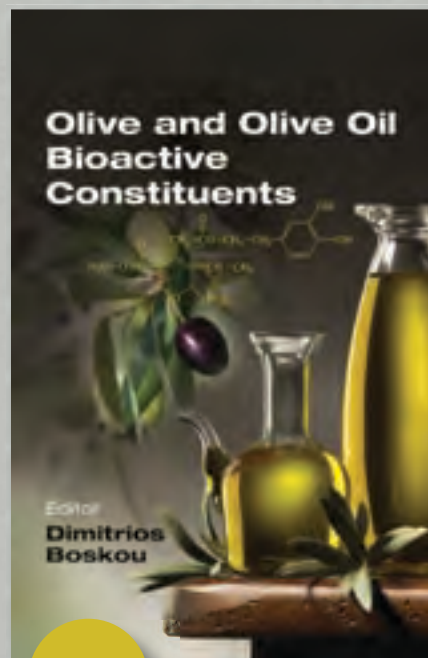
Edited by Enrique Martínez-Force, Nurhan Turgut Dunford, and Joaquín J. Salas

2015. Hardback. TBD pages.

ISBN 978-1-893997-94-3. Product code 224

List: \$230 • AOCS Member: \$175

This comprehensive reference book delivers key information on all aspects of sunflower. With over 20 chapters, this book provides an extensive review of the latest developments in sunflower genetics, breeding, processing, quality, and utilization, including food, energy and industrial bioproduct applications. World-renowned experts in this field review U.S. and international practices, production, and processing aspects of sunflower.



NEW



Olive and Olive Oil Bioactive Constituents

Edited by Dimitrios Boskou

2015. Hardback. TBD pages.

ISBN 978-1-630670-41-2. Product code 277

List: \$165 • AOCS Member: \$125

The market is flooded with products posing as elixirs, supplements, functional foods, and olive oil alternatives containing phenols obtained from multiple olive sources. This technically-oriented book will be of value to nutritionists and researchers in the biosciences. It unravels the body of science pertaining to olive minor constituents in relation to new chemical knowledge, technological innovations, and novel methods of recovery, parallel to toxicology, pharmacology, efficacy, doses, claims, and regulation.

Topics include: the biological importance of bioactive compounds present in olive products; developments and innovations to preserve the level of bioactives in table olives and olive oil; and importance of variety, maturity, processing of olives, storage, debittering of olives and table olives as a valuable source of bioactive compounds.

Argentine biodiesel exports rose 40% year-to-year in 2014 to almost 1.6 million metric tons (MMT), according to Reuters and the Federal Statistics Agency. Total production rose 29% to 2.6 MMT. The Argentine government cut the tax on biodiesel exports in 2014 to boost the industry, which had been affected by European biodiesel import tariffs, Reuters said. The export numbers almost certainly will increase in 2015, now that the US Environmental Protection Agency decided in January 2015 to allow Argentine biodiesel producers an alternative feedstock-tracking plan under the Renewable Fuel Standard.

■ ■ ■

BiofuelsDigest.com (BD) reports that the throughput of biofuels through Rotterdam—Europe's most important port for the handling, storage and production of biofuels—decreased by 15% in 2014 compared to 2013, representing a drop of 3.8 million metric tons. "This is primarily due to a decrease in imports of biodiesel from Indonesia and Argentina as a result of extra import duties and taxes on dumping," BD noted.

■ ■ ■

In late February 2015, Archer Daniels Midland Co. (ADM; Chicago, Illinois, USA) "temporarily" shifted production at its Velva, North Dakota, USA, processing facility from biodiesel to other products and cut production at its other North American facilities, ADM spokesperson Jackie Anderson said in an emailed statement to the Reuters news service. Production had not resumed at the time *Inform* went to press in March.

■ ■ ■

The government of Ethiopia is offering tax breaks and free land leases for up to seven years to biofuels developers, according to a blogger for the *Financial Times* (see <http://tinyurl.com/Ethiopia-biodiesel>). The goal is to raise biodiesel production to at least 450 million liters/year within the next five years. "Ethiopia's fuel bill weighs heavily on its current account and foreign currency reserves," the blog account noted. "It currently has reserves to cover only 2.2 months' worth of imports—almost half the 4.3 months it had in 2010–11." ■

ENERGY



EU draft law caps biofuel production

A draft law to cap the production of traditional biofuels and accelerate the shift to alternative sources such as seaweed and waste was approved by the Environment Committee of the European Commission on February 24, 2015. It aims to reduce greenhouse gas emissions that result from the increasing use of agricultural land to produce biofuel crops. A second reading agreement may take place in the second quarter of 2015, according to a news release from the European Parliament (EP).

"This has been an enormously challenging file. And at the same time a very interesting one. And it's complicated technically and technologically. I love this kind of political challenge and hope we will take it to a good result in the trilogues," said lead member of the EP Nils Torvalds of Finland after the committee's amendments to the draft law were approved by 39 votes to 26, with four

abstentions. (Trilogues are meetings attended by representatives of the EP, the Council, and the Commission.)

Current legislation requires EU member states to ensure that renewable energy accounts for at least 10% of energy consumption in transport by 2020. However, the draft law mandates that first-generation biofuels from food crops should not exceed 6% of the final energy consumption in transport by 2020. Further, advanced biofuels, sourced from seaweed or certain types of waste, should account for at least 1.25% of energy consumption in transport by 2020.

Using farmland to produce biofuel crops reduces the area available for food crops. This adds to pressure to free up more land, often through deforestation, to grow more food—a process known as indirect land use change (ILUC). However, deforestation in itself increases greenhouse gas emissions, which may cancel out part of the beneficial effects of using biofuels.

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The European Biodiesel Board released a statement by Secretary General Raffaello Garofalo, who noted that the draft law “brings the unreasonable ILUC fight one step forward in an escalation where the losers are European citizens and the winner is fossil petrol.” Garofalo also suggested that the EP “ignored recent international ISO evidence that ILUC figures are completely unreliable.”

The EP called as long ago as 2008 for the ILUC factor to be taken into account in EU biofuels policy, which has a budget of €10 billion per year.

Miscanthus outperforms switchgrass and corn stover

A recent study simulated a side-by-side comparison of the yields and costs of producing ethanol using miscanthus, switchgrass, and corn stover. The fast-growing energy grass miscanthus was the clear winner. Models predict that miscanthus will have higher yield and profit, particularly when grown in poor-quality soil. It also outperformed corn stover and switchgrass in its ability to reduce greenhouse gas emissions.

“One of the reasons for interest in these second-generation cellulosic feedstocks is that if they can be grown on low-quality soil, they wouldn’t compete for land with food crops, such as corn. The study shows that although miscanthus yield was slightly lower on marginal, low-quality land, a farmer would have an economic incentive to grow miscanthus on the lower quality land first rather than diverting their most productive cropland from growing corn,” said University of Illinois at Urbana-Champaign (UIUC; USA) agricultural economist Madhu Khanna. Khanna co-authored the study along with a team of economists and environmental and crop scientists from the Energy Biosciences Institute at UIUC.

According to Evan DeLucia, professor in integrative biology at UIUC, “There has been skepticism about whether energy crops can be grown on low-quality land. What has been lacking is a side-by-side analysis that isolates the effect of soil quality on yield. In this study, we do that. We were able to keep all of the conditions the same and only change the soil attributes,” he said.

The study used real data from UIUC’s energy farm and other locations across the country to calibrate the model so that the findings are generalizable. The model simulated yields and greenhouse gas savings under 30 years of variable weather conditions.

Another goal of the study was to examine the cost and greenhouse gas implications of using these sources of biomass for biofuel production. The study found that even if corn stover is harvested responsibly (removing only 30–50%, depending on tillage choice) there was still a loss in soil carbon and the overall savings in greenhouse gas emissions were much smaller than those seen with switchgrass and miscanthus.

“It’s tempting to use corn stover because it’s already there—farmers who grow corn don’t have to plant another crop to produce biofuel feedstock,” Khanna said. “But in some cases corn stover is only about 59% cleaner than gasoline while miscanthus is about 140% cleaner. So if we want to reduce greenhouse gas emissions and lower the carbon intensity of our fuel, energy grasses such as miscanthus and switchgrass are going to result in the biggest reductions, not corn stover.”

Making the choice of miscanthus-based ethanol more pleasing at the pump for consumers is another consideration. Khanna

says that a price on carbon would be one way to equalize the cost of using gasoline and ethanol for consumers when filling up their tanks.

“Ethanol made from miscanthus would need a much smaller carbon price to make it desirable to produce and for consumers to purchase as compared to ethanol from switchgrass and corn stover. Even though corn stover may in some cases be cheaper to produce, it is a much more expensive way to reduce greenhouse gas emissions than energy grasses,” Khanna said.

The study appeared in *Environmental Science & Technology* (<http://dx.doi.org/10.1021/es5052588>, 2015). It was supported by funding from the North Central Regional Sun Grant Center at South Dakota State University through a grant provided by the US Department of Energy Office of Biomass Programs and from the Energy Biosciences Institute, University of California, Berkeley.

New technology for renewable jet fuel

Emerging Fuels Technology (EFT; Broken Arrow, Oklahoma, USA) has begun licensing what the *Biofuels Digest* calls “an innovative new technology that significantly reduces the cost of manufacturing renewable fuels from bio oils.” Such fuels typically are known as HEFA fuels, or hydro processed esters and fatty acids. They are certified for 50% blends with conventional jet fuel.

According to the report, the EFT technology benefits from three synergistic features of Fischer-Tropsch (F-T) gas-to-liquids plant design: lower operating and capital expenses as well as higher yield. However, co-processing paraffins derived from syngas as part of the F-T process with paraffins derived from hydroprocessing esters and fatty acids is not currently covered under the ASTM International D7566 standard for aviation fuel.

See the complete article at <http://tinyurl.com/EFT-Jet>.

Grant for glycerin purification

Biofuel producers in Western Canada will soon be able to purify and convert raw glycerol more cost-effectively, thanks to an investment of more than \$500,000 investment, announced in late February 2015.

With this funding, researchers at the University of Saskatchewan, led by Professor of Chemical Engineering Ajay Dalai, will be able to purchase specialized equipment for the development and commercialization of new, more efficient and affordable glycerol purification and conversion technologies.

Glycerol, a byproduct of processing vegetable oils into biodiesel, must be separated from biodiesel, as it does not burn effectively. Although raw glycerol has limited commercial value, the new purification technology could double the price that companies can charge for the substance, in turn adding more value to biodiesel production.

The university plans to develop and file three patents: one for the purification technology, and two for the conversion technologies. A Saskatchewan start-up company is expected to manufacture all three technologies for commercial use, and subsequently market them. ■

BRIEFS

The European Patent Office recently awarded the Barry Callebaut Group Patent EP 2 152 091 for a process to produce milk chocolate with only 25% of fat by weight, versus 36% fat in a comparable chocolate. The process involves using a powder that replaces some of the fat in the chocolate with other ingredients such as sugar, sweeteners, fibers, or proteins. The powder is at least 15% fat by weight and at least 40% other ingredients, with the fat partially coating the other ingredients. The powder is mixed with other chocolate components, then refined and conched.

The calorie reduction offered by the process will depend on which ingredients are used to replace fat in the powder. "Sugar as filler will have a caloric reduction of 5–10% while sweeteners and/or dietary fibers as filler will have a reduction of 15–30%," says Marijke De Brouwer, global R&D manager at Barry Callebaut (Zürich, Switzerland). While reduced-fat chocolates have thus far failed to achieve commercial success because of compromised flavor, De Brouwer claims that Callebaut's patented process produces chocolate with "good taste and texture."



Since 2003, total investments in the Gulf Cooperation Council's edible oil industry have more than doubled to \$646 million, according to a report from the Gulf Organization for Industrial Consulting (GOIC; Doha, Qatar). The workforce in the edible oil industry has doubled, while the number of factories and total capacity have increased by 50%. The GOIC attributes the boom to growing populations and affluence in the region, which consumed 1.2 million metric tons of vegetable, animal, and marine oil in 2013. Because of the Gulf's lack of agricultural capacity, most edible oil is imported in the form of unrefined oils, which are then refined within the region. Saudi Arabia has the largest

CONTINUED ON PAGE 301

FOOD, HEALTH & NUTRITION



EVOO stable for short-term deep-frying

Fast-food restaurants eschew extra virgin olive oil (EVOO) for deep-frying because it is more costly than seed oils such as corn, canola, or soybean. Also, EVOO has a lower smoke point—the temperature at which an oil starts to break down and produce visible smoke—than refined seed oils, making it unsuitable for long-term, repetitive deep-frying. However, a recent study in the *Journal of the American Oil Chemists' Society* indicates that home cooks may want to add EVOO to their deep-frying repertoire because the oil is more stable than refined seed oils during short-term deep-frying of French fries (<http://dx.doi.org/10.1007/s11746-015-2599-2>, 2015). In addition, EVOO may

enhance the nutritional value and flavor of the fried food.

Researchers led by Alexandre Guedes Torres at the Federal University of Rio de Janeiro, in Brazil, compared the oxidative stability and changes in fatty acid composition of EVOO, refined soybean oil, and refined sunflower oil during short-term deep-frying of French fries. The team deep-fried French fries in the oils at 180°C for up to 75 minutes of successive frying, taking oil samples at 25, 50, and 75 minutes.

The investigators found that the three varieties of EVOO tested—from Spain, Brazil, and Portugal—had lower degrees of oleic and linoleic acid losses and lower formation of free fatty acids and carbonyl compounds than the refined seed oils after 75 minutes of frying. When the researchers analyzed the French fries, they found that

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deep-frying transferred oleic acid, tocopherols, and flavor compounds from EVOO into the French fries, possibly enhancing their nutritional value and flavor. “EVOO is a highly attractive medium for short-term deep-frying of French fries,” the researchers conclude.

Fat—the sixth taste?

Fat may soon be joining sweet, salty, sour, bitter, and umami as one of the basic tastes, according to a recent review in the journal *Flavour* (<http://dx.doi.org/10.1186/2044-7248-4-5>, 2015). Recognizing fat as a taste may change how scientists view food consumption and obesity, say Russell S.J. Keast and Andrew Costanzo, researchers at Deakin University in Burwood, Australia.

Although Aristotle classified fat as a taste as early as 330 BCE, more recently fat has been associated with the texture, flavor release, and thermal properties of foods. In their review, Keast and Costanzo argue that fat satisfies most, if not all, of the criteria for a substance to be considered a taste. Just like other taste substances such as sugars or proteins, fatty acids in food bind to receptors on taste bud cells of the tongue, generating an electrical signal that travels to the brain. This signal has physiological effects, such as stimulating satiety hormones that cause a person to feel full.

While people can readily identify sweet or salty foods, the perception of fat as a taste is less well defined. However, animal and human studies suggest that individuals who are less sensitive to fatty acids are more prone to overeating and obesity. “In other words, the more you taste fat, the less fat you eat,” say the researchers. Lean individuals may have more fat receptors on their taste buds than obese individuals, causing them to feel full sooner and consume less food. In addition, diet appears to modulate a person’s perception of fat: A high-fat diet can make people less sensitive to the taste of fat, whereas a low-fat diet increases sensitivity.

“The next five to 10 years should reveal, conclusively, whether fat can be classified as the sixth taste, but no matter what, there appears to be a functional significance to oral chemosensing of fats,” the authors say.

Statins increase diabetes risk by 46%

Doctors prescribe statins to lower serum cholesterol. However, some studies indicate that the drugs can have an unpleasant side effect: They increase a person’s risk of developing type 2 diabetes by varying degrees (10–22%). Now a new report in *Diabetologia* suggests that the diabetes risk associated with statins may be much higher than previously recognized (<http://dx.doi.org/10.1007/s00125-015-3528-5>, 2015).

Prior studies of statins and diabetes risk have had limitations. They typically included only participants at high risk of cardiovascular disease, excluding those in the general population who take statins for prevention. Also, most previous studies relied on participants’ self-reporting of diabetes or on their fasting glucose measurements, which may miss people with

newly developed diabetes who have not yet been diagnosed. Finally, prior population-based studies did not examine the mechanism of diabetes induction by statins.

To address these concerns, researchers led by Markku Laakso at the University of Eastern Finland and Kuopio University Hospital conducted a six-year follow-up study of 8,749 nondiabetic men (aged 45–73 years) enrolled in the Metabolic Syndrome in Men (METSIM) study. At the beginning of the study, 2,142 men (24.5%) were on a statin medication. Six years later, 625 men had developed diabetes, as determined by physician diagnosis, oral glucose tolerance test, HbA1c levels, or start of an antidiabetic medication.

When the researchers compared the diabetes incidence in men treated or untreated with statins, they found that statin use increased diabetes risk by 46%, after adjustment for age, body mass index, smoking, and other confounding factors. Looking at individual statins, Laakso found that only simvastatin and atorvastatin increased diabetes risk in a dose-dependent manner, whereas four other types of statins did not increase the risk of diabetes.

The research also uncovered a possible mechanism for diabetes induction by some statins. Men treated with statins showed, on average, 24% decreased insulin sensitivity and 12% decreased insulin secretion compared with untreated men. These effects were dose-dependent for simvastatin and atorvastatin. Hyperglycemia worsened in men treated with statins.

“The higher risk of type 2 diabetes found in our study suggests that previous studies may have underestimated the significance of statin-induced diabetes,” the authors say. However, they caution that their study included only white men and may not be applicable to women or other ethnic groups.

Common food emulsifiers alter mouse gut microbiome

Two emulsifiers used to improve the texture and shelf life of processed foods alter the community of microbes—or microbiome—in the mouse gut, according to new research published in *Nature* (<http://dx.doi.org/10.1038/nature14232>). These microbial changes promoted colitis, obesity, and metabolic syndrome in mice administered the emulsifiers at low levels in their chow or drinking water.

Since the mid-20th century, the incidence of inflammatory bowel disease, which includes Crohn’s disease and ulcerative colitis, and of metabolic syndrome, an obesity-related disorder linked with diabetes and cardiovascular disease, have dramatically increased. “A key feature of these modern plagues is alteration of the gut microbiota in a manner that promotes inflammation,” says Andrew T. Gewirtz at Georgia State University, in Atlanta, USA. “Food interacts intimately with the microbiota so we considered what modern additions to the food supply might possibly make gut bacteria more proinflammatory.”

The researchers fed two commonly used food emulsifiers, carboxymethylcellulose and polysorbate-80, to mice at levels (up to 1% of the water/chow) comparable to those used in processed foods. Compared with mice that did not receive emulsifiers, mice treated with either emulsifier showed changes in

species composition of their gut microbiomes, with a shift toward microbial species that more strongly promote inflammation. The emulsifier-altered microbiota were better able to digest and infiltrate the dense mucus layer that lines the intestine, which normally lacks bacteria.

Wild-type mice fed the emulsifiers showed signs of chronic intestinal inflammation. They also ate more food, gained more weight, and had more hyperglycemia and insulin resistance—hallmarks of metabolic syndrome—than their non-emulsifier-fed littermates. Genetically engineered mice with abnormal immune systems developed colitis when treated with the emulsifiers.

These emulsifier-induced effects were not observed in germ-free mice that lack gut microbiota, indicating that the altered gut microbiome underlies the intestinal inflammation and metabolic syndrome. Fecal transplants from emulsifier-treated mice to non-emulsifier-treated, germ-free mice caused inflammation and metabolic changes in the recipients, further supporting the role of the microbiome in the emulsifier-induced effects.

“[These results] suggest that the broad use of emulsifying agents might be contributing to an increased societal incidence of obesity/metabolic syndrome and other chronic inflammatory diseases,” the authors say. However, the results need to be confirmed in human populations before any definitive conclusions can be made. ■

oil refining capacity (59.5%), followed by the United Arab Emirates (29.1%). Vegetable oils make up 80% of all oils used in the region, animal oils 19%, and marine oils 1%.

■■■

Current guidelines from the US Environmental Protection Agency (EPA) and the Food and Drug Administration (FDA) advise pregnant and nursing women to limit their consumption of albacore tuna to six ounces per week. Tuna can be contaminated with methylmercury, a neurotoxin that may adversely affect cognitive thinking, memory, language, and motor skills of children exposed to the toxin *in utero*.

However, tuna is also an affordable and abundant source of long-chain omega-3 fatty acids, which have been linked with decreased cardiovascular disease and improved infant neurodevelopment. After reviewing available evidence, the 2015 US Dietary Guidelines Advisory Committee (DGAC) has concluded that even for pregnant women who ate double the recommended amount of albacore tuna, “all evidence was in favor of net benefits for infant development and [cardiovascular disease] risk reduction.” Further, the DGAC recommends that the EPA and FDA reevaluate their recommendations that pregnant and breastfeeding women (or those who may become pregnant) limit their tuna consumption. ■

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The Finnish Food Safety Authority Evira has introduced a DNA bar code system that will help establish the origin of a food, according to a March 16, 2015, news release downloaded from <http://evira.fi>. “A given lactic acid bacterial strain can be added to origin-protected cheeses, allowing the origin of the cheese to be ascertained by means of the DNA of the strain,” says Annikki Welling of Evira’s Research Unit for Chemistry and Toxicology. “The added lactic acid bacteria do not change the taste, texture, or composition of the cheese.” In the future, synthetic DNA bar codes may be sprayed onto a food surface, or added to foods such as olive oil to make it easier to verify their authenticity.

■ ■ ■

The European Food Safety Authority (EFSA) may ask biotech companies for more data on genetically modified crop safety when the firms apply for reauthorization after 10 years. The draft guidance document issued by EFSA’s GMO unit late last year (downloaded from efsa.europa.eu) describes mandatory requirements for renewal applications, such as post-market environmental monitoring reports and updated bioinformatics.

■ ■ ■

The Chinese government has pledged additional support for research on genetic modification techniques, especially for crops, according to the government’s No. 1 Central Document issued on February 1, 2015. The No. 1 Central Document is the first major policy document issued each year by the Central Committee of the Communist Party of China. This year’s document, which focuses on agricultural modernization, emphasizes the need for more studies to establish the safety of GM technology. It also encourages scientists to engage the Chinese public on the benefits of GM crops, which many in the country consider unsafe to eat. Since the late 1990s, land areas planted with GM crops have declined in China. The new document indicates that China is now ready to become a world leader in the development and application of GM technology. ■

BIOTECHNOLOGY



IARC links glyphosate to cancer

Glyphosate, the broad-spectrum herbicide in Monsanto’s popular weed killer Roundup, is “probably carcinogenic to humans,” according to a panel of experts convened by the World Health Organization’s International Agency for Research on Cancer (IARC). Their assessments of glyphosate and four other agricultural chemicals were published online March 20, 2015, in *The Lancet Oncology* ([http://dx.doi.org/10.1016/S1470-2045\(15\)70134-8](http://dx.doi.org/10.1016/S1470-2045(15)70134-8), 2015).

Many of today’s agriculture crops have been genetically engineered to be resistant to glyphosate. These so-called “Roundup-ready” crops have further increased the popularity of glyphosate, making it the world’s most widely produced herbicide by volume. In addition to agricultural use, glyphosate is frequently used as a weed killer by home gardeners.

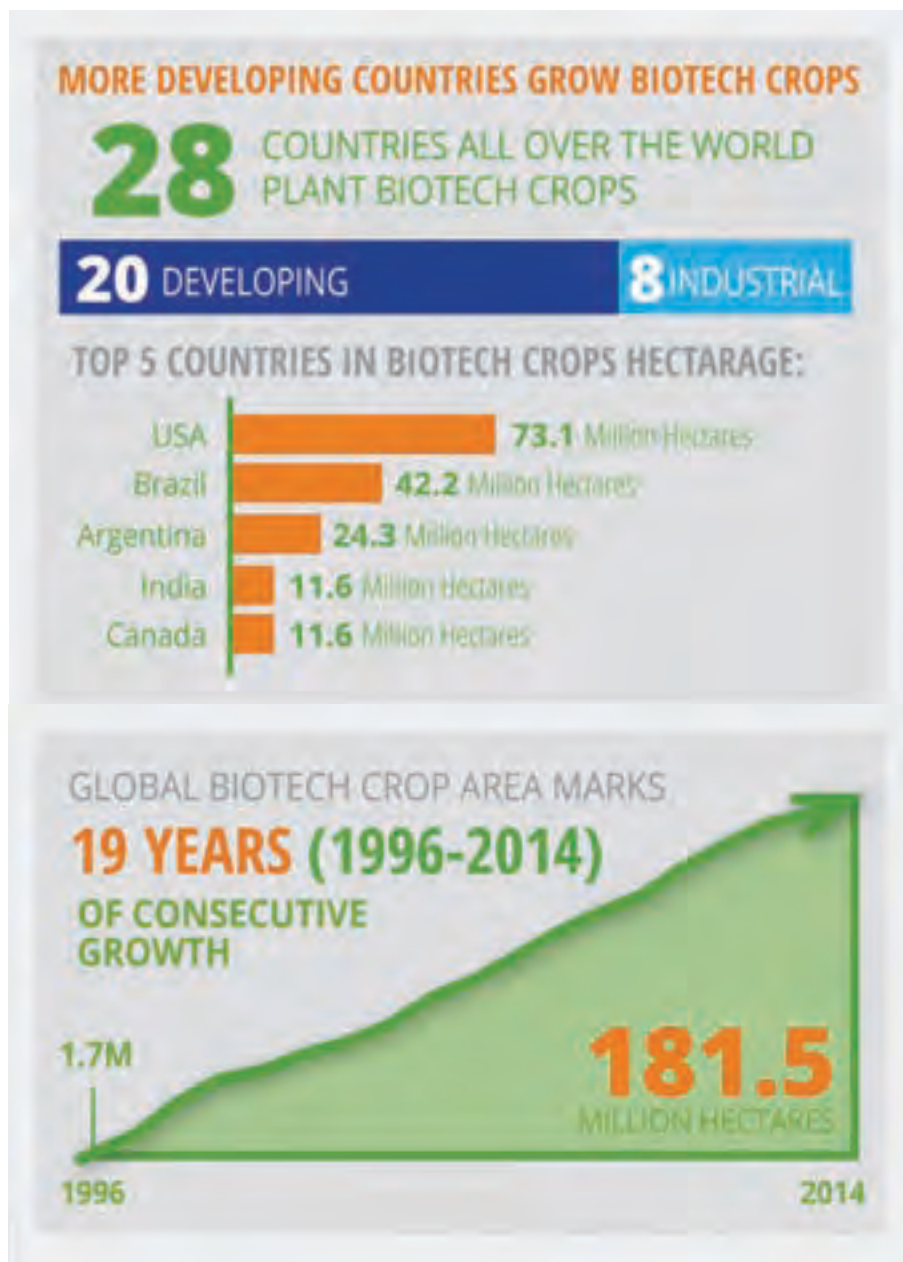
IARC, which regularly reviews the carcinogenicity of industrial chemicals, foods, and even occupations, rated glyphosate

as category 2A, “probably carcinogenic to humans.” Although there is little evidence linking glyphosate to cancer in humans, studies in which mice and rats exposed to the herbicide developed tumors, and the ability of glyphosate to damage DNA in cultured human cells, provided sufficient evidence to classify glyphosate in category 2A, the report says.

Because IARC has no regulatory authority, the panel’s decision does not affect glyphosate’s label, registration, or use. However, the report may influence public perception, as well as future decisions about product safety made by governmental regulatory agencies. The US Environmental Protection Agency (EPA) is conducting a formal review of glyphosate, which the EPA does not currently consider carcinogenic to humans.

A Monsanto official responded to the IARC report in a news release dated March 23, 2015, downloaded from <http://news.monsanto.com>. “We are outraged with this assessment,” said Robb Fraley, Monsanto’s chief technology officer. “This conclusion is

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Reprinted with permission from James, Clive, Global status of commercialized biotech/GM crops for 2014: ISAAA Brief 49. ISAAA, Ithaca, NY, USA, 2014, <http://www.isaaa.org>.

inconsistent with the decades of ongoing comprehensive safety reviews by the leading regulatory authorities around the world that have concluded that all labeled uses of glyphosate are safe for human health. This result was reached by selective 'cherry picking' of data and is a clear example of agenda-driven bias."

Anti-GMO group requests emails of researchers

On January 28, 2015, a nonprofit group opposed to genetically modified (GM) products sent US Freedom of Information Act (FOIA) requests to 14 scientists at four universities, asking them to turn over any email correspondence with large agricultural companies including Monsanto, Syngenta, DuPont, and Dow, as well as biotech and grocery trade groups and certain public relations firms.

The group, US Right to Know (USRTK), based in Oakland, California, hopes to expose ties between scientists and industry that may unduly influence research on GM products. The executive director of USRTK, Gary Ruskin, helped manage Proposition 37, the unsuccessful 2012 California ballot initiative that would have required labeling of foods containing GM ingredients. Now Ruskin and USRTK are targeting scientists who either spoke out publicly against Proposition 37 or wrote pro-GM articles posted on the website GMO Answers (<http://gmoanswers.com>).

Kevin Folta, a biologist at the University of Florida in Gainesville, USA, had already complied with the FOIA request, according to a February 19 post on his blog (<http://kfolta.blogspot.com>). He writes, "Frankly, I don't care. Nothing was done wrong. There was no crime committed, and my handful of interactions with anyone in the Big Ag world aren't too exciting." However, some researchers worry that USRTK's tactics will stifle academic freedom on an increasingly contentious issue.

Biotech crops show continued growth in 2014

A record 181.5 million hectares of biotech crops were grown globally in 2014, up 6 million hectares from 2013, according to a report released January 28 by the International Service for the Acquisition of Agri-Biotech Applications (ISAAA Brief 49-2014; downloaded from <http://isaaa.org>). A total of 28 countries grew biotech crops last year, representing more than 60% of the world's population.

Since 1996, more than 10 biotech crops have been approved and grown worldwide, including maize (corn), soybean, cotton, papaya, eggplant, and potato. The biotech crops were genetically modified to introduce benefits to the farmer and consumer, such as drought tolerance, insect and disease resistance, herbicide tolerance, and increased nutrition and quality.

In 2014, the United States led biotech crop production at 73.1 million hectares, up 4% (3 million hectares) from 2013. Brazil ranked second with 42.2 million hectares, and Argentina was third with 24.3 million hectares of biotech crops. Twenty of the 28 nations that grew biotech crops were developing countries. Among these, India led the way with 11.6 million hectares planted in 2014, followed by China at 3.9 million hectares.

The report highlights key benefits of biotech crops, such as alleviating hunger, increasing the income of small farmers, reducing pesticide use, mitigating carbon dioxide emissions, and reducing the need for more cultivated acres to feed the

world's growing population. A recent meta-analysis by Klumper and Qaim (*PLoS One*, <http://dx.doi.org/10.1371/journal.pone.0111629>, 2014), which was cited in the report, indicates that genetic modification technology has reduced chemical pesticide use by an average of 37%, increased crop yields 22%, and increased farmer profits 68% from 1995 to 2014.

According to a statement on the ISAAA website, the ISAAA is a not-for-profit organization with an international network of centers designed to contribute to the alleviation of hunger and poverty by sharing knowledge and crop biotechnology applications.

How healthful is high-oleic soybean oil?

Oil from genetically modified soybeans that contain a high proportion of oleic acid has been touted as a more healthful alternative to commodity soybean oil. However, new research suggests that, at least in mice, high-oleic soybean oil may promote obesity, glucose intolerance, and fatty liver—but not insulin resistance. The study results were presented March 6, 2015, at the Endocrine Society's 97th annual meeting in San Diego, California, USA.

Conventional soybean oil contains about 55% linoleic acid, a polyunsaturated fat. Linoleic acid is not very oxidatively stable, so in the past manufacturers used a process called partial hydrogenation to increase soybean oil's shelf life and heat stability. However, this hydrogenation generated trans fats, now widely thought to be detrimental to health. In contrast, Plenish™ high-oleic soybean oil (from DuPont Pioneer) contains more than 75% oleic acid (a "heart-healthy" monounsaturated fat abundant in olive oil), less than 3% α -linolenic acid, and no trans fat. Oleic acid is very stable, eliminating the need for hydrogenation.

Poonamjot Deol and Frances Sladek at the University of California, Riverside, USA, wanted to test the metabolic effects of high-oleic soybean oil in a model organism. So they fed four groups of 12 mice each different diets for six months. The control group received a low-fat diet, in which 5% of daily calories came from fat. The other three groups received diets with 40% of calories from fat—an amount common in the standard Western diet. One diet was high in saturated fat from coconut oil, and one had 41% of the saturated fat replaced with commodity soybean oil. The final group had 41% of the saturated fat replaced with high-oleic soybean oil.

Mice fed either soybean oil exhibited increased incidence of fatty liver, glucose intolerance, and obesity than mice fed coconut oil. However, mice that received the high-oleic soybean oil had less fat tissue: They weighed about 30% more than controls on a low-fat diet, whereas mice on the diet containing commodity soybean oil weighed 38% more than controls. Mice fed coconut oil weighed only about 13% more than controls. Although commodity soybean oil promoted insulin resistance, the high-oleic soybean oil did not.

"The genetically modified soybean oil does seem to have fewer negative metabolic consequences than regular soybean oil in mice, but it may not necessarily be as health[ful] as olive oil, as has been assumed by its fatty acid composition, and it is certainly less health[ful] than coconut oil, which is primarily saturated fat," says Sladek. ■

Mintec

STATISTICAL ANALYSIS FROM MINTEC

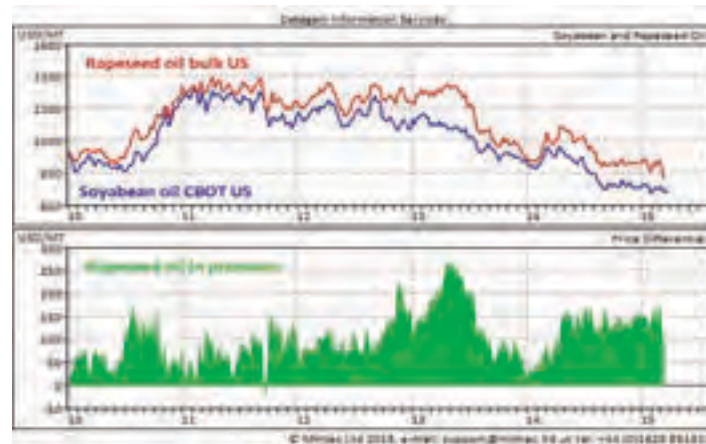
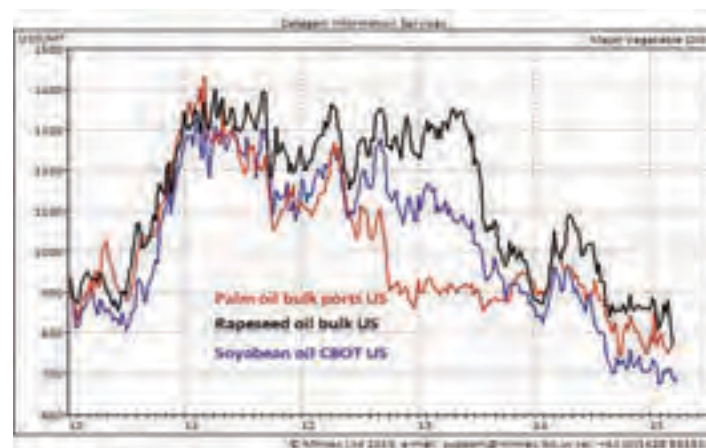
Loraine Hudson

Prices for all major vegetable oils remained low in the first quarter of 2015. Palm oil, rapeseed oil, and soybean oil prices are all down sharply compared to the same period in 2014. Production levels of the three major oils are all expected to increase.

Global production of palm oil is forecast to reach a record high of 62.4 million metric tons (MMT) in 2014/15, up 5% year-on-year. Global soybean oil production is also forecast to rise 5% year-on-year to 47.4 MMT. Global rapeseed oil production is expected at 27.0 MMT, up 2% year-on-year.

Palm oil demand for biodiesel production was low in Indonesia at the beginning of 2015, as a result of the competitive prices of crude oil compared to palm oil. However, this effect had been somewhat offset by an increase in a subsidy for palm-based biofuels. In Malaysia however, re-imposed export tariffs on palm oil in March resulted in palm oil becoming unattractive as a feedstock for biofuel. As a result, monthly usage of palm oil for biofuel in Malaysia is expected to fall during 2015.

In addition, low prices of crude oil are expected to weigh on biodiesel production in Argentina and Brazil, resulting in reduced demand for soybean oil as a biodiesel feedstock. Low European prices for rapeseed have led to producers shifting production towards rapeseed oil due to better profitability. In Canada, rapeseed oil production is expected to rise 4% year-on-year to 3.2 MMT.



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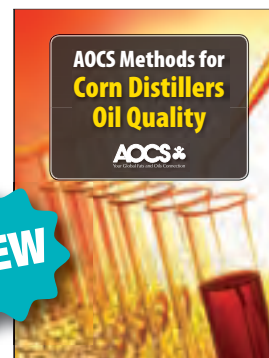


The *Methods of Analysis for Cosmetic Ingredients* are a collection of AOCS Official Methods chosen by SCC for its membership. Using these standardized methods in regular quality control and testing of ingredients will provide producers and consumers with additional assurance of the quality of the final products.

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This PDF contains 19 methods for determining the quality of corn distillers oil, including: cleanliness, purity, water content, acidity, sulfur, phosphorus, and oxidative stability. It includes information from the newest release of the *Official Methods and Recommended Practices of the AOCS*, 6th Edition, 3rd Printing.



Método Oficial de AOCS

NUEVO

Los Métodos Oficiales y Prácticas Recomendadas de AOCS contienen reconocida metodología de actualidad requerida para las pruebas técnicas del Programa de Competencia Técnica de Laboratorios (Laboratory Proficiency Program - LPP), así como para la Certificación de Laboratorios de AOCS. Los métodos de AOCS son internacionalmente reconocidos para el comercio y algunos de ellos están incluidos en la lista de la Comisión del Codex Alimentarius.

AOCS se complace en ofrecer sus Métodos en Español. Consulte más información en <http://www.aocs.org/esmethods>

BRIEFS

University of Arizona startup company GlycoSurf (Tucson, Arizona, USA) has finalized an exclusive license agreement for a technology to synthesize glycolipid surfactants, according to a news release issued by Tech Launch Arizona on March 13, 2015. GlycoSurf, which has hired Chett Boxley as its CEO, is now poised to enter the marketplace.

GlycoSurf, LLC, was founded in 2013 by University of Arizona researchers Jeanne E. Pemberton, Raina M. Maier, and Cliff S. Cross. The licensed technology involves chemical synthesis of nontoxic, biodegradable surfactants that may be used as environmentally friendly replacements for petroleum-based surfactants in cosmetics and personal care products, as well as in environmental applications such as oil spill clean-ups.



The US Environmental Protection Agency (EPA) has developed a new Safer Choice label to indicate cleaning and other products that are safer for consumers and the environment. The label, which was developed in collaboration with manufacturers, environmental and health advocates, and consumers, replaces the 15-year-old Design for the Environment, or "DfE," label. Products bearing the Safer Choice label will begin to appear in stores in the third or fourth quarter of 2015.

Currently, more than 2,000 products qualify to carry the Safer Choice label. There are three versions of the label: the regular Safer Choice label, a Safer Choice label for business or industrial/institutional products, and a Safer Choice label for fragrance-free products. All versions include the statement "Meets US EPA Safer Product Standards."

The Safer Choice program is essentially the same as the DfE program but with new labels designed to better resonate with consumers. Steps to apply for the Safer Choice label, which include third-party and EPA evaluation of all product ingredients and packaging, can be found at www2.epa.gov/safer-choice/steps-get-safer-choice-label-your-product. ■

HOME & PERSONAL CARE



Reconfigurable complex emulsions

Researchers have found a new way to fabricate complex emulsions that can be remodeled by surfactant, light, or pH change (*Nature*, <http://dx.doi.org/10.1038/nature14168>, 2015). The reconfigurable emulsions could find diverse applications in drug delivery, cosmetics, chemical sensing, and environmental cleanup.

Many foods, personal care products, cosmetics, and medicines contain emulsions—tiny droplets of a liquid suspended in another immiscible liquid, often with the help of surfactants. Reconfigurable emulsions would allow scientists to control the release of compounds within droplets, or otherwise alter the properties of an emulsion on demand.

In the new method, researchers led by Timothy M. Swager and Daniel Blankschtein at the Massachusetts Institute of Technology, in Cambridge, USA, made use of the temperature-sensitive miscibility of hydrocarbon, silicone, and fluorocarbon liquids to make complex emulsions. In the simplest case, hexane and perfluorohexane were immiscible at room temperature, but with gentle heating (above 23°C) in an aqueous solution of a nonionic fluorosurfactant (Zonyl FS-300), the liquids mixed to form an emulsion in water. The researchers then cooled the mixture below 23°C, and the hexane and perfluorohexane phases separated to form a hexane-in-perfluorohexane-in-water (H/F/W) double emulsion.

The researchers found that they could manipulate the configuration of the

CONTINUED ON NEXT PAGE

emulsions with different surfactants. By making emulsions using light- or pH-sensitive surfactants and then exposing the emulsions to light or acid, respectively, the team converted F/H/W droplets to H/F/W droplets. The droplets switched because of changes in surface tension between their layers. At the midpoint of the reversal, the liquids became what are known as Janus droplets, in which each component of the droplet makes up one hemisphere. The researchers also made four-phase emulsions that they reconfigured by adding increasing amounts of a different surfactant.

The researchers say that the method is easily scalable for industrial purposes. “You can use these emulsions for delivery applications, cleanup applications, anything where you need to protect something, shield something, or pick up and deliver something,” says Vishnu Shresht, a co-author of the study. “It’s like a package that you can open and close at will.”

Efficient new catalysts for making olefins from seed oils

Linear α -olefins (LOAs) are the building blocks of many commodity chemicals such as surfactants, lubricants, waxes, perfumes, and antimicrobial agents, as well as fine specialty chemicals and pharmaceuticals. A process called ethenolysis can produce LAOs from fatty acids in seed oils—a renewable source of the chemicals. However, the process is not cost-effective at an industrial scale. Now researchers have synthesized new cyclic alkyl amino carbene (CAAC) ruthenium complexes that are remarkably active catalysts for ethenolysis, enabling large-scale synthesis of LAOs from seed oils (*Angewandte Chemie*, doi.org/10.1002/anie.201410797, 2015).

Many fatty acids from seed oils contain at least one carbon–carbon double bond, which can be split by ethylene (ethenolysis) to produce LAOs. A catalyst is needed to speed up the reaction, but the catalysts identified so far have relatively low turnover numbers (TONs). TON is a measure of catalyst efficiency, and TONs of at least 35,000 and 50,000 are recommended for the cost-effective manufacturing of specialty and commodity chemicals, respectively. Previous catalysts for this reaction have TONs of 2,000–35,000 and require unacceptably high amounts of catalyst (10–100 ppm).

So Vanessa M. Marx, Robert H. Grubbs, Guy Bertrand, Scott C. Virgil, and their colleagues at California Institute of Technology and the University of California, San Diego, synthesized 13 new catalysts based on 4 existing CAAC ruthenium catalysts. They then tested the catalysts in the ethenolysis of methyl oleate, a fatty acid methyl ester derived from seed oils, in ethylene gas.

Many of the catalysts had TONs greater than 100,000 using only 3 ppm of catalyst. The combination of a small R¹ and a large R² substituent on the CAAC ruthenium catalyst appeared to be ideal. The purity of ethylene gas also significantly affected the TON. When the researchers used 99.995% pure ethylene (versus 99.95% used previously), the best catalyst had a TON of 340,000 at only 1 ppm of catalyst.

The new CAAC ruthenium catalysts have the highest reported TONs for any ethenolysis catalyst to date, well above the minimum value of 50,000 required for industrial

production. The researchers anticipate that the catalysts will find many applications in the “economically and environmentally sustainable production of LAOs, as well as other valuable terminal olefins, especially through the transformation of seed oils and their derivatives.”

Engineered yeast strain makes lipids for oleochemicals, biofuels

A new mutant yeast strain makes large amounts of lipids that could be used to produce biofuel or oleochemicals found in many household and personal care products. The improved lipid yield could make the yeast strain an economically viable platform for efficient oleochemical production (*Metabolic Engineering*, <http://dx.doi.org/10.1016/j.ymben.2015.02.003>, 2015).

Microbes such as bacteria, yeast, and microalgae have been used to produce oleochemicals including alkanes, alkenes, fatty alcohols, fatty esters, and polyunsaturated fatty acids. However, the yield of lipids typically is not high enough to compete with conventional processes for chemical and fuel production. In previous work, Hal S. Alper and colleagues at The University of Texas at Austin (USA) genetically engineered a highly oleaginous strain of the yeast *Yarrowia lipolytica* that produces lipid titers exceeding 25 g/L in bioreactors. Yet improvements in titers and yields were still needed to make chemical production by the yeast economically viable.

So Alper and his colleagues turned to evolution to produce a more efficient oleaginous yeast strain. The team treated the previously engineered *Y. lipolytica* strain with ethyl methanesulfonate (EMS), which caused random mutations in the yeast’s DNA. They allowed the mutagenized yeast cells to grow in a liquid culture containing high-glucose medium, which triggers lipid production, and then centrifuged the cells at a low speed. Some cells did not settle to the bottom of the tube but instead floated at the top of the medium. Fluorescent staining with a dye called Nile Red showed that these floating cells were full of lipids, whereas cells that settled were not.

The researchers reasoned that the floating cells had mutations that allowed them to produce abundant lipids. So they isolated the floating cells and propagated them through five rounds of growth and centrifugation to select the hardest lipid-producing cells. The researchers then plated the floating cells on agar plates containing Nile Red to identify the yeast colonies with the highest lipid production.

The highest-yielding yeast strain produced 38.9 g/L of lipids, and 87% of its cellular content was lipid. The new strain also produced lipids faster than the parental strain (about 0.51 g/L/h compared with 0.21 g/L/h). Whole-genome sequencing revealed that a mutation in the succinate semialdehyde dehydrogenase (*uga2*) gene was primarily responsible for the increased lipogenesis of the mutant strain. The evolutionary engineering approach yielded lipid titers that are the highest to date for this organism. “This work presents an improved host strain that can serve as a platform for efficient oleochemical production,” the researchers say. ■

OSHA offers enforcement relief for HazCom 2012 compliance

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

The US Occupational Safety and Health Administration (OSHA) will provide enforcement “relief” for product formulators, manufacturers and importers who can demonstrate that they cannot comply with the June 1, 2015, implementation deadline for the new hazard communication standard (HazCom 2012) because they could not get classification information from their suppliers, an OSHA official has said.

HazCom 2012, which aligns the US hazard communication standard with the UN Globally Harmonized System (GHS) of classification and labeling of chemicals, says that chemical manufacturers and importers must update their classifications, container labels and safety datasheets (SDSs) for mixtures. Distributors are allowed to ship products labelled under the old scheme until December 1, 2015.

In response to a petition for extension of the compliance deadline, the agency considered it “reasonable to give a little bit of extra time, so we are giving people six months to develop the 2012 compliance SDSs and, once that is done, six months to update the label,” Maureen Ruskin, director of OSHA’s Office of Chemicals Hazards–Metals, told the GlobalChem conference in Baltimore, Maryland, USA, [in early March]. But that relief will be contingent on companies showing that they had done “reasonable diligence” and “good faith efforts” to get the information from their suppliers, she said.

Enforcement guidance, issued by OSHA on February 9, makes it clear that upstream chemical suppliers that do not have HazCom 2012-compliant SDSs or labels available for downstream mixture manufacturers or importers will not be in compliance, and must provide compliant SDSs to such customers, “with the first shipment and after an SDS is updated.” Furthermore, if a downstream mixture manufacturer or importer asks for an updated SDS prior

to receiving a new shipment, the upstream supplier must provide it immediately.

Where a mixture producer has asserted that it was unable to comply with the June 1 deadline, it must be able to demonstrate that it exercised “reasonable diligence and good faith” to do so. The guidance sets out the steps it must take in order to be able to demonstrate this. An OSHA spokeswoman told *Chemical Watch* that manufacturers and formulators, who are experiencing delay in getting information from their suppliers, should “document all efforts to obtain the required information, such as attempts to contact them to obtain the proper information, reasonable efforts to find alternate suppliers, who could provide timely and accurate classification, or to find relevant data themselves.”

Regarding the amount of extra time companies will be allowed, the guidance says mixture manufacturers or importers must create compliant SDSs within six months of the date they receive all ingredient hazard information, rather than the three-month period stated in HazCom 2012. They must then create compliant container labels within six months of the date that they developed their compliant SDSs.

Those that exercised reasonable diligence and good faith to obtain the necessary classification information, but did not receive it, will be allowed to continue using SDSs and labels that comply with the previous standard, HCS 1994. Similarly, a distributor can continue to transport mixtures bearing HCS 1994-compliant labels, after the December 1, 2015, deadline stated in HazCom 2012. OSHA officials will decide how long such firms can do this on a case-by-case basis.

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Further Information

OSHA enforcement guidance (https://www.osha.gov/dcp/enforcement/hazcom_enforcement-memo.html)



Obesity in Latin America: strategies to reduce calories in confectionery and bakery products

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

Leslie Kleiner

The US Centers for Disease Control and Prevention reports that obesity is a common and serious concern; over a third of US adults are obese, leading to obesity-related conditions such as heart disease, stroke, and type 2 diabetes [1]. Although strategies to combat the “global obesity epidemic” in the United States are often discussed and debated, those in other geographical regions are not mentioned as frequently. In this article, I briefly discuss strategies for obesity prevention in some Latin-American countries, then focus in more detail on methods to reduce calories in bakery and confectionery applications.

In one study regarding obesity prevention, Kain, *et al.* [2] described current strategies to combat obesity in Chile, Mexico, Colombia, Brazil, and Peru—all of which have high obesity rates in adults and/or children. For example, the obesity rate of adults in Chile, Mexico, Brazil, and Colombia is of 35%, 35%, 20%, and 16.5%, respectively, with malnutrition coexisting with obesity in some regions of Colombia, Mexico, and Peru. Programs that have been developed to control obesity in these countries are implemented through governmental policies, academic settings, and/ or the private sector [2]. For example, Colombia developed general policies to improve the population’s nutrition and lifestyle, while Chile and Brazil focused on schools to educate the public regarding better food and lifestyle choices.

In Mexico, one way of addressing obesity prevention is by enforcing taxation on foods with a defined high-caloric value. The Mexican “Junk Food Tax” (imposed on January 1, 2014) imposes an 8% tax increase on a variety of

processed foods with a caloric value of 275 kcal/100 g or greater. Some of these foods include snacks, confectionery, custards, jams, peanut butter, ice cream, cookies, and others. The tax also applies to ingredients commonly found in baked goods, such as dried fruits, cocoa, and gelatin [3]. Based on this tax and the desire to promote healthy living, there is an ongoing effort to reformulate food products in Mexico so as to reduce their calories.

CALORIE REDUCTION IN CONFECTIONERY APPLICATIONS

The bulk of the calories in confectionary applications come from fat or from sugar, depending on the application. Examples of fat-based applications are those with high contents of cocoa butter, cocoa butter equivalents (CBEs), cocoa butter replacers (CBRs), or cocoa butter substitutes (CBSs). In a recent interview, Donna Studenka, senior applications sci-

entist at Loders Croklaan, explained how the compositions and applications of CBEs, CBRs, and CBSs differ.

“CBEs are essentially identical to cocoa butter with respect to their triglyceride composition,” she said. “They perform exactly the same as cocoa butter in confectionery products, with the same calorie count.” CBEs may be used across all applications as a partial or total replacement for cocoa butter, which costs more than any of its equivalents, replacers, or substitutes. They are often used in place of cocoa butter in white confections, and also as a first layer of enrobed bars containing oily nuts.

Conventional cocoa butter replacers (CBRs) were based on the partial hydrogenation of oil, and consequently had undesirable contents of trans-fatty acids. Today, CBR alternatives without trans-fats are available, but most are based primarily on the use of palm oil. “These palm-based CBRs require additional time to set up, and as such are used in many confectionery fillings and spreads, but are rarely if ever used in coating applications,” Studenka explained.

CBSs, are made from lauric fats (often palm kernel or coconut oils), have physical properties similar to cocoa butter, and can completely replace cocoa butter in chocolate. However, they cannot be used as a partial replacement for cocoa butter, because blending them with cocoa butter causes the chocolate to become unacceptably soft.

Although reducing fat content in confectionery applications is possible, Studenka points out that this may affect the desired sensory and functional properties of the finished product. “When the fat is reduced, other ingredients will need to be added or adjusted to achieve the target finished product properties. If more structure is needed, higher melting fractions may need to be used in the fat blend formula”

However, compound coatings can be formulated into reduced-sugar or sugar-free types. One alternative to reducing calories in compound coatings is to formulate coatings with sugar alcohols such as maltitol in lieu of sugar. Philippe Levresse, who works in technical sales for Roquette America Inc., explains that sugar has 4 kcal/g, while maltitol has 2.1 kcal/g, so substituting with maltitol can reduce calories considerably. As maltitol is about 90% as sweet as sucrose, a one-to-one substitution of maltitol for sugar leads to coatings with a similar mouth-feel and sweetness to those made with sugar. It is also possible to reduce sugar by adding ingredients with a higher nutritional value, such as soluble fibers. For example, reduced sugar coatings can be formulated with soluble fiber to achieve an overall reduction in calories of about 20% when compared to a full-sugar control formulation.

One option for reducing calories in coated pieces is to reduce the sugar or fat in the center while leaving the coating unchanged. This strategy provides the flexibility of working with a traditional full-sugar and full-fat coating, since the calories of the coating and center combined will be lower if the calories in either are reduced. A variety of modifications are possible. In the case of truffles, sugar replacement with maltitol, partial fat replacement with high-lipid algae flour, or a combination of the two, can achieve the desired reduction in calories. Examples include reduced-sugar truffles and

Further reading

1. <http://www.cdc.gov/obesity/data/adult.html>
2. Kain, J., *et al.*, Obesity prevention in Latin America, *Curr. Obes. Rep.* 3: 150–155, 2014.
3. GAIN Report Number MX3324 (4/9/2014), <http://gain.fas.usda.gov/>

pralines in which some or all of the sugar has been replaced with maltitol, and reduced-fat truffles in which the fats have been partially replaced with high-lipid algae flour.

CALORIE REDUCTION IN BAKERY APPLICATIONS

The diverse strategies for reducing calories in bakery applications include the use of lower-caloric margarines. I recently talked with Dilip Nakhasi, director of innovation, Bunge Oils, about the saturate-sparing technology developed at Bunge Oils. This technology uses proprietary non-lipid ingredients, blending, and crystallization techniques to reduce saturate levels in shortening by about 40%. When this technology is applied to margarine, which traditionally contains about 80% fat, the result is a less caloric margarine mimetic. Saturate-sparing margarine is a trans-free product that can be used in lieu of traditional margarine or shortening to make cookies, cakes, and biscuits. Due to its composition it can aid in the overall goal of reducing calories in bakery goods. Although this product is not yet commercially available, it will be first produced in the US and then in Mexico, and its use will contribute to the global effort to reduce calories in bakery goods.

Another approach to reducing calories in bakery products involves decreasing the use of sugar. Kristen Coad, food scientist for bakery and frozen dessert applications at Roquette America Inc., explained that the sugar sucrose can be substituted in a one-to-one ratio by maltitol in many bakery products, such as cookies, cakes, brownies, muffins, and fillings. If all of the sugar in a baked good is replaced by maltitol, high-intensity sweeteners can be used to increase its sweetness. Coad also explained that calories can also be reduced by replacing sugar with combinations of maltitol and soluble fiber, or by partially replacing fats in the formulation of bakery goods. In many US bakery applications, partially replacing the fat with high-lipid algal flour can create a mouth-feel and texture that is similar to the full-fat product. A combination of soluble and insoluble fibers can also act as a fat mimic.

These are just some of the many approaches and novel ingredients that can be used to reformulate food products, reduce calories, and fight obesity in Latin America and other geographical areas as well.

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.



Enzymatic interesterification

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

W. David Cowan

The aim of interesterification is to modify the melting properties of a fat or fat blend to produce a fat suitable for incorporation into margarine or other food fat product. In Fig. 1, the Solid Fat Content (SFC) profile of a blend of soybean and fully hardened soybean oil is measured before and after interesterification. Both enzymatic and chemical interesterification produces a softening of the oil. By changing the proportion or the type of fats, different melting profiles can be achieved, according to the application for the final fat product. For example, fats for margarine for use in a warm climate should have a higher melting point than one

intended for a cool climate or where air conditioning is used.

As practiced today enzymatic interesterification uses an immobilized lipase held in vertical columns and with the fat blend to be interesterified, pumped down the column. The enzyme catalyzes the exchange of fatty acids between the fats in the blend and the resulting product has the desired melting properties. In contrast to chemical interesterification, the enzyme process is a continuous one, with the oil blend being fed into the top of the reactor and continuously removed from the base. The individual reaction vessels have a water jacket to prevent solidification of the fat and the enzyme granules are held on a split screen to prevent them from getting into the oil blend as it leaves the reactor. The shape of the screen as an inverted triangle allows oil to pass through the screen gaps (150nm), while the larger enzyme particles (average 450 nm) are retained.

Fig. 2 shows the layout of a single reactor, containing between 250 and 1000 kg of enzyme. Flow rates through the column are relatively slow, of the order of 1-2 kg oil/kg enzyme/hour. An industrial installation will contain a number of reactors in series to maximize the utilization of the enzyme catalyst.

If a single reactor is used then as enzyme activity decreases, the flow rate through the column will need to be reduced in order that the required degree of conversion can be obtained. This would result in a decreasing daily output, from the interesterification unit, which is not desirable. By operating several reactors in series, enzyme activity will be the lowest in the first reactor in the series and effectively 100% in the last one. The conversion is thus shared along the reactor chain, with ~95% being completed before the oil passes into the last reactor in the line. Eventually there will not be any residual activity in the first reactor and it can be isolated from the others, the old enzyme removed and fresh added. By altering the valves controlling flow, this “new” reactor can be placed at the end of the line and production continued. If only a single reactor is used then production would have to stop while there was still 10-15% residual activity left in the enzyme column.

The cause of the loss of activity has been studied to determine if it were possible to reduce it and to be certain that enzyme protein was not leaching into the oil. Measure-

SFC of fats before and after modification

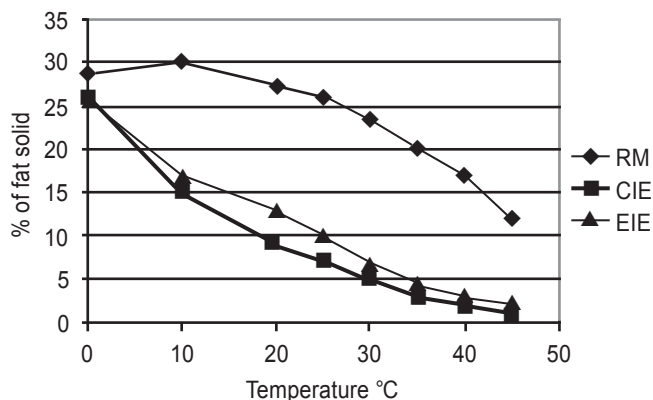


FIG. Solid fat content (SFC) of a 75% soybean: 25% fully hardened soybean oil before (RM) and after chemical (CIE) or enzymatic interesterification (EIE).

ments of protein content in the immobilized enzyme product have revealed that this remains constant even though the measured activity in the enzyme column is decreasing [1]. Also measurements of lipase activity in the oil coming from the column have not shown the presence of active enzyme or enzyme protein, suggesting that the cause of activity reduction is not physical loss of enzyme from the column.

If physical loss of enzyme is not responsible for the observed decrease in activity then other factors must be involved. Oils often contain unsaturated fatty acids which are prone to oxidation and this process can result in oxidation products being present in the oil. Refining is intended to remove these products but it has been established that when primary oxidation products as measured by the peroxide value are too high, then enzyme inactivation will occur. The rate of inactivation is proportional to the PV and at a level of PV>4, the working life of the enzyme will quickly be reduced to about 50% of the maximum value [2]

However, oxidation products are not the only materials found in oil that can cause enzyme inactivation. While the enzyme is insensitive to the presence of free fatty acids, citric acid or mineral acid residues can also reduce enzyme activity. Oils do contain small amounts of water, generally <0.2%, and this water can dissolve citric acid added as a chelating agent for metals or mineral acids (sulfuric and phosphoric) found in the bleaching earth used upstream of the interesterification process to refine the oil. Uptake of the acids into the enzyme granules results in a lowering of pH and hence a decrease in enzyme activity. The effects of these two components are additive, so for maximum enzyme working life, levels should be minimized.

Analytical methods are available to determine PV and mineral acidity in oil and the levels can be minimized by the correct preparation of the oil prior to interesterification.

David Cowan is a project manager at Novzymes A/C in Chesham, Bucks, England. He can be reached at dc@novozymes.com.

Further reading

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2. Holm, H.C. and W.D. Cowan, The evolution of enzymatic interesterification in the oils and fats industry, *Eur. J. Lipid Sci. Technol.* 110: 679–691, 2008, doi: <http://dx.doi.org/10.1002/ejlt.200800100>.

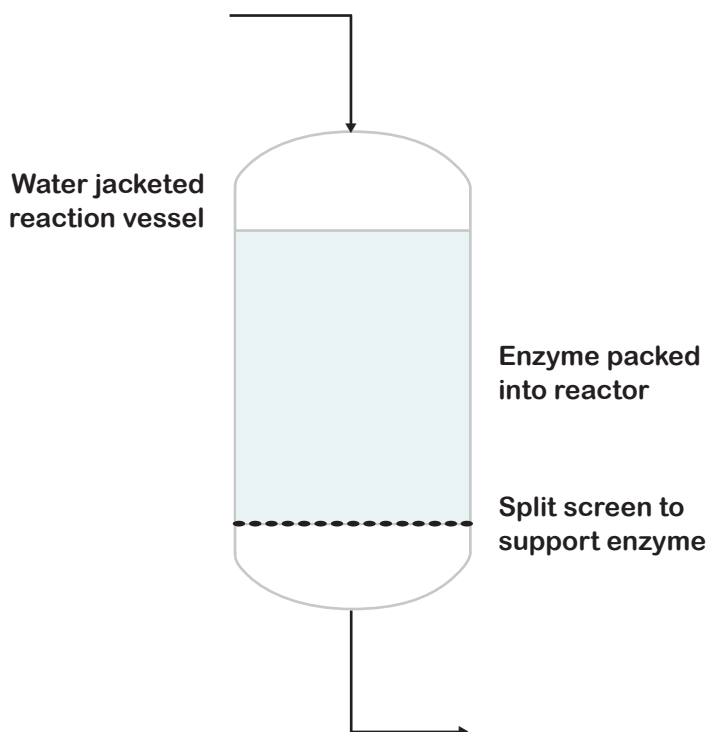


FIG. 2. Enzyme reactor for interesterification

AOCS MEETING WATCH

October 27–30, 2015. SODEOPEC2015, Hyatt Regency Miami, Miami, Florida, USA. <http://sodeopec.aocs.org>

November 12–13, 2015. AOCS Oils and Fats World Market Update 2015, The Convention Centre Dublin, Dublin, Ireland. <http://worldmarket.aocs.org>

May 1–4, 2016. 107th AOCS Annual Meeting & Expo, Calvin L. Rampton Salt Palace Convention Center, Salt Lake City, Utah, USA.

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PATENTS

Compositions comprising lecithin oils and NSAIDs for protecting the gastrointestinal tract and providing enhanced therapeutic activity

Lichtenberger, L.M., University of Texas System, US8865187, October 21, 2014

A novel pharmaceutical composition is provided by which nonsteroidal anti-inflammatory drugs (NSAIDs) are added directly to phospholipid-containing oil such as lecithin oils or to a biocompatible oil to which an phospholipid has been added to make a NSAID-containing formulation that possess low gastrointestinal (GI) toxicity and enhanced therapeutic activity to treat or prevent inflammation, pain, fever, platelet aggregation, tissue ulcerations and/or other tissue disorders. The composition of the invention are in the form of a non-aqueous solution, paste, suspension, dispersion, colloidal suspension, or in the form of an aqueous emulsion or microemulsion for internal, oral, direct, or topical administration.

Method for making carbon nanotube-based composite

Wang, L., *et al.*, Hon Hai Precision Industry Co., Ltd. and Tsinghua University, US8871300, October 28, 2014

A method for making a carbon nanotube based composite is provided. In the method, carriers, solution containing metal ions, and a carboxylic acid solution are mixed to form a mixed solution containing a complex compound. A reducing agent is added into the mixed solution. The metal ions are reduced to metal particles absorbed on the surface of the carriers. The carriers having the metal particles absorbed thereon are purified to obtain the carbon nanotube based composite.

Grease-like gel for repelling insects and preventing undesirable behavior in hoofed animals

Willey, R. and R. Numata, Pacific Tech Industries, Inc., US8871814, October 28, 2014

Grease-like compositions are provided for repelling insects and preventing undesirable behavior in hoofed animals. The compositions utilize nontoxic mineral, synthetic, or vegetable oil based gels containing silica, clay, urea, polytetrafluoroethylene, or metallic soap thickeners and capsaicin.

Adhesive Cream

Bogaert, J.P., US8871828, October 28, 2014

Improved composition of a fixing cream, in particular for tooth prostheses, containing at least one oil and/or fat, preferably of a plant type, at least one water-soluble polymer selected from the group of cellulose derivatives, at least one alkyl vinyl ether/maleic acid anhydride copolymer, the fatty acid content of the plant oil or fat contained consisting of at least 20 % bwt of unsaturated fatty acids.

Method for the froth flotation of coal

Hines, J.B., *et al.*, Georgia Pacific Chemicals LLC, US8875898, November 4, 2014

The present invention relates to the beneficiation of coal by the process of froth flotation and specifically relates to a process for the froth flotation of coal using a collector consisting essentially of a fatty acid monoester of a polyol, a fatty acid diester of a polyol, or a mixture thereof.

Biobased wax compositions and applications

McKeown, S., *et al.*, Galacta Chemicals LLC and Collateral Agent A/S, US8876918, November 4, 2014

Eicosapentaenoic acid (EPA) compositions and EPA-rich polar lipids for prophylactic or therapeutic applications are described. Production from certain cultured micro-organisms (like *Nitzschia laevis*) promotes synthesis of EPA, including polar lipids including EPA. The EPA-rich polar lipids themselves may be used as polar compounds. EPA can be selectively hydrolysed from particular positions in isolated polar lipids by lipase activity, then optionally further purified. The process bypasses reliance on diminishing fish stocks and on physico-chemical processes that may not adequately separate desirable *n*-3 HUFAs from unwanted products like DHA also found in fish oil and cultured organisms.

Production of ultrapure EPA and polar lipids from largely heterotrophic culture

Griffiths, H.D. and K.T. Geiringer, Photonix Corp., Ltd., US8877465, November 4, 2014

Eicosapentaenoic acid (EPA) compositions and EPA-rich polar lipids for prophylactic or therapeutic applications are described. Production from certain cultured micro-organisms (like *Nitzschia laevis*) promotes synthesis of EPA, including polar lipids including EPA. The EPA-rich polar lipids themselves may be used as polar compounds. EPA can be selectively hydrolysed from particular positions in isolated polar lipids by lipase activity, then optionally further purified. The process bypasses reliance on diminishing fish stocks and on

physico-chemical processes that may not adequately separate desirable *n*-3 HUFAs from unwanted products like DHA also found in fish oil and cultured organisms.

Method to prepare β -functionalized aliphatic esters

Dierker, M., *et al.*, Cognis IP Management GmbH, US8877958, November 4, 2014

The invention pertains to a new route to prepare β -functionalized carboxylic acid esters in a one-pot reaction, by reacting an olefinic acid ester in the presence of a catalyst system, comprising a Rh(I)-complex, together with an aryl boron or a diamine as nucleophilic compounds, and under oxygen-free conditions and elevated temperatures.

Deoxygenation of fatty acids for preparation of hydrocarbons

White, J.F., *et al.*, Battelle Memorial Institute, US8882990, November 11, 2014

Embodiments of methods for making renewable diesel by deoxygenating (decarboxylating/decarbonylating/dehydrating) fatty acids to produce hydrocarbons are disclosed. Fatty acids are exposed to a catalyst selected from a) Pt and MO_3 on ZrO_2 (M is W, Mo, or a combination thereof), or b) Pt/Ge or Pt/Sn on carbon, and the catalyst decarboxylates at least 10% of the fatty acids. In particular embodiments, the catalyst consists essentially of 0.7 wt% Pt and 12 wt% WO_3 , relative to a mass of catalyst, or the catalyst consists essentially of a) 5 wt% Pt and b) 0.5 wt% Ge or 0.5 wt% Sn, relative to a mass of catalyst. Deoxygenation is performed without added hydrogen and at less than 100 psi. Disclosed embodiments of the catalysts deoxygenate at least 10% of fatty acids in a fatty acid feed, and remain capable of deoxygenating fatty acids for at least 200 min. to more than 350 hr.

Oil or fat composition

Abe, T., *et al.*, Kao Corp., US8884043, November 11, 2014

Provided is an oil or fat composition, which has an MCPD-FS content (ppm) of 13 ppm or less, the content being measured by a Deutsche Gesellschaft fuer Fettwissenschaft (DGF) standard method C-III 18(09), has a diacylglycerol content of 15 mass% or more, and is subjected to deodorization treatment.

Process for producing fatty alcohols from fatty acids

Wild, R., *et al.*, Davy Process Technology, Ltd., US8884078, November 11, 2014

In a process for the production of fatty alcohol(s) a fatty acid or fatty acid mixture is subjected to esterification with a lower alkanol to form a stream comprising the corresponding lower alkyl ester or esters. The stream is vaporized and subjected to hydrogenation to form a stream comprising fatty alcohol(s)

and an amount of unconverted lower alkyl ester(s). This stream is subjected to transesterification in a wax ester reactor in the presence of a solid transesterification catalyst. Fatty alcohol(s) and wax ester(s) are then separated by distillation to yield a fatty alcohol(s) product and a stream comprising wax ester(s). The stream of wax ester(s) is passed to a second hydrogenation zone to effect hydrogenation in the liquid phase to revert the wax ester(s) to fatty alcohol(s), which are returned to the distillation separation step.

Hydrogenation of fatty acids using a promoted supported nickel catalyst

Bouwman H.J., *et al.*, BASF Corp., US8884042, November 11, 2014

The invention is directed to a process for the hydrogenation of unsaturated fatty acids to produce saturated fatty acids, said process comprising hydrogenating the unsaturated fatty acid in the presence of hydrogen and a supported nickel catalyst, said supported nickel catalyst comprising an oxidic support, 5 to 80 weight percent of nickel, calculated as atomic nickel on the weight of the catalyst, 0.1 to 10 weight percent of a copper promoter, calculated as atomic copper on the weight of the catalyst and 1 to 10 weight percent of a group II metal, calculated as metallic oxide on the weight of the catalyst.

Methods for decreasing the incidence of necrotizing enterocolitis, colic, and short bowel syndrome in an infant, toddler, or child

Davis, S.R., *et al.*, Abbott Laboratories, US8877812, November 4, 2014

Disclosed are nutritional formulations including predigested fats that can be administered to preterm infants, infants, toddlers, and children for improving tolerance, digestion, and absorption of nutrients and for reducing the incidence of necrotizing enterocolitis, colic, and short bowel syndrome. The predigested fats include fatty acid-containing monoglycerides and/or a fatty acid component.

Plastic implant impregnated with a degradation protector

Kunze, A., *et al.*, Rush University Medical Center, US8889166, November 18, 2014

A plastic implant device for a mammal that contains a rare earth metal compound tracer and a method for detecting degradation such as wear of the implanted device are disclosed. The

EXTRACTS & DISTILLATES

A comparison of actual versus stated label amounts of EPA and DHA in commercial omega-3 dietary supplements in the United States

Kleiner, A.C., *et al.*, *J. Sci. Food Agric.* 95: 1260–1267, 2015
<http://dx.doi.org/10.1002/jsfa.6816>.

Eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) are associated with health benefits throughout life and are obtained primarily through fish and fish oil supplements. Due to the growing popularity of dietary supplements, 47 commercial fish, krill, and algal oil supplements were analyzed for EPA, DHA, and other fatty acids. For fish- and krill-based supplements, the range of EPA was 81.8 to 454.6 mg g⁻¹ oil and DHA was 51.6 to 220.4 mg g⁻¹ oil. For algal oil supplements, EPA ranged from 7.7 to 151.1 mg g⁻¹ oil and DHA ranged from 237.8 to 423.5 mg g⁻¹ oil. The percentage of the stated label amount for EPA and DHA ranged from 66 to 184% and 62 to 184%, respectively. Only 10 supplements (21% of those tested) had at least 100% of the stated label amount of EPA, while 12 supplements (25% of those tested) had at least 100% of the stated amount of DHA. Over 70% of the supplements tested did not contain the stated label amount of EPA or DHA. These results indicate that the quality of fish oil supplements is not being adequately monitored by manufacturers or government agencies and increased testing is needed to ensure regulatory compliance.

Lathosterol-to-cholesterol ratio in serum predicts cholesterol-lowering response to plant sterol consumption in a dual-center, randomized, single-blind placebo-controlled trial

Mackay, D.S., *et al.*, *Am. J. Clin. Nutr.* 101: 432–439, 2015
<http://dx.doi.org/10.3945/ajcn.114.095356>.

Benefits of plant sterols (PS) for cholesterol lowering are compromised by large variability in efficacy across individuals. High fractional cholesterol synthesis measured by deuterium incorporation has been associated with nonresponse to PS



Journal of the American Oil Chemists' Society (April)

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- Kurek K., *et al.*, Sphingolipid metabolism in colorectal adenomas varies depending on histological architecture of polyps and grade of nuclear dysplasia
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- Zheng, J.-L., Z. Luo, W. Hu, Y.-X. Pan, and M.-Q. Zhuo, Dietary fenofibrate reduces hepatic lipid deposition by regulating lipid metabolism in yellow catfish *Pelteobagrus fulvidraco* exposed to waterborne Zn
- Jun Y.-W., *et al.*, Analysis of phosphoinositide-binding properties and subcellular localization of GFP-fusion proteins

consumption; however, prospective studies that show this association have yet to be conducted. The goal was to test whether the lathosterol-to-cholesterol ratio (L:C ratio), a surrogate marker of endogenous cholesterol synthesis, serves as an a priori predictor of cholesterol lowering in response to PS consumption. Sixty-three mildly hypercholesterolemic adults who were preselected as possessing either high endogenous cholesterol synthesis [HS; $n = 24$; L:C = 2.03 ± 0.39 $\mu\text{mol}/\text{mmol}$ (mean \pm SD)] or low endogenous cholesterol synthesis (LS; $n = 39$; L:C = 0.99 ± 0.28 $\mu\text{mol}/\text{mmol}$) on the basis of baseline L:C consumed 2 g PS/d or a placebo for 28 d with the use of a dual-center, single-blind, randomized crossover design. Plasma lipid and noncholesterol sterol concentrations were measured at the end of each phase. PS consumption lowered total cholesterol (TC; -0.25 ± 0.05 mmol/L; $P < 0.0001$) and LDL cholesterol (-0.17 ± 0.04 mmol/L; $P < 0.0001$) overall. Specifically, LS individuals responded to PS treatment with a reduction in TC (-0.40 ± 0.07 mmol/L; $P < 0.0001$) and LDL cholesterol (-0.29 ± 0.05 mmol/L; $P = 0.0002$), whereas HS individuals failed to show cholesterol lowering (TC: -0.09 ± 0.09 mmol/L; $P = 0.2843$; LDL cholesterol: -0.05 ± 0.07 mmol/L; $P = 0.4917$). The odds of LS participants responding to PS consumption with cholesterol lowering better than the mean cholesterol lowering in all participants were 4.25 (95% CI: 1.242, 14.556; $P = 0.0211$) for TC and 3.36 (95% CI: 1.112, 10.161; $P = 0.0317$) for LDL cholesterol, which was higher than for HS participants. The L:C ratio predicts the extent of reduction in circulating TC and LDL cholesterol in response to PS consumption. Cholesterol synthesis assessment may thus have a use in identifying responders and nonresponders to PS therapy. This trial was registered at <https://clinicaltrials.gov/show/NCT01131832>.

n-3 Long-chain PUFAs reduce respiratory morbidity caused by iron supplementation in iron-deficient South African schoolchildren: a randomized, double-blind, placebo-controlled intervention

Malan, L., *et al.*, *Am. J. Clin. Nutr.* 101: 668–679, 2015
<http://dx.doi.org/10.3945/ajcn.113.081208>.

Although iron supplementation in malaria-free areas mostly reduces infectious morbidity, it can sometimes increase morbidity from infections as a result of the dependence of pathogenic microorganisms on iron. Supplementation with *n*-3 (ω -3) long-chain polyunsaturated fatty acids (LCPUFAs) improved morbidity in several human studies. However, information on the combined effect of iron and *n*-3 LCPUFA supplementation on infectious morbidity is limited.

We determined whether *n*-3 LCPUFAs and iron supplementation, alone or in combination, affected absenteeism and illness in iron-deficient schoolchildren with low fish intake. A

total of 321 South African children (aged 6–11 y) with iron deficiency (ID) were randomly divided into 4 groups to receive 1) iron plus placebo, 2) a mixture of docosahexaenoic acid and eicosapentaenoic acid (DHA/EPA) plus placebo, 3) iron plus DHA/EPA, or 4) placebo plus placebo as oral supplements 4 times/wk for 8.5 mo. Morbidity was recorded, and iron-status indexes were measured. The total phospholipid fatty acid composition of peripheral blood mononuclear cell membranes was analyzed in a subsample ($n = 130$). Iron supplementation increased the number of days with illness when all symptoms were considered ($B: 0.87$; 95% CI: 0.71, 1.03) as well as illness that was specifically caused by respiratory symptoms ($B: 1.45$; 95% CI: 1.21, 1.70), whereas DHA/EPA reduced the number of days with illness at school ($B: -0.96$; 95% CI: $-1.33, -0.59$). The increases caused by iron were reduced to the levels seen in the placebo plus placebo group when iron was provided in combination with DHA/EPA as indicated by significant iron \times DHA/EPA interactions (both $P < 0.001$). Iron supplementation increased morbidity (mostly respiratory) in iron-deficient South African schoolchildren with low DHA/EPA intake, but when iron was given in combination with DHA/EPA, this effect was prevented. This trial was registered at <https://clinicaltrials.gov/show/NCT01092377>.

A calorie-restriction diet supplemented with fish oil and high-protein powder is associated with reduced severity of metabolic syndrome in obese women

Su, H-Y, *Eur. J. Clin. Nutr.* 69: 322–328, 2015
<http://dx.doi.org/10.1038/ejcn>.

The prevalence of metabolic syndrome (MetS) and obesity has increased worldwide, as well as in Taiwan, particularly in women aged >40 years. The purpose of this study was to elucidate the effects of a calorie-restriction diet (CR) supplemented with protein and $n-3$ polyunsaturated fatty acids (PUFAs) on women with MetS. A total of 143 eligible female participants were recruited and assigned to four dietary interventions such as 1500-kcal CR, calorie-restriction meal-replacement diet (CRM), calorie-restriction diet with fish oil supplementation (CRF) and calorie-restriction meal-replacement diet with fish oil supplementation (CRMRF). The changes in anthropometric measures, metabolic profiles, inflammatory response and the Z-score of severity of MetS were evaluated.

Results: Among 143 female MetS patients enrolled, 136 patients completed the 12-week study. After the 12-week dietary interventions, we observed reductions in body weight (BW), body mass index (BMI) and waist circumference (WC) in all groups. BMI and triglyceride (TG) levels decreased significantly in the CRM, CRF and CRMRF groups, but not in the CR group. The homeostasis model assessment of insulin resistance (HOMA-IR) had significantly improved in all four groups, and the levels of interleukin-6 (IL-6) and C-reactive

protein (CRP) had significantly decreased in the CRF and CRMRF groups. Following the interventions, the changes in waist circumference (WC), mean arterial pressure (MAP), fasting blood glucose (FBG), TGs, HOMA-IR, CRP and IL-6 significantly correlated with the reductions in Z-score of MetS severity. Our study results indicate that a calorie-restriction dietary intervention combined with various macronutrients can reduce the severity of MetS in women and increase recovery from MetS by almost twofold in comparison with a CR alone.

Dispersive micro-solid-phase extraction of herbicides in vegetable oil with metal-organic framework MIL-101

Li, N., *et al.*, *J. Agric. Food Chem.* 63: 2154–2161, 2015
<http://dx.doi.org/10.1021/jf505760y>.

Dispersive microsolid-phase extraction based on metal-organic framework has been developed and applied to the extraction of triazine and phenylurea herbicides in vegetable oils in this work. The herbicides were directly extracted with MIL-101 from diluted vegetable oils without any further cleanup. The separation and determination of herbicides were carried out on high performance liquid chromatography. The effects of experimental parameters, including volume ratio of n -hexane to oil sample, mass of MIL-101, extraction time, centrifugation time, eluting solvent, and elution time were investigated. The Student's t test was applied to evaluate the selected experimental conditions. The limits of detection for the herbicides ranged from 0.585 to 1.04 $\mu\text{g/L}$. The recoveries of the herbicides ranged from 87.3 to 107%. Our results showed that the present method is rapid, simple, and effective for extracting herbicides in vegetable oils.

Ion mobility spectrometry versus classical physico-chemical analysis for assessing the shelf life of extra virgin olive oil according to container type and storage conditions

Garrido-Delgado, R., *et al.*, *J. Agric. Food Chem.* 63: 2179–2188, 2015, <http://dx.doi.org/10.1021/jf505415f>.

An experimental study was conducted to assess the stability of a single-variety (*Arbequina*) extra virgin olive oil (EVOO) as a function of container type and storage conditions over a period of 11 months. EVOO quality was assessed by using ion mobility spectrometry (IMS), which provides increased simplicity, expeditiousness, and relative economy. The results were compared with the ones obtained by using the official method based on classical physico-chemical analysis. Bag-in-box, metal, dark glass, clear glass, and polyethylene terephthalate containers

holding EVOO were opened on a periodic basis for sampling to simulate domestic use; in parallel, other containers were kept closed until analysis to simulate the storage conditions on market shelves. The results of the physico-chemical and instrumental analyses led to similar conclusions. Thus, samples packaged in bag-in-box containers preserved oil quality for 11 months, better than other container types. The HS-GC-IMS results confirm that 2-heptenal and 1-penten-3-one are two accurate markers of EVOO quality.

Cultivar origin and admixture detection in turkish olive oils by SNP-based CAPS assays

Uncu, A.T., *et al.*, *J. Agric. Food Chem.* 63: 2284–2295, 2015
<http://dx.doi.org/10.1021/acs.jafc.5b00090>.

The aim of this study was to establish a DNA-based identification key to ascertain the cultivar origin of Turkish monovarietal olive oils. To reach this aim, we sequenced short fragments from five olive genes for SNP (single nucleotide polymorphism) identification and developed CAPS (cleaved amplified polymorphic DNA) assays for SNPs that alter restriction enzyme recognition motifs. When applied on the oils of 17 olive cultivars, a maximum of five CAPS assays were necessary to discriminate the varietal origin of the samples. We also tested the efficiency and limit of our approach for detecting olive oil admixtures. As a result of the analysis, we were able to detect admixing down to a limit of 20%. The SNP-based CAPS assays developed in this work can be used for testing and verification of the authenticity of Turkish monovarietal olive oils, for olive tree certification, and in germplasm characterization and preservation studies.

Recovery potential of cold press byproducts obtained from the edible oil industry: physicochemical, bioactive, and antimicrobial properties

Karaman, S., *et al.*, *J. Agric. Food Chem.* 63: 2305–2313, 2015
<http://dx.doi.org/10.1021/jf504390t>.

Physicochemical, bioactive, and antimicrobial properties of different cold press edible oil byproducts (almond (AOB), walnut (WOB), pomegranate (POB), and grape (GOB)) were investigated. Oil, protein, and crude fiber content of the byproducts were found between 4.82 and 12.57%, between 9.38 and 49.05%, and between 5.87 and 45.83%, respectively. GOB had very high crude fiber content; therefore, it may have potential for use as a new dietary fiber source in the food industry. As GOB, POB, and WOB oils were rich in polyunsaturated fatty acids, AOB was rich in monounsaturated fatty acids. Oil byproducts were also found to be rich in dietary mineral contents, especially potassium, calcium, phosphorus, and magnesium. WOB had highest total phenolic (802 ppm), flavonoid

(216 ppm), and total hydrolyzed tannin (2185 ppm) contents among the other byproducts. Volatile compounds of all the byproducts are mainly composed of terpenes in concentration of approximately 95%. Limonene was the dominant volatile compound in all of the byproducts. Almond and pomegranate byproduct extracts showed antibacterial activity depending on their concentration, whereas those of walnut and grape byproducts showed no antibacterial activity against any pathogenic bacteria tested. According to the results of the present study, walnut, almond, pomegranate, and grape seed oil byproducts possess valuable properties that can be taken into consideration for improvement of nutritional and functional properties of many food products.

Ergostatrien-3 β -ol from *Antrodia camphorata* inhibits diabetes and hyperlipidemia in high-fat-diet treated mice via regulation of hepatic related genes, glucose transporter 4, and AMP-activated protein kinase phosphorylation

Kuo, Y-H., *J. Agric. Food Chem.* 63: 2479–2489, 2015
<http://dx.doi.org/10.1021/acs.jafc.5b00073>.

This study was designed to explore the effects and mechanism of ergostatrien-3 β -ol (EK100) from the submerged whole broth of *Antrodia camphorata* on diabetes and dyslipidemia in high fat diet (HFD)-fed mice for 12 weeks. The C57BL/6J mouse fed with a high fat diet (HFD) could induce insulin resistance and hyperlipidemia. After 8 week of induction, mice were receiving EK100 (at three dosages) or fenofibrate (Feno) or rosiglitazone (Rosi) or vehicle by oral gavage 4 weeks afterward. HFD-fed mice display increased blood glucose, glycated hemoglobin (HbA1c), total cholesterol (TC), triglyceride (TG), insulin, and leptin levels. These blood markers were significantly lower in EK100-treated mice, and finally ameliorated insulin resistance. EK100 treatment exhibited reduced hepatic ballooning degeneration and size of visceral adipocytes. Glucose transporter 4 (GLUT4) proteins and phosphorylation of Akt in skeletal muscle were significantly increased in EK100- and Rosi-treated mice. EK100, Feno, and Rosi treatment led to significant increases in phosphorylation of AMP-activated protein kinase (phospho-AMPK) protein in both skeletal muscle and liver. Moreover, EK100 caused a decrease in hepatic expressions of phosphoenolpyruvate carboxykinase (PEPCK) and glucose-6-phosphatase (G6 Pase), and decreased glucose production. EK100 lowered blood TG level by inhibition of hepatic fatty acid synthesis by dampening sterol response element binding protein-1c (SREBP-1c) but increasing expression of peroxisome proliferator activated receptor α (PPAR α). Moreover, EK100-treated mice reduced blood TC levels by decreased hepatic expressions of SREBP2, which plays a major role in

the regulation of cholesterol synthesis. EK100 increased high-density lipoprotein cholesterol (HDL-C) concentrations by increasing expressions of apolipoprotein A-I (apo A-I) in liver tissue. Our findings manifest that EK100 may have therapeutic potential in treating type 2 diabetes associated with hyperlipidemia in HFD-fed mice by regulation of GLUT4, PEPCK, G6 Pase, SREBP1c, SREBP2, apo A-I, and AMPK phosphorylation.

Controlled-release fertilizer prepared using a biodegradable aliphatic copolyester of poly(butylene succinate) and dimerized fatty acid

Lubkowski, K., *et al.*, *J. Agric. Food Chem.* 63: 2597–2605
<http://dx.doi.org/10.1021/acs.jafc.5b00518>.

The preparation and characterization of a controlled-release multicomponent (NPK) fertilizer with the coating layer consisting of a biodegradable copolymer of poly(butylene succinate) and a butylene ester of dilinoleic acid (PBS/DLA) is reported. The morphology and structure of the resulting polymer-coated materials and the thickness of the covering layers were examined using X-ray diffraction and scanning electron microscopy coupled with energy dispersive X-ray analysis. The mechanical properties of these materials were determined with a strength-testing machine. Nutrient release was measured in water using spectrophotometry, potentiometry, and conductivity methods. The results of the nutrient release experiments from these polymer-coated materials were compared with the requirements for controlled-release fertilizers. A conceptual model is presented describing the mechanism of nutrient release from the materials prepared in this study. This model is based on the concentrations of mineral components inside the water-penetrated fertilizer granules, the diffusion properties of the nutrients in water, and a diffusion coefficient through the polymer layer. The experimental kinetic data on nutrient release were interpreted using the sigmoidal model equation developed in this study.

Effect of *trans*, *trans* CLA egg enrichment from CLA-rich soy oil on yolk fatty acid composition, viscosity and physical properties

Shinn, S.E., *et al.*, *J. Agric. Food Chem.* 63: 2506–2513, 2015
<http://dx.doi.org/10.1021/jf504759w>.

CLA egg accumulation studies using *cis*, *trans* (*c,t*) isomers have been effective, but they reported adverse egg quality. *trans*, *trans* (*t,t*) CLA isomers have shown superior nutritional effects in rodent studies, but reports of *t,t* CLA-rich yolks are limited. The objectives were to determine the effect of *t,t* CLA-rich soy oil in feed on egg yolk viscosity, and yolk quality during refrigerated storage. Yolk fatty acids, viscosity, weight, index, moisture, pH, and vitelline membrane strength (VMS) were determined

at 0, 20, and 30 storage days. CLA had minimal effect on fatty acid profiles, relative to *c,t* reports. CLA-rich yolk viscosity was greater than controls, and CLA yolks maintained higher viscosities during storage. Yolk weight and index were not affected by *t,t* CLA-rich soy oil. Yolks with the greatest CLA concentrations had the greatest VMS after 20 days of storage, and yolks containing lower CLA levels maintained greater VMS throughout 30 days of storage, relative to controls.

Carotenoids of aleurone, germ, and endosperm fractions of barley, corn and wheat differentially inhibit oxidative stress

Masisi, K., *et al.*, *J. Agric. Food Chem.* 63: 2715–2724, 2015
<http://dx.doi.org/10.1021/jf5058606>.

The antioxidant potential of carotenoids from aleurone, germ, and endosperm fractions of barley, corn, and wheat has been evaluated. HPLC analysis confirmed the presence of lutein and zeaxanthin carotenoids (nd-15139 µg/kg) in extracts of cereal grain fractions. The antioxidant properties using 2,2-diphenyl-1-picrylhydrazyl, oxygen radical absorbance capacity, 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) assays revealed significantly higher ($P < 0.001$) antioxidant activity in the germ than in the aleurone and endosperm fractions. Using 3-(4,5-dimethylthiazolyl-2)-2,5-diphenyltetrazolium bromide (MTT) assay, 2,2'-azobis (2-amidinopropane) dihydrochloride (AAPH)-induced cell loss was effectively reduced by preincubating Caco-2, HT-29, and FHs 74 Int cells with carotenoid extracts. Moreover, carotenoid extracts reduced ($P < 0.001$) AAPH-induced intracellular oxidation in the cell lines, suggesting antioxidant activity. Of the 84 antioxidant pathway genes included in microarray array analysis (HT-29 cells), the expressions of 28 genes were enhanced ($P < 0.05$). Our findings suggest that carotenoids of germ, aleurone, and endosperm fractions improved antioxidant capacity and thus have the potential to mitigate oxidative stress.

Microbial synthesis of plant oxylipins from γ -linolenic acid through designed biotransformation pathways

Kim, S-U., *J. Agric. Food Chem.* 63: 2773–2781, 2015
<http://dx.doi.org/10.1021/jf5058843>.

Secondary metabolites of plants are often difficult to synthesize in high yields because of the large complexity of the biosynthetic pathways and challenges encountered in the functional expression of the required biosynthetic enzymes in microbial cells. In this study, the biosynthesis of plant oxylipins—a family of oxygenated unsaturated carboxylic acids—was explored to enable a high-yield production through a

designed microbial synthetic system harboring a set of microbial enzymes (i.e., fatty acid double-bond hydratases, alcohol dehydrogenases, Baeyer–Villiger monooxygenases, and esterases) to produce a variety of unsaturated carboxylic acids from γ -linolenic acid. The whole cell system of the recombinant *Escherichia coli* efficiently produced (6Z,9Z)-12-hydroxydodeca-6,9-dienoic acid (**7**), (Z)-9-hydroxynon-6-enoic acid (**15**), (Z)-dec-4-enedioic acid (**17**), and (6Z,9Z)-13-hydroxyoctadeca-6,9-dienoic acid (**2**). This study demonstrated that various secondary metabolites of plants can be produced by implementing artificial biosynthetic pathways into whole-cell biocatalysis.

Bioaccessibility and digestive stability of carotenoids in cooked eggs studied using a dynamic *in vitro* gastrointestinal model

Nimalaratne, C., *et al.*, *J. Agric. Food Chem.* 63: 2956–2962, 2015, <http://dx.doi.org/10.1021/jf505615w>.

Among dietary carotenoids, lutein and zeaxanthin are known to protect against age-related macular degeneration, a leading cause of irreversible vision loss in the elderly. Egg yolk is rich in lutein and zeaxanthin, however, the effect of cooking and gastrointestinal digestion on yolk carotenoids is poorly understood. An *in vitro* dynamic gastrointestinal model (TIM-1) was used to investigate the digestive stability and bioaccessibility of carotenoids from boiled, fried, and scrambled eggs. Bioaccessibility but not digestive stability was significantly affected by the method of cooking. The main egg carotenoids, all-*E*-lutein and all-*E*-zeaxanthin, were stable during the digestion with average recoveries of 90 and 88%, respectively. No *trans*–*cis* isomerization of carotenoids was observed during digestion. Both all-*E*-lutein and all-*E*-zeaxanthin from scrambled eggs showed significantly lower bioaccessibility compared to boiled eggs. The results indicate that the bioaccessibility of egg carotenoids can be affected by different food preparation methods

Use of various vegetable oils in designing photoprotective nanostructured formulations for UV protection and antioxidant activity

Ioana, G.B., *et al.*, *Ind. Crops Prod.* 67: 18–24, 2014 <http://dx.doi.org/10.1016/j>.

Vegetable oils are well-known for their beneficial health effects, mainly due to their antioxidant activity, which is maintained or enhanced when they are encapsulated at nanoscale. This study aims to design new nanostructured lipid carriers (NLCs) containing various vegetable oils and their combinations in order to obtain efficient formulations with UV protection performance and antioxidant activity. Pomegranate seed oil, wheat germ oil, blackcurrant seed oil, sesame seed oil, carrot root oil, raspberry



Lipids countdown continues

We are down to No. 2 on the “Fab 5” list of pivotal original papers representing the broad areas of research featured in the first 49 volumes of the AOCS journal *Lipids*. All five articles, which were chosen by Editor-in-Chief Eric J. Murphy in celebration of the journal’s 50th volume, are available for free download at <http://tinyurl.com/Lipids-Fab-5>.

This paper, which has been cited more than 1,145 times, was published in 1997 by researchers at the University of Wisconsin-Madison (USA). The study was one of the first to examine the effect of conjugated linoleic acid, or CLA, on the body composition of mice. The results, in brief: Mice fed a CLA-supplemented diet exhibited 57% and 60% lower body fat and 5% and 14% increased lean body mass relative to controls. “The effects of CLA on body composition,” the authors write, “appear to be due in part to reduced fat deposition and increased lipolysis in adipocytes, possibly coupled with enhanced fatty acid oxidation in both muscle cells and adipocytes.”

seed oil and rice bran oil were used for obtaining NLCs, as carriers for a photoprotective agent that absorbs the UVA radiation: diethylamino hydroxybenzoyl hexyl benzoate. The NLCs were characterized by mean particle size, physical stability over time, and entrapment efficiency of the UVA filter. The UV protection performance was assessed using both *in vitro* sun protection factor (SPF) and the UVA protection factor (UVAPF). The antioxidant activity was determined by chemiluminescence analysis. All vegetable oils used led to the development of appropriate NLCs, having mean particle sizes ranging between 100 nm and 145 nm and good physical stability with zeta potential values less negative than –35 mV. The UV protection factors were evaluated on the individual vegetable oils and on cream formulations based on NLCs. Out of the seven cream formulations, the best UV protection was assured by the pomegranate seed oil based cream resulting in a SPF of 4.1 and an UVAPF of 7.8. Knowing that a synergistic interaction can occur among the active compounds of different oils, new NLCs were developed by using a mixture of pomegranate seed oil and one of the other oils. The NLCs based on pomegranate seed oil combined with wheat germ oil have shown the best entrapment efficiency (70%) and photoprotection (SPF of 5.1 and a UVAPF of 9.5). All vegetable oils showed a good antioxidant activity, which was improved by their incorporation into NLCs. The results have shown that vegetable oils

Glycerolysis pretreatment of high FFA feedstocks

Joseph Valdespino

When contemplating a biodiesel manufacturing venture, the foremost concern must be securing a sustainable, long-term supply of feedstock oils. The forecasted prices of those feed oils are also of extreme importance, since feedstock cost can comprise 65% to 85% of total operating costs. Because of this, the trend in the industry has been toward producing biodiesel from less expensive, lower-grade waste oils and fats. A few examples are used cooking oils, rendered animal fats, and trap greases. These materials are also commonly called “yellow grease,” “tallow,” and “brown grease,” respectively.

The third critical element of a successful biodiesel manufacturing venture is proper process design. The aforementioned low-cost feedstock oils do indeed offer opportunity to maximize profit. However, they require additional processing, which adds to both capital and operating costs. Nevertheless, with proper process design, additional costs can be minimized

and full advantage can be taken of the substantial savings available when using these types of feedstock.

Low-grade oils and fats must be heated, dewatered, and filtered or centrifuged to remove solids debris. Such precleaning is usually done by an independent rendering service, but it can easily be integrated into a biodiesel plant design. Such integration is becoming more common, as many waste oil generators and grease collectors opt to produce biodiesel themselves.

Next, due to the harsh operating conditions that most waste oils have been subjected to, virtually all of them have decomposed to some extent and contain varied levels of free fatty acids (FFA). Yellow greases typically contain 5–15 wt. FFA. Brown grease can contain up to 90% FFA. These free fatty acids are problematic in biodiesel manufacture. During the transesterification (TE) reaction (where triglycerides are reacted with methanol to form methyl esters and glycerin), the free fatty acids react preferentially with the base catalyst (typically sodium or potassium methoxide) to form sodium or potassium soaps. Soap formation, at a minimum, results in costly product yield loss. Worse yet, if enough soaps are formed, they can cause severe emulsification of the reaction mix and complete process failure.

Therefore, the FFA in the feed oil must either be *removed* from the feed oil, or they must be *converted* to methyl esters via a separate and additional pretreatment step before admitting the feed oil to the TE reactor.

A COMPARISON OF SIX PRETREATMENT METHODS

Each of the processing methods listed in Table 1 has its advantages and disadvantages. It is beyond the scope of this report to explain each of these in detail, but here are some brief summaries.

Caustic washing (CW) and steam stripping (SS) result in removal of FFA from the feed oil.

CW is the low-tech approach and involves minimal capital and operating costs. SS involves higher capital and operating costs and is generally used in the refining of higher-margin, virgin vegetable oils for human consumption.

The general advantage of these two methods is their simplicity and efficiency. The main downside of both of these methods is that simple removal of the FFA results in costly

- Low-grade oils are increasingly being used for biodiesel production due to their low cost.
- They require additional processing, however, which can add to the capital and operating costs of a production facility.
- This article compares the advantages and disadvantages of six pretreatment approaches and explains the author's conclusion that glycerolysis is the best option.

% FFA vs. Time - 460°F vs. 350°F

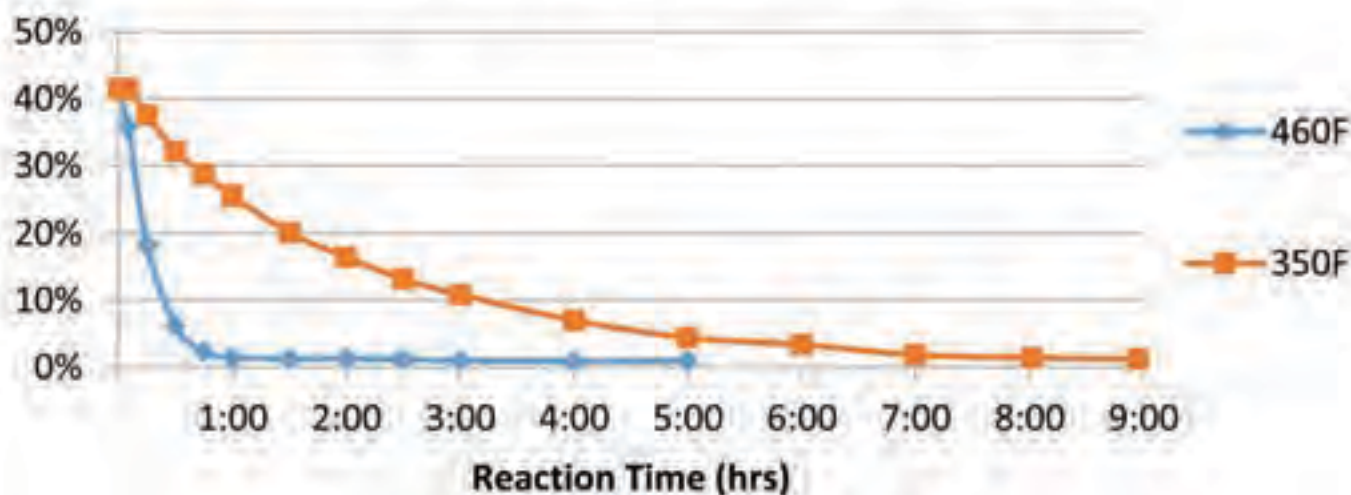


FIG. 1. Glycerolysis reaction: % FFA vs time vs temperature. The lower temperature run at 350°F shows the much slower performance that would result if 150 psi steam were used as the heating media.

biodiesel product yield loss. These two schemes also generate unnecessary waste streams that require further processing and/or disposal.

Both acid esterification (AE) and glycerol esterification (GE), also known as glycerolysis, result in conversion of the FFA in the feed oil to methyl esters, thereby boosting biodiesel product yield. Because of this fact, either of these methods is preferred over the first two methods.

Acid esterification has traditionally been the most popular approach for pretreatment of high-FFA feedstocks. AE involves reaction of the feed oil with methanol at moderate temperature (~130°F) under the influence of an acid catalyst (typically sulfuric acid). The FFA in the feed oil are converted to methyl esters while the balance of triglycerides in the oil remain unaffected. The downside of AE is that it generates water as a byproduct, which along with the acid catalyst then contaminates the excess methanol that is used. This contamination complicates recovery of that methanol for reuse.

Glycerolysis, or GE, is a relative newcomer to this industry. It has been used successfully on a commercial scale by a handful of biodiesel producers since about 2006. GE involves reaction of the feed oil with recycled glycerin at an elevated temperature (>400°F). The FFA and also the triglycerides in the oil are all converted to mono- and diglycerides. GE does not require the use of methanol, therefore there is no methanol contamination.

Enzyme catalysis (EC) and supercritical methanol (SCM) are new emerging technologies that are just recently being tried on a commercial scale. Both of these methods attempt to simultaneously esterify the FFAs in the feed oil to methyl-esters and

TABLE 1. Six approaches that have been used for pretreatment of high FFA oils.

High FFA feed oil pretreatment methods
1. caustic washing
2. steam stripping
3. acid esterification
4. glycerolysis
5. enzyme-catalyzed esterification
6. supercritical methanol esterification

also transesterify the balance of triglyceride fats in the feed to methyl-esters in one single step.

Potential drawbacks of EC are high enzyme catalyst cost and limited catalyst life. However, there have been recent reports of promising new work on recoverable polymer pellets impregnated with immobilized enzymes. Another downside of EC is that, like the AE process, it generates water as a reaction product, which then contaminates the excess methanol used and complicates recovery of that methanol for reuse.

CONTINUED ON NEXT PAGE

EC also leaves some unconverted FFA in the crude biodiesel, which requires further processing to get the biodiesel into spec.

The potential drawbacks of SCM are incomplete esterification reactions, the generation of complex and/or unpredictable byproducts, and difficult product purification. Also, the use of flammable methanol at high-temperature and pressure conditions invokes concerns about process safety and high equipment costs upon scale up.

WHY GLYCEROLYSIS IS THE BEST OPTION

After careful consideration of all of the above options, we at Superior Process Technologies (SPT) [www.superiorprocesstechnologies.com] have concluded that glycerolysis is the best method for the pretreatment of high FFA feed oils to make biodiesel. SPT recommends both GE pretreatment and Continuous Processing as standard features in all of our biodiesel process design projects. The rest of this article is devoted to explaining the GE process.

GE is an age-old chemical treatment used in the oleochemicals industry to esterify fatty acids with glycerin to form mono- and diglycerides.

A stoichiometric excess of glycerin is added to the feed oil and the mixture is vigorously agitated and heated to an elevated temperature ($> 400^{\circ}\text{F}$) at atmospheric pressure. Both the FFA in the feed oil and the balance of triglycerides are all converted to mono- and diglycerides. Any unreacted glycerin remains in the mix. When these intermediate products are transferred to the downstream TE reaction process, the mono- and diglycerides are trans-esterified to form methyl esters, just as triglycerides would be in untreated feed oil. The excess glycerin merely passes through the TE reaction unaffected and joins the new glycerin formed during TE.

Required reaction time for GE is dependent upon reaction temperature. This reaction does not require the use of methanol, nor does it require any catalyst. The water byproduct formed upon esterification of the FFA is simply vented off as steam. This GE pretreatment can be performed on fats and oils that contain any level of FFA, even up to 100%, and can reduce acid number of the feed to less than 1.0 (i.e. $< 0.5\%$ FFA), thus allowing unlimited multi-feedstock capability.

Due to the corrosive nature of fatty acids at high temperature, GE does require that the reactor be made of stainless steel.

After the TE reaction, the glycerin byproduct (plus excess glycerin from GE) is separated from the biodiesel by gravity settling and decanting (or centrifuge). A large amount of methanol and majority of the unreacted catalyst go with the glycerin phase. The catalyst dissolved in the glycerol/methanol is neutralized with a small amount of sulfuric acid, thus forming sodium sulfate salt. The methanol is then stripped off for recovery. The salts are then removed from the glycerin via filtration or distillation, and a portion of the glycerin is recycled back to GE for pretreatment of new feed oil.

Biodiesel manufacture requires the use of a lot of methanol. Methanol is used in stoichiometric excess amounts to drive the reversible transesterification reaction forward in the right direction. For any biodiesel venture to be economically

feasible, it is imperative that this excess methanol be recovered and recycled for reuse. Any contaminants accumulated in that excess methanol must be removed prior to recycle. This is especially true with regard to water. The use of wet methanol in the TE reaction causes destruction of the catalyst to form caustic (NaOH). This caustic then reacts with the triglycerides in the oil (and any FFA still present) to form soaps instead of methyl esters.

As mentioned earlier, the main advantage of Glycerolysis (GE) over Acid Esterification (AE) in the pretreatment of high FFA feed oils is that GE does not contaminate methanol with water (and acid) as does AE. Therefore, with GE, the excess methanol used in the downstream TE reaction can be drystripped from both the biodiesel and glycerin phases using simple, less expensive flash-evaporation equipment. When using AE as a pretreatment method, the methanol used in the TE reaction is dry-stripped the same as above, but then it is mixed with the wet, acidic methanol from the AE step. Now, that whole mass of wet methanol must be neutralized, and then dried using an expensive and energy-consuming fractionating distillation column under high reflux.

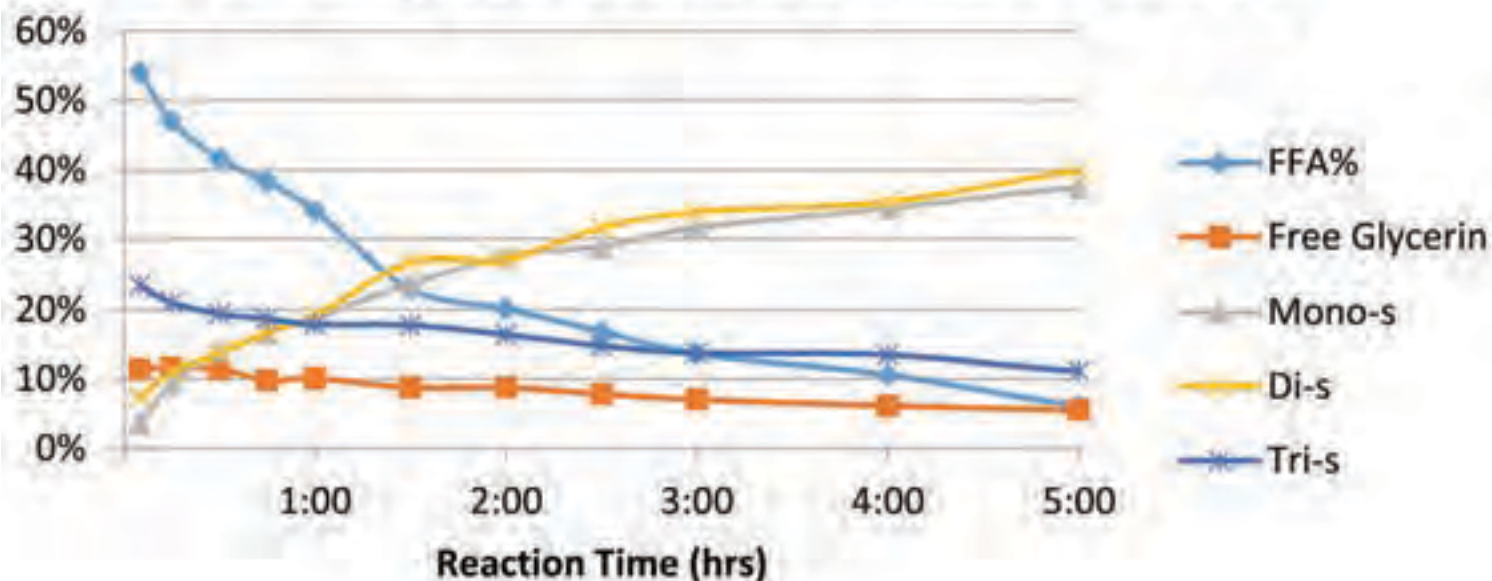
There is a common misconception that since glycerolysis requires high temperature processing, it is a more energy-intensive process than AE. Actually, the reverse is true, as long as continuous process design is used. In continuous processing, the hot product leaving the glycerolysis reactor can be used (via heat exchanger) to pre-heat the incoming feed stream so as to "economize" on heat energy. Once up and running, the net amount of added energy to merely keep the process up to temperature is minimal. In fact, due to the advantage of not having to distill wet methanol, GE actually results in around 50% reduction in overall thermal energy requirement compared to AE. Glycerolysis does, however, require the installation of a thermal-oil heater to provide this utility. Standard 150 psig steam is not hot enough to do the job. Furthermore, with proper process design, the same high temperature thermal-oil system can also service low temperature users in the plant, and the installation of a steam boiler can be avoided altogether.

Through extensive testing in our SPT laboratory, we have determined the optimal temperature for the glycerolysis reaction to be around 460°F . The graph in Fig. 1 (page 323) shows glycerolysis reaction data generated in our laboratory using feed oil with 50% FFA content (43% after dilution with glycerin). Recycled byproduct glycerin was used in these experiments. Refined USP grade glycerin can be used with similar results.

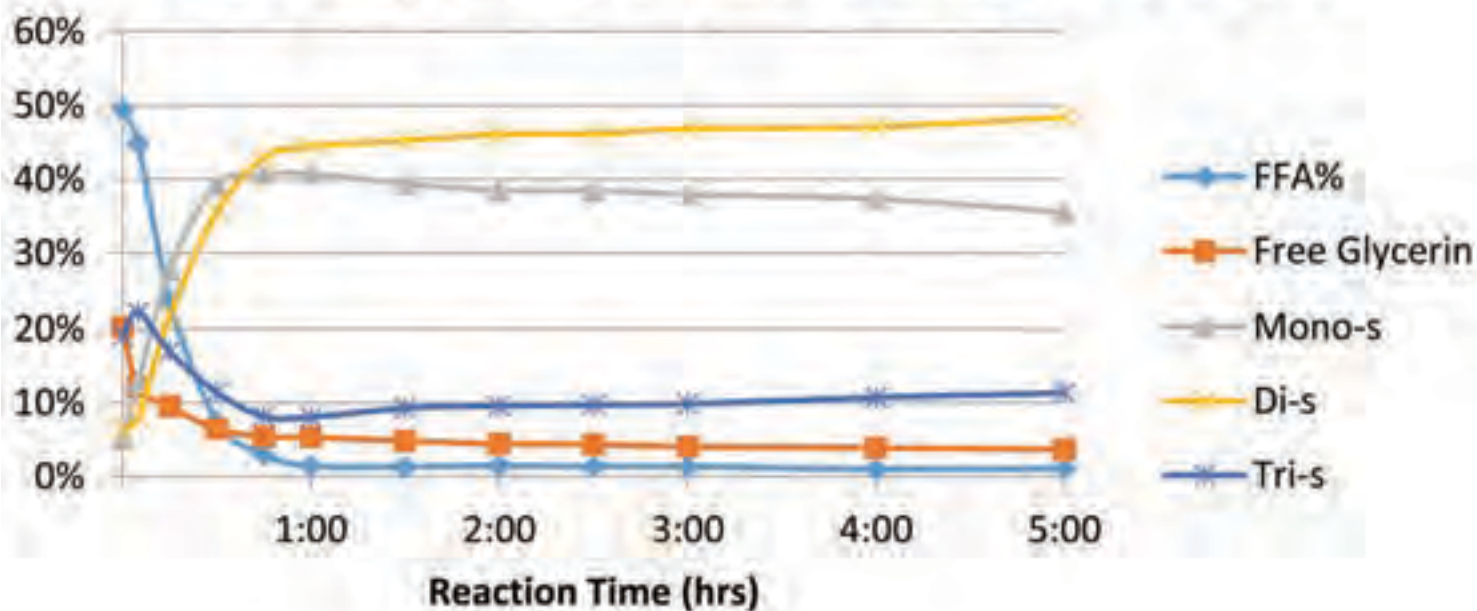
The SPT engineering design team uses chemical reaction kinetics data such as those shown in Figs. 2 and 3 to develop continuous glycerolysis process designs for commercial scale plants. If you feel that you could benefit from this technology in your next biodiesel project, please contact us.

Joseph Valdespino received a degree in Chemical Engineering (BSChE) at the University of Florida in 1974 and has worked in the oleochemicals industry for 30 years. Joseph joined the SPT design group in 2007. He can be contacted at jvaldespino@superiorpt.com.

Oil Composition Versus Time - at 350°F



Oil Composition Versus Time - at 460°F



FIGS. 2 and 3. show the relative amounts of mono-, di-, and triglyceride molecular species in the feed oil vs time throughout glycerolysis reactions at these two different temperatures (by gas chromatography (GC) analysis).

Evolving co-products from the ethanol industry

- The US ethanol industry is comprised of more than 200 ethanol plants that produced more than 13.3 billion gallons of ethanol and about 38 million metric tons (MMT) of distillers grains co-products annually in 2014 (Renewable Fuels Association, 2014, <http://www.ethanolrfa.org/>).

- Historically, the major co-products produced have been wet and dried distillers grains with solubles, wet distillers grains, and condensed distillers solubles. One metric ton of corn produces about 300 kg of CO₂, 300 kg of distillers grains (10% moisture), 417 liters of ethanol, and 0 to 13 kg of distillers corn oil (Renewable Fuels Association, 2014).

- Revenue from the sale of these co-products represented about 10% of total revenue a decade ago, but in 2014, sales value of co-products represented about 24% of total revenue. This trend is likely to continue. As a result, ethanol plants are becoming biorefineries and diversifying their portfolio of co-products to become less reliant on the volatility of ethanol profitability.

Jerry Shurson and Brian Kerr

The most significant change in ethanol co-product production over the past five years has been distillers corn oil (DCO) extraction. Only dry grind ethanol plants, which comprise about 90% of ethanol production, produce DCO. This means that about 85% of all U.S. ethanol plants are producing DCO. Distillers corn oil is being used in animal feed (43%), biodiesel (53%), and other industrial applications (4%), but cannot be used in human foods. The pending Association of American Feed Control Officials' definition specifies that DCO contain less than 1% moisture, < 2.5% unsaponifiables, < 1% insolubles, > 85% total fatty acids, and < 15% free fatty acids. Average extraction rates of DCO per MT of corn processed are 10.7 k (0.6 lbs/bu), representing more than 1 million MT of DCO production in 2014. Prices of DCO are comparable to those for yellow grease and range from \$0.30 to \$0.45/lb.

The metabolizable energy (ME) value of DCO for swine and poultry is comparable to soybean oil, and both of these sources of oil provide more energy than other vegetable oils and animal fats used in animal feeds. However, the free fatty acid content of DCO can vary from 2 to 18% among sources. Several studies have shown that increasing free fatty acid content of feed fats and oils decreases digestible energy (DE) and ME content for swine and poultry. Recent studies conducted by our group have shown this to be the case for swine,

but less so for poultry. More studies are needed to confirm these results.

REDUCED-OIL DDGS

As a result of widespread adoption of corn oil extraction in the US ethanol industry, the quantity of DDGS produced has declined proportionately, and the energy and nutrient content has become more variable. Crude fat content among DDGS sources ranges from 4.2 to 12.2%, neutral detergent fiber content ranges from 21 to 34%, and crude protein content ranges from 24 to 35%. It is important to note that oil extraction does not always result in an increase in other chemical components in DDGS among sources due to the large variability in chemical composition among sources. As a result, the ME content of reduced-oil DDGS can range from about 2,900 kcal/kg to 3,500 kcal/kg for swine. However, contrary to conventional wisdom, our research results have shown that oil content of DDGS is a poor predictor of ME content for swine and poultry. It appears that digestibility is one of the contributing factors to this poor relationship, as research evidence suggests that only about 50% of the oil in DDGS is digestible in swine. Additional research evidence has shown that fiber digestibility of DDGS in swine ranges from 23 to 55%, which also contributes to the unpredictability of ME content of reduced-oil DDGS.

To help nutritionists and the feed industry manage this variability, we have developed and validated ME prediction equations for reduced-oil DDGS for swine and poultry. These “nutritional tools” are useful to accurately determine ME content of individual sources of DDGS with variable oil content using chemical composition data. Additional studies have been conducted with swine to determine the net energy (NE) content of DDGS sources with variable oil content, as well as to develop NE prediction equations. Further research studies are being planned to validate the accuracy of these NE prediction equations in swine.

Despite the unpredictability of ME content, the addition of reduced-oil DDGS to growing-finishing pig diets has resulted in improvements in pork fat quality. One of the limiting factors to using high amounts of high-oil DDGS in growing-finishing pig diets has been that high-oil DDGS causes pork fat to become softer and more oily. This has occurred because corn oil present in DDGS is very high in polyunsaturated fatty acids (PUFA)—particularly linoleic acid (C18:2)—and the fatty acid composition of a pig’s diet affects the amount of PUFA deposited in its adipose tissue. Soft pork fat makes bacon slicing more difficult while decreasing shelf-life stability and consumer acceptance. Such problems have historically been managed by limiting the amount of high-oil DDGS in pig diets, especially during the final weeks before harvest. Today, because the oil content of DDGS has been reduced by oil extraction, acceptable pork fat quality can be achieved by using higher dietary inclusion rates and feeding DDGS diets for a longer period of time before harvest.

It appears that reducing the oil content of DDGS slightly reduces amino acid digestibility in swine. No studies have been conducted to determine if there is a similar effect in poultry.



However, if future research studies confirm that there is, amino acid digestibility will be a factor to consider when evaluating reduced-oil DDGS in swine and poultry diets relative to other competing protein ingredients such as soybean meal. Corn protein also has a poor amino acid profile relative to the amino acid requirements of swine and poultry. In particular, its lysine content is low relative to its crude protein content. This disadvantage is magnified when DDGS is produced from corn, and the heating processes used to dry DDGS further reduces its amino digestibility to that of corn and soybean meal. The use of economically available crystalline amino acids, however, helps alleviate this concern.

FUTURE CO-PRODUCTS

Numerous technologies are being evaluated and are beginning to be implemented in ethanol plants to produce new corn co-products. The overall trend of these technologies is to produce higher protein, higher fiber, and lower-oil co-products.

CONTINUED ON NEXT PAGE



High-protein DDGS

Technologies are being evaluated to increase ethanol production within existing ethanol plants and also to produce a DDGS co-product that is higher in protein (42 to 50%). While the higher protein content of these co-products may be attractive to DDGS and feed ingredient marketers, corn protein is relatively low in several essential amino acids such as lysine, tryptophan, and arginine relative to soybean meal as well as the requirements for swine and poultry. This, coupled with low oil

and high fiber content, will likely lead to their use primarily in dairy and beef cattle feeds, where more feeding value can be captured. On the other hand, technology exists for the fiber to be further separated and used in cellulosic ethanol production. If significant adoption of this technology occurs, the feeding value of high-protein, low-fiber, low-oil DDGS will significantly change and affect its relative feeding value for various species.

High-protein—yeast-based co-products

Additional technologies are being tested and implemented in a few ethanol plants to produce a high-protein (50%) co-product with a more suitable amino acid profile for pigs and poultry. These co-products derive a significant amount of their protein and amino acids from yeast used during the ethanol fermentation process. While many of the essential amino acids (i.e. lysine) are lower than soybean meal, these co-products are very attractive protein sources in swine and poultry diets. Results from recent studies have shown that the ME content of yeast co-products (*Sacchromyces cerevisiae* and *P. stipidas*) have greater ME content than soybean meal for swine. A high ME content, along with favorable amino acid profiles, digestibility, and content, as well as phosphorus content and digestibility, make these new co-products highly interesting to swine and poultry nutritionists.

Isobutanol co-products

The implementation of isobutanol technology will significantly affect the nutritional composition of co-products. No studies have been published on the types, composition, and feeding value of this technology's co-products, but such studies are needed to determine their optimal use for various food animal species.

Microalgae

Microalgae production for use in biofuels has been a popular topic and research area for several years. Limited amounts of de-oiled microalgae have been evaluated for use in animal feeds with positive results. There are numerous strains of microalgae that vary in crude protein (45 to 65%), lipids (5 to 10%), and lysine (4.6 to 7.0%) content. In addition to the high protein and amino acid content of microalgae, some strains have the ability to produce and store significant amounts of essential long-chain fatty acids, such as eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), which are required in relatively large amounts in fish feeds. Therefore, aquaculture nutritionists are keenly interested in the potential to use microalgae co-products as partial or complete replacements for fish meal in aquaculture feeds.



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Jerry Shurson is a professor in the Department of Animal Science at the University of Minnesota, St. Paul, Minnesota, USA. He can be contacted at shurs001@umn.edu.

Brian Kerr is lead scientist at the US Department of Agriculture—Agricultural Research Service, National Laboratory for Agriculture and the Environment in Ames, Iowa, USA. He can be contacted at Brian.Kerr@ARS.USDA.GOV.

Algae secrets:

How to harvest microalgae for applications worldwide

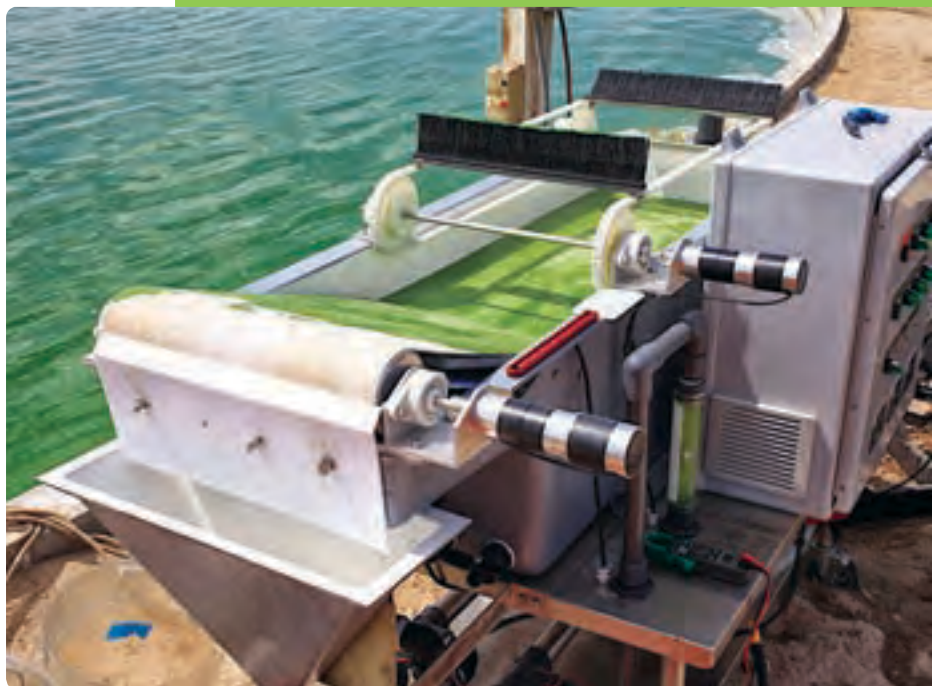
Riggs Eckelberry

With their omega-3 rich fatty acids, algae act as a feedstock for a variety of seemingly disparate industries, from food to cosmetics to dietary supplements. While algae-based bio-fuels are just starting to get off the ground, immediate applications are virtually limitless with the right growth strategies and harvesting technologies in place. It should come as no surprise, then, that in the United States alone, more than 50 research institutions and 100 companies are working on algae technology across the value chain.

The aquaculture feed industry, for one, is ripe for disruption from algae solutions. This \$100 billion sector is expected to increase by 33% between 2012 and 2022, compared to an increase of only 3% in capture fisheries, according to the United Nations report, *The State of World Fisheries* (<http://tinyurl.com/bg9mscy>). Today, feed costs represent close to 50% of fish farmers' operating costs (*ReportLinker*, <http://tinyurl.com/mcxd57r>), but by using algae as the feedstock for fish feed, farmers can reduce this expense by more than a third.

Wastewater cleanup, a growing global challenge, is another industry for which algae are as a potential solution. Studies by the

- Algae have great potential as a feedstock for a wide range of industries.
- Unfortunately, traditional harvesting methods are expensive and do not produce algae in high enough yields.
- The following article describes a new harvesting technology that uses electric pulses to remove solids, de-water, and concentrate microalgae at the commercial scale.



OriginOil's EWS Algae technology supports New Global Energy's tilapia farming operations in Southern California.

CONTINUED ON NEXT PAGE



OriginOil CEO Riggs Eckelberry at the company's EWS Algae showcase at Aqua Farming Tech in California's Coachella Valley.

US Environmental Protection Agency (<http://tinyurl.com/mtnruns>) show that water and wastewater infrastructure capital investment requirements come close to \$1 trillion over the next 20 years in the United States alone. In South Asia, more than \$83 billion over the next 10 years is required to meet water supply, sanitation, and solid waste development goals, according to the World Bank (<http://tinyurl.com/mjm79lz>). Growing algae in wastewater supports the sector in two ways: It naturally consumes contaminants in the water, and it can also be removed for use in downstream products such as fuel and chemicals. Algae can improve the economics and environmental impact of wastewater treatment.

ALGAE HARVESTING

Harvesting algae is much like panning for gold. While the prospects of the algae industry seem lucrative and abundant, it is actually quite difficult to scale algae harvesting operations. Traditional methods to harvest algae in high concentrations—like centrifugal force and chemical approaches—are both time and energy intensive. These methods usually cause damage to the algal cell wall, reduced shelf life, and bacterial infestation. Chemical treatment leaves residue in the water stream, requiring post-process treatment, while centrifugal separators are limited to a batch production approach, adding considerable cost and limiting the scale-up of algae harvesting operations.

Almost all applications of microalgae require highly concentrated strains with a long shelf life. In many cases, upstream producers need to transport and ship algae to separate processing facilities; without a long-shelf life, this is next to impossible. Most microalgae quickly degrade, some species in a matter of hours—not nearly enough time to transport algae a meaningful distance. Additionally, it is easier to ship, store, and package highly concentrated algae. Increasing microalgae concentration

from 0.1% (the usual concentration for most autotrophic species) to 10% divides the logistics cost by a factor of 100.

A NEW APPROACH

To successfully address the core challenges associated with algae harvesting, OriginOil has developed a low-energy, primarily non-chemical harvesting technology called Electro Water Separation for Algae (EWS Algae). EWS Algae uses electric pulses to remove solids, de-water and concentrate micro-algae at the commercial scale. The technology makes use of a two-stage process: The first uses electro-coagulation to concentrate algae; in the second stage, a cloud of micro-bubbles is released via specifically engineered electrodes and lifts the algae to the surface of the water, where it is raked off as an intact, bacteria-free concentrate with improved shelf life. This process can run continuously, driving efficiency in the harvesting process.

Beyond the efficiencies inherent in OriginOil's low-energy process, the technology can also improve industrial facilities' environmental impact. EWS is capable of removing all suspended solids found in water, including contaminants like oils, bacteria, and other organic matter, providing an increased benefit for algae growers that need to treat wastewater.

OriginOil has deployed its EWS system to licensing partners worldwide. For example, environmental engineering firm Ennesys implemented OriginOil's EWS technology in a commercial demonstration setting outside Paris. At the La Defense complex, a model of sustainable building techniques, EWS is paired with the Ennesys system to sanitize liquid sewage, turn the urea in the water into nitrates to feed algae, and then to harvest the algae for energy to cool the building.

For aquaculture farmers, OriginOil's EWS technology also has demonstrated its ability to harvest algae from water for a nutrient-rich fish feed substitute. Algalosol, a leader in large-scale algae production, is integrating OriginOil's system at a facility in Bangladesh to demonstrate microalgae production for fish feed. The 6.5-acre site will produce approximately 25 tons of microalgae per month. For partner Catalina Sea Ranch, the first offshore shellfish ranch in US federal waters, OriginOil's EWS provides a dual benefit for its hatchery. The system harvests algae and also removes dissolved nitrogenous waste and contaminants, including bacteria and heavy materials.

In Japan, OriginOil's EWS technology has been implemented by eco-solutions provider Orca Vision at a vertical farming pilot that includes both vegetable and algae production. Orca Vision's urban farms are designed to use hydroponics to deliver high quality and nutrient-rich vegetables through an innovative vertical design that reduces distribution costs as farms can be located near customers. Without OriginOil's low-energy technology, Orca Vision would not be able to cost-effectively harvest the algae at the vertical farms.

Algae are incredible organisms. They're one of the fastest growing plants in the world and have a ferocious appetite for nutrient-rich waste. With that in mind, it's no wonder the US federal government and private investors have injected more than \$1 billion into the domestic industry (Algal Biomass Organization). In partnership with Idaho National Laboratory, OriginOil is currently researching alternative pathways to overcome the two key barriers to commercializing algae biofuels: high cost of production, and low yield of target biofuel and bioproduct feedstock. Continued innovation in the algae field will open new opportunities for companies across the algae supply chain.

Riggs Eckelberry is an experienced technology entrepreneur and the CEO of OriginOil. He serves on the Advisory Board of the National Algae Association and was recently named one of the Top 125 People In the Bioeconomy. He can be contacted at sales@originoil.com.



OriginOil's EWS Algae Model A12, which enables algae harvesting at 12 liters per minute.

Patents (cont. from 315)

tracer can also be present with a separate antioxidant or the tracer compound can be the salt of a C6-C22 unsaturated carboxylic acid. The rare earth metal compound tracer is released when the prosthetic is worn down or otherwise degraded in the mammalian body in which it was implanted. The presence and amount of released tracer present in a body fluid or tissue sample measured and is proportional to the degree of degradation of the implant.

Process for the distillation of fatty acid esters

Horlacher, P. and D. Hietsch, Cognis IP Management GmbH, US8889895, November 18, 2014

The present invention relates to a method for working up a mixture which contains esters of EPA and/or DHA with monohydric alcohols having 1 to 6 carbon atoms, esters of other fatty acids with monohydric alcohols having 1 to 6 carbon atoms, and free cholesterol, wherein the method comprises adding a transesterification catalyst to the mixture, converting at least some of the free cholesterol into esterified cholesterol and thereafter distilling the mixture, wherein the distillation is carried out in such a manner that a product is obtained which contains EPA and/or DHA, both in the form of esters thereof with monohydric alcohols having 1 to

6 carbon atoms, in a higher relative amount, based on all fatty acids in free or bound form present in the product, than said mixture. In addition, the present invention relates to a product which is obtainable by the method according to the invention, wherein the product contains 10 to 99.99% bwt of esters of EPA and/or of DHA with monohydric alcohols having 1 to 6 carbon atoms and 0.0001 to a maximum of 0.3% bwt weight of cholesterol in free or bound form.

Environmentally benign anti-icing or deicing fluids

Sapientza, R., et al., MLI Associates LLC, US8906251, December 9, 2014

Deicing compositions comprised of glycerol-containing by-products of triglyceride processing processes are disclosed.

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.



Processing advances for surfactants manufacturing

Icilio Adami and Emanuele Caimi

Anionic surfactants are by far the most important category when it comes to traditional surfactant applications—both in terms of production and use, as well as the broad range of applications they support.

ANIONIC SURFACTANTS: FROM SPECIALTIES TO COMMODITIES... AND BACK?

The anionic surfactants used in household, personal care, and industrial/institutional applications account for about 70% of

- Surfactants manufacturing is becoming increasingly challenging as more and more customers request highly specialized molecules with superior performance.
- Meanwhile, new applications for anionic, nonionic, and cationic surfactants offer fresh opportunities in high-value sectors that demand flexible, sustainable, and environmental friendly solutions.
- Reliable and cost-effective hardware solutions can help surfactant producers meet these challenges and opportunities. In this article, two professionals at Desmet Ballestra—a company with 55 years of experience developing advanced and effective technologies for multinational, private, and public companies engaged in surfactants production worldwide—share their perspective on the latest market trends and related manufacturing advances.

the global surfactants market, while those used in household and personal care products constitute about 60% of the surfactant demand. The percentage that anionic use in household and personal care products contributes to the overall surfactant demand is much higher today than it was a decade ago thanks to the progressive development of anionic surfactants derived by natural sources, mainly palm and coconut oil derivatives. Sulphated fatty alcohol, ethoxysulphated fatty alcohol, and sulpho-fatty acid methylester productions have become viable alternatives to the classical petrochemical-derived homologues.

Nevertheless, the petrochemical route to surfactants hasn't lost attractiveness, as anionic surfactants with applications and expected performances that are different from those needed in household and personal care products continue to be developed and produced. For example, anionic surfactants characterized by high molecular weight and obtained by sulphonation of feedstocks like mono-alkylates with C_{18} - C_{20} alkyl chains, long-chain olefins, and by sulphation of etho-propoxylates based on alkylphenols or fatty alcohols are finding applications in enhanced oil recovery (EOR) and in special processes where lubrication and polymerization are the key.

Increasing profitability and investments in anionic surfactants have led to major improvements in production technology. This has enabled the industry to establish high-quality standards coupled with optimized energy demand. The quality specifications that have been achieved for the main types of anionics are shown in Fig.1; they reflect the implementation of important improvements and innovations in the sulphonation/sulphation process.

For example, the effects of simultaneous neutralization, stripping, and de-aeration provided by vacuum-type neutralization make it possible to produce high-quality, dioxane-free ethoxysulphates suitable for personal care applications such as shampoos, bath foams, and many cosmetic products.

In contrast, the new exhaust gas treatment, which is based on the reaction between fresh organic and unconverted SO_3 , allows the acidic drippings that are traditionally generated by the exhaust gas cleaning section to be eliminated. This reduces the environmental impact. Finally, the heat recovery system reduces the energy demands of the whole sulphonation process, thus improving the overall sustainability of the process.

Because the quality of anionic surfactants is the key factor in their commercialization and use, it is important that the specific production technology be continuously committed to further improving the "cost vs benefits" ratio of the anionic surfactants it supports.

CONTINUED ON PAGE 334

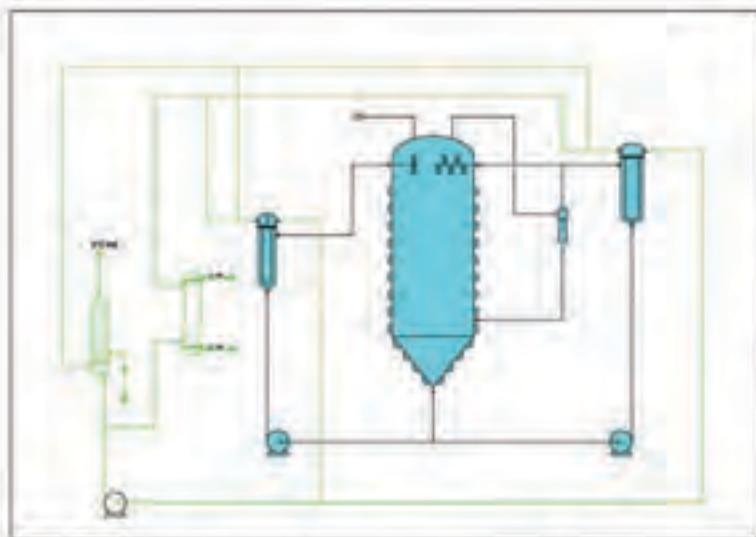


FIG. 1. Schematic diagram of an enhanced loop reactor.

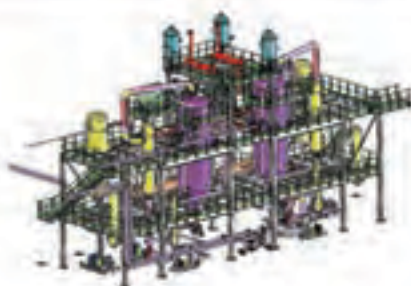


TABLE 1. Quality standard improvement in anionic surfactant production

PRODUCT	ACTIVE MATTER (% wt)	FREE OIL (on 100% A.M.)	Na ₂ SO ₄ (on 100% A.M.)	KLETT COLOUR (on 5% A.M. solution)
LABSO ₃ H	97.5 - 97.7	0.8 - 1.0	0.5 - 0.6 (♦)	10 - 20
FAS (C ₁₂ -C ₁₄ nat.)	70 - 75	0.8 - 1.0	0.8 - 1.2	5 - 10
FAS (C ₁₃ -C ₁₅ synt.)	70 - 73	1.0 - 1.2	0.9 - 1.3	15 - 25
FAS (C ₁₆ -C ₁₈ nat.)	68 - 70	1.5 - 1.8	1.2 - 1.7	35 - 50
AES-2EO (*)	70 - 73	0.5 - 1.0	0.6 - 1.1	3 - 10
AES-3EO (**)	70 - 73	0.8 - 1.0	0.6 - 1.0	5 - 15
AOS (C ₁₄₋₁₆ / C ₁₄₋₁₈)	70 - 75	1.0 - 1.6	1.0 - 1.5	25 - 40
MES (C ₁₂₋₁₄ / C ₁₅₋₁₈)	68 - 70 (***)	0.8 - 1.5	1.2 - 1.5	20 - 30 (****)

(♦) : As H₂SO₄ content

(*) : 1,4 Dioxane content of max. 30 ppm (on AM basis) with FO = 2% max. (on AM)

(**) : 1,4 Dioxane content of max. 30 ppm (on AM basis) with FO = 2,5% max. (on AM)

(***) : Di-salt content = 5% max. (on tot. AM)

(****) : Color figures after bleaching

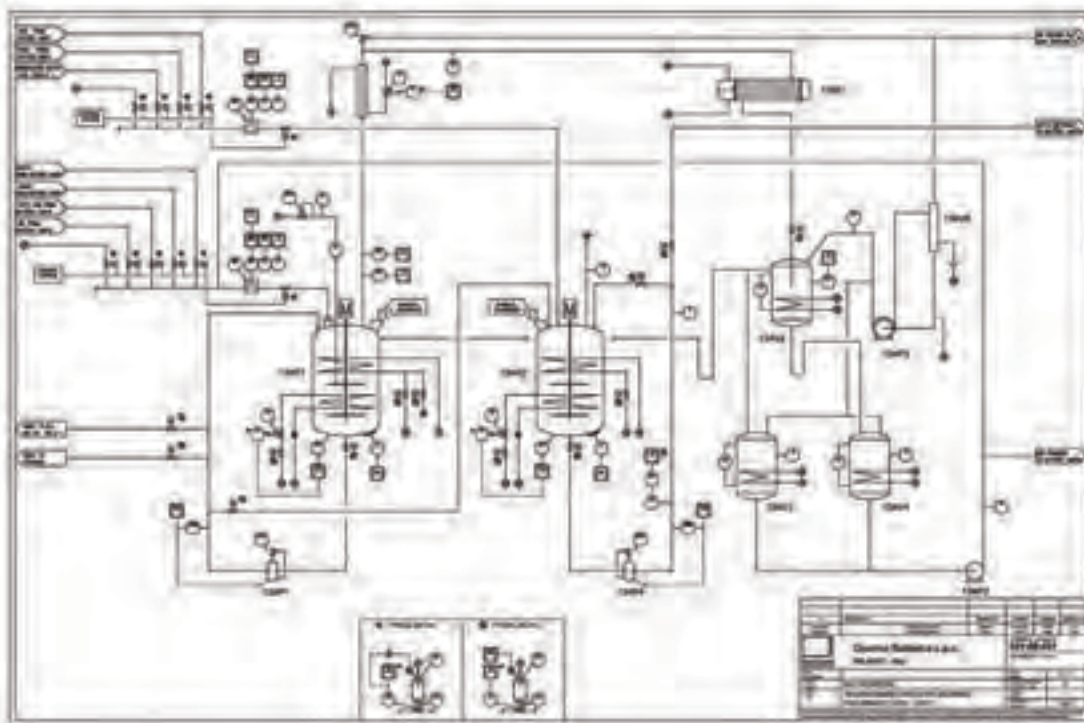


FIG. 2. A plant for cationic and amphoteric production

NONIONIC SURFACTANTS: FROM STAND-INS TO LEADING ACTORS

Nonionic surfactants, which can be synthesized in a wide variety of molecular structures, are the category that has enjoyed the largest increase in use and production. In addition to their use in laundry detergents, cosmetics, and personal care formulations, nonionics are used on a massive scale in industrial applications such as food processing, textile treatments, lubricants, rubber and elastomer production, paper working, and many other niches where their specificities are highly appreciated.

Hundreds of nonionic surfactants can be categorized under the very generic name of ethoxylates, and their manufacturing is chemically complex and highly regulated due to the use of hazardous chemicals such as ethylene oxide (EO).

Ethoxylates are the result of EO condensation with organic precursors such as fatty alcohols, esters, and glycols; their production is based on batch processes that are characterized by the highest safety levels and designed to offer maximum flexibility and energy optimization.

The new enhanced loop reactor (ELR) is the last generation of “alkoxylation” reactor, capable of coupling the highest operational safety level with full flexibility and the most advanced control of the reaction parameters (Fig. 1, page 333).

The ELR shows its versatility when producing ethoxylates characterized by a growth ratio—the volume ratio between the final product and its precursor—of up to 70, thanks to the ELR’s unique design and the double-effect gas-liquid contact system. This latter system represents a step change in process intensification, as reactions occurring in both liquid and gaseous phases shorten batch time-length. Moreover, the reactor is designed and featured as “intrinsically safe,” ensuring its mechanical integrity even in the case of an accidentally primed explosion.

The ELR is the most advanced system for the production of high molecular-weight alkoxyates. It is therefore the best choice for the production of high-molecular-weight and combined ethopropoxyates, which are experiencing significant growth in applications such as EOR.

CATIONIC AND AMPHOTERIC SURFACTANTS: THE EVERLASTING PROMISES

Cationic and amphoteric surfactants are characterized by a wide typology of molecules and functional groups. These ensure their surfactant properties, but so far their production and use has been limited.

The specific production technology for these surfactants is typically based on batch “multipurpose” plants in which the several reactions involved in the synthesis of both cationics and amphoteric can be conveniently accomplished, limiting the need for huge investments in production hardware.

Operational flexibility and full control of the batch sequences are the winning characteristics of the “multipurpose” batch plant (Fig. 2) in which fatty acids, esters, oils and tertiary alkyl-amines are reacted with alkylating agents to yield quaternary cationic surfactants as well as amphoteric surfactants such as fatty amine propionates, betaines, amido-alkyl betaines and imidazoline derivatives.

Ilcilio Adami is the R&D technologies director of Desmet Ballestra. He has more than 35 years’ experience in the surfactants and detergents field. Adami can be contacted at ilcilio.adami@ballestra.com. Emanuele Caimi is the sales area manager of Desmet Ballestra for North and South America projects. He can be contacted at Emanuele.Caimi@ballestra.com.

Extracts & Distillates (cont. from 321)

and especially their combinations can be used as renewable raw materials in designing effective and eco-friendly photoprotective nanostructured formulations.

Oxyphytosterols as active ingredients in wheat bran suppress human colon cancer cell growth: identification, chemical synthesis, and biological evaluation

Zhu, Y., *et al.*, *J. Agric. Food Chem.* 63: 2264–2276, 2015
<http://dx.doi.org/10.1021/jf506361r>.

Consumption of whole grains has been reported to be associated with a lower risk of colorectal cancer. Recent studies illustrated that phytochemicals in wheat bran (WB) may protect against colorectal cancer. There is a growing interest in the phytosterol contents of foods as either intrinsic or added components due to their beneficial health effects. However, little is known whether phytosterols in WB contribute the observed chemopreventative activity of the grain. In the present study, we directly purified and identified four oxyphytosterols 1–4 from sterol-enriched fraction of WB, and also successfully synthesized five sterol oxides 5–8 and 13. Using these nine compounds as references, we outlined a comprehensive profile of steroids in WB using tandem liquid chromatography mass spectrometry with electrospray ionization (LC-ESI/MSⁿ, $n = 2–3$) techniques for the first time. Among them, three sterol oxides 13, 14, and 18 are novel compounds, and 14 compounds 3, 4, 6–11, 13, 14, 16, and 18–20 were reported in WB for the first time. Our results on the inhibitory effects of available sterol oxides 1–8 and 13 against the growth of human colon cancer cells HCT-116 and HT-29 showed that compounds 2–8 exerted significant antiproliferative effects, with oxysterol 8 being the most active one in both cells. We further demonstrated that four most active sterol oxides 5–8 could induce cell death through the apoptosis pathway. Our results showed that phytosterols, particularly oxyphytosterols, in WB possess significant antiproliferative properties, and thereby may greatly contribute the observed chemoprevention of the whole grain wheat.

Bioavailability of long-chain *n*-3 fatty acids from enriched meals and from microencapsulated powder

Hinriksdottir, H.H., *Eur. J. Clin. Nutr.* 69: 344–348, 2015
<http://dx.doi.org/10.1038/ejcn.2014.250>.

Despite the potential benefits of long-chain *n*-3 polyunsaturated fatty acids (LC *n*-3 PUFAs), intake is often low because

of low consumption of oily seafood. Microencapsulated fish oil powder can improve tolerance and acceptance of LC *n*-3 PUFAs. Bioavailability is important to achieve efficacy. We investigated the bioavailability of LC *n*-3 PUFAs from microencapsulated powder in comparison with meals enriched with liquid fish oil. Participants ($N=99$, age ≥ 50 years) of this 4-week double-blinded dietary intervention were randomized into three groups. Group 1 ($n=38$) received 1.5 g/d eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) as ready-to-eat meals enriched with liquid fish oil; group 2 ($n=30$) received the same amount of these LC *n*-3 PUFAs as microencapsulated fish oil powder and regular meals; and group 3 ($n=31$) was the control group, which received placebo powder and regular meals. Blood samples were taken from fingertips at baseline and at the end point. Seventy-seven subjects (77.8%) completed the study. The amount of EPA in blood doubled in both groups that received LC *n*-3 PUFAs ($P<0.05$), but it did not change in the control group. The changes in DHA were less but still significant in both intervention groups. According to multivariate analysis, both intervention groups had higher end-point LC *n*-3 PUFA concentrations compared with placebo, but differences between intervention groups were not significant. Bioavailability of LC *n*-3 PUFAs in encapsulated powder is very similar to the bioavailability of LC *n*-3 PUFAs in ready-to-eat meals enriched with liquid fish oil. Thus, encapsulated powder can be considered useful to increase LC *n*-3 PUFA concentrations in blood.

Cholesterol-lowering activity of sesamin is associated with down-regulation on genes of sterol transporters involved in cholesterol absorption

Liang, Y.T., *et al.*, *J. Agric. Food Chem.* 63: 2963–2969, 2015,
<http://dx.doi.org/10.1021/jf5063606>.

Sesame seed is rich in sesamin. The present study was to (i) investigate the plasma cholesterol-lowering activity of dietary sesamin and (ii) examine the interaction of dietary sesamin with the gene expression of sterol transporters, enzymes, receptors, and proteins involved in cholesterol metabolism. Thirty hamsters were divided into three groups fed the control diet (CON) or one of two experimental diets containing 0.2% (SL) and 0.5% (SH) sesamin, respectively, for 6 weeks. Plasma total cholesterol (TC) levels in hamsters given the CON, SL, and SH diets were 6.62 ± 0.40 , 5.32 ± 0.40 , and 5.00 ± 0.44 mmol/L, respectively, indicating dietary sesamin could reduce plasma TC in a dose-dependent manner. Similarly, the excretion of total fecal neutral sterols was dose-dependently increased with the amounts of sesamin in diets (CON, 2.65 ± 0.57 ; SL, 4.30 ± 0.65 ; and SH, 5.84 ± 1.27 μ mol/day). Addition of sesamin into diets was associated with down-regulation of mRNA of intestinal Niemann–Pick C1 like 1 protein (NPC1L1),

CONTINUED ON NEXT PAGE

acyl-CoA:cholesterol acyltransferase 2 (ACAT2), microsomal triacylglycerol transport protein (MTP), and ATP-binding cassette transporters subfamily G members 5 and 8 (ABCG5 and ABCG8). Results also showed that dietary sesamin could up-regulate hepatic cholesterol-7 α -hydroxylase (CYP7A1), whereas it down-regulated hepatic 3-hydroxy-3-methyl-glutaryl-CoA (HMG-CoA) reductase and liver X receptor alpha (LXR α). It was concluded that the cholesterol-lowering activity of sesamin was mediated by promoting the fecal excretion of sterols and modulating the genes involved in cholesterol absorption and metabolism.

Feeding laying hens stearidonic acid-enriched soybean oil, as compared to flaxseed oil, more efficiently enriches eggs with very long-chain *n*-3 polyunsaturated fatty acids

Elkin, R.G., *J. Agric. Food Chem.* 63: 2789–2797, 2015
<http://dx.doi.org/10.1021/jf505185u>.

The desaturation of α -linolenic acid (ALA) to stearidonic acid (SDA) is considered to be rate-limiting for the hepatic conversion of ALA to eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) in humans, rodents, and chickens. Thus, we hypothesized that feeding laying hens SDA, as a component of the oil derived from the genetic modification of the soybean, would bypass this inefficient metabolic step and result in the enrichment of eggs with EPA and DHA at amounts comparable to that achieved by direct supplementation of hens' diet with these very long-chain (VLC) *n*-3 polyunsaturated fatty acids (PUFAs). In a 28-d study, laying hens incorporated 0.132 mg, 0.041 mg, or 0.075 mg of VLC *n*-3 PUFAs into egg yolk for each milligram of ingested dietary ALA derived primarily from conventional soybean oil (CON), dietary ALA derived primarily from flaxseed oil (FLAX), or dietary SDA derived from SDA-enriched soybean oil, respectively. Moreover, the amounts of total yolk VLC *n*-3 PUFAs in eggs from hens fed the CON (51 mg), FLAX (91 mg), or SDA (125 mg) oils were markedly less than the 305 mg found in eggs from fish oil-fed

hens. Unexpectedly, SDA appeared to be more readily incorporated into adipose tissue than into egg yolk. Since egg yolk FAs typically reflect the hens' dietary pattern, these tissue-specific differences suggest the existence of an alternate pathway for the hepatic secretion and transport of SDA in the laying hen.

Comparison and characterization of soybean and sunflower lecithins used for chocolate production by high-performance thin-layer chromatography with fluorescence detection and electrospray mass spectrometry

Krüger, S., *et al.*, *J. Agric. Food Chem.* 63: 2893–2901
<http://dx.doi.org/10.1021/jf506332f>.

The scarce availability of nongenetically modified soybeans on the world market represents a growing problem for food manufacturers. Hence, in this study the effects of substituting soybean with sunflower lecithin were investigated with regard to chocolate production. The glycerophospholipid pattern of the different lecithin samples was investigated by high-performance thin-layer chromatography fluorescence detection (HPTLC-FLD) and by HPTLC-positive ion electrospray ionization mass spectrometry (ESI⁺-MS) via the TLC-MS Interface and by scanning HPTLC-matrix-assisted laser desorption ionization-time-of-flight mass spectrometry (MALDI-TOFMS). Especially, the contents of phosphatidylcholine (PC) and phosphatidylethanolamine (PE) were of interest due to the influencing effects of these two glycerophospholipids on the rheological parameters of chocolate production. The lecithin substitution led to only slight differences in the rheological parameters of milk and dark chocolate. Limits of detection (LODs) and limits of quantification (LOQs) of seven glycerophospholipids were studied for three detection modes. Mean LODs ranged from 8 to 40 mg/kg for HPTLC-FLD and, using a single-quadrupole MS, from 10 to 280 mg/kg for HPTLC-ESI⁺-MS as well as from 15 to 310 mg/kg for HPTLC-FLD-ESI⁺-MS recorded after derivatization with the primuline reagent. ■

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