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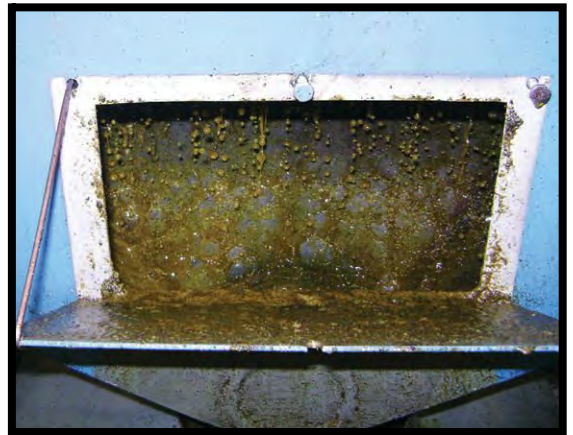
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To be a global forum to promote the exchange of ideas, information, and experience, to enhance personal excellence, and to provide high standards of quality among those with a professional interest in the science and technology of fats, oils, surfactants, and related materials.

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## The science behind optimal frying

Understanding the frying process can lead to better food and fat quality, a higher degree of control over changing oil chemistry, and improved profitability. Laboratory studies shed new light on the changes that occur in food and oil during different types of frying.

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## November

November 1–2, 2012. 2nd ICIS Asian Surfactants Conference, Marina Bay Sands, Singapore. Information: [www.icisconference.com](http://www.icisconference.com)November 4–8, 2012. 32nd Practical Short Course on Vegetable Oil Extraction, Texas A&M University, Food Protein R&D Center, College Station, Texas, USA. Information: Rich Clough, phone: +1 979-862-2262; fax: +1 979-845-2744; email: [rclough@tamu.edu](mailto:rclough@tamu.edu); [www.foodprotein.tamu.edu](http://www.foodprotein.tamu.edu)November 5–9, 2012. WMF meets IUPAC: 7th Conference of The World Mycotoxin Forum and the XIIIth IUPAC International Symposium on Mycotoxins and Phycotoxins, Rotterdam, Netherlands. Information: [www.wmfmeetsiupac.org](http://www.wmfmeetsiupac.org)November 6, 2012. Foundation Certificate in Sensory Principles, Leatherhead, Surrey, UK. Information: [www.leatherheadfood.com/training-and-conferences](http://www.leatherheadfood.com/training-and-conferences)November 7–8, 2012. MassSpec 2012: New Horizons in MS Hyphenated Techniques and Analyses, Biollis, Singapore. Information: [www.sepscience.com](http://www.sepscience.com)November 11–13, 2012. Practical Short Course on Biodiesel/Biofuel from Algae and Other Feedstocks, Texas A&M University, College Station, Texas, USA. Information: [www.foodprotein.tamu.edu](http://www.foodprotein.tamu.edu)November 11–15, 2012. SETAC North America, Long Beach, California, USA. Information: [www.setac.org](http://www.setac.org)November 13–14, 2012. Predicting and Controlling the Shelf-Life of Foods, Leatherhead, Surrey, UK. Information: [www.leatherheadfood.com/training-and-conferences](http://www.leatherheadfood.com/training-and-conferences)November 13–15, 2012. Health Ingredients Europe and Natural Ingredients, Messe Frankfurt, Germany. Information: [www.hieurope.ingredientsnetwork.com](http://www.hieurope.ingredientsnetwork.com)November 14–15, 2012. Novel Sources for Omega-3 for Food and Feed, Copenhagen, Denmark. Information: [www.euro-fedlipid.org](http://www.euro-fedlipid.org)November 15–16, 2012. 10th Practical Short Course: Omega-3 and Nutritional Lipids, Frankfurt am Main, Germany. Information: [www.smartshortcourses.com](http://www.smartshortcourses.com)November 19–20, 2012. World Cocoa Conference, Abidjan, Côte d'Ivoire. Information: [www.icco.org](http://www.icco.org)November 20–23, 2012. 2012 European Federation of Food Science and Technology Annual Meeting, Montpellier, France. Information: [www.effostconference.com](http://www.effostconference.com)November 23–24, 2012. 67th Annual Convention of the Oil Technologists' Association of India, Mumbai, India. Information: [www.otai.org](http://www.otai.org)November 23–24, 2012. International Conference and Exhibition on Cosmetology & Cosmetics, Hyderabad, India. Information: [www.omicsonline.org](http://www.omicsonline.org)November 27–29, 2012. Chocolate Confectionery Production, Leatherhead, Surrey, UK. Information: [www.leatherheadfood.com/training-and-conferences](http://www.leatherheadfood.com/training-and-conferences)

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## AOCS Meeting Watch

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October 29–31, 2012. Singapore 2012: World Conference on Fabric and Home Care, Shangri-La Hotel, Singapore. Information: email: [meetings@aocs.org](mailto:meetings@aocs.org); phone: +1 217-693-4821; fax: +1 217-693-4865; email: [meetings@aocs.org](mailto:meetings@aocs.org); [singapore.aocs.org](http://singapore.aocs.org)

April 28–May 1, 2013. 104th AOCS Annual Meeting & Expo, Palais des congrès de Montréal, Montréal, Québec, Canada. Information: email: [meetings@aocs.org](mailto:meetings@aocs.org); phone: +1 217-693-4821; fax: +1 217-693-4865; [AnnualMeeting.aocs.org](http://AnnualMeeting.aocs.org)

August 20–23, 2013. 15th AOCS Latin American Congress and Exposition on Fats and Oils, Sheraton Santiago Hotel and Convention Center, Santiago, Chile. Information: email: [meetings@aocs.org](mailto:meetings@aocs.org); phone: +1 217-693-4821; fax: +1 217-693-4865; [www.aocs.org/meetings](http://www.aocs.org/meetings)

May 4–7, 2014. 105th AOCS Annual Meeting & Expo, The Henry B. Gonzalez Convention Center, San Antonio, Texas, USA. Information: phone: +1 217-693-4821; fax: +1 217-693-4865; email: [meetings@aocs.org](mailto:meetings@aocs.org); [aocs.org/meetings](http://aocs.org/meetings)

May 3–6, 2015. 106th AOCS Annual Meeting & Expo, Rosen Shingle Creek, Orlando, Florida, USA. Information: phone: +1 217-693-4821; fax: +1 217-693-4865; email: [meetings@aocs.org](mailto:meetings@aocs.org); [aocs.org/meetings](http://aocs.org/meetings)

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November 27–29, 2012. National Advanced Biofuels Conference & Expo, Houston, Texas, USA. Information: [www.advancedbiofuel-sconference.com](http://www.advancedbiofuel-sconference.com)

November 28–29, 2012. 2nd Annual Waste Conversion Congress West Coast, Long Beach, California, USA. Information: [www.renewable-waste.com/waste-conversion-west](http://www.renewable-waste.com/waste-conversion-west)

## December

December 4–5, 2012. European Advanced Biofuels Congress, Brussels, Belgium. Information: [www.greenpowerconferences.com](http://www.greenpowerconferences.com)

December 4–5, 2012. International Algae Conference, Berlin, Germany. Information: <http://algaecongress.com>

December 4–5, 2012. 12th Practical Short Course: Trends and Markets in Aquaculture Feed Ingredients, Nutrition, Formulation and Optimized Feed Production and Quality Management, Ghent, Belgium. Information: [www.smartshortcourses.com](http://www.smartshortcourses.com)

December 4–5, 2012. Labeling Requirements and Implications for Foods Marketed in the United States, Arlington, Virginia, USA. Information: [www.ift.org/meetings-and-events/short-courses.aspx](http://www.ift.org/meetings-and-events/short-courses.aspx)

December 5, 2012. Cosmetic Raw Materials, New York, New York, USA. Information: [www.sconline.org](http://www.sconline.org)

December 5–7, 2012. Algae Technology Europe, Ghent, Belgium. Information: [www.smartshortcourses.com](http://www.smartshortcourses.com)

December 6–9, 2012. World Congress on Clinical Lipidology, Budapest, Hungary. Information: [www.clinical-lipidology.com](http://www.clinical-lipidology.com)

## 2013

January 28–February 2, 2013. American Cleaning Institute Annual Meeting & Industry Convention, Orlando, Florida, USA. Information: [www.cleaninginstitute.org](http://www.cleaninginstitute.org)

February 3–8, 2013. Feeds and Pet Food Extrusion, College Station, Texas, USA. Information: <http://foodprotein.tamu.edu> ■

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# THE SCIENCE

## behind optimal frying

### Christian Gertz and Richard Stier

Christian Gertz is director of the Official Institute of Chemical and Food Analysis located in Hagen, Germany. His areas of research include all aspects of frying processing—analytics, Hazard Analysis and Critical Control Points (HACCP), thermo-oxidative degradation, heat-stabilizing agents, and fat quality—and analysis of fats and oils. In 1982, he received the Josef Schormüller Foundation Award given by the German Food Chemical Society, and in 2004 he received the Normann Medal from the German Society for Fat Research (DGF). He may be contacted at christian.gertz@dr-gertz.de.

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Pan frying, deep frying, and shallow frying can be differentiated by the duration and intensity of the interaction between oil and air, coupled with the heat and mass transfer between the food and the frying medium. However, all three types of frying can be complicated by the changing composition and structure of the food being fried and the dramatic changes in the frying oil itself. Such complexity makes it difficult to anticipate and to solve problems such as oil uptake, the need for *trans*-fats alternatives, and the formation of acrylamide and the potential formation of unhealthful degradation products.

A good understanding of the frying process and the potential changes that may occur in the food and oil can help manufacturers optimize their operations and improve restaurant frying in ways that lead to better food and fat quality, a higher degree of control over changing oil chemistry, and improved profitability. While actual practice tends to be based on trial and error, the road to optimal frying should include well-designed frying studies.

### Frying process

Many different methods have been used to simulate the frying process in the laboratory. These take into account frying temperatures and heat transfer into the food, changing fat chemistry, and moisture loss from the food. Various theoretical studies have provided insights into understanding both oil uptake and the heat transfer mechanisms that take place during deep-fat frying. For example, it was previously assumed that the oil that accumulated on the surface of a food during frying entered the food after it was removed from the fryer and began to cool.

Frying heats the water of a frying material to boiling, creating first an outer layer of gelatinized starch and denatured proteins.

This layer develops a porous structure, or crust, with a number of air- or water-filled pores. (The aim is to achieve a regular distribution of pores in the crust at the very beginning of frying.) After this boundary zone is dehydrated, water migrates with constant pressure from the center of the product toward the outer layer to replace the moisture that is lost during heating (Fig. 1). When no more moisture can leave the product, the vapor pressure will drop within the food as the temperatures increase from 100–103°C in the crust to the temperature of the surrounding oil. It is also the moment that formation of acrylamide in the crust starts due to the Maillard reaction.

This insight makes it more likely that the microstructure—and the process by which it is formed during the first moments of frying—has the greatest influence on oil uptake as well as the odor, appearance, texture, and crispness of fried foods. Consequently, the aim is to achieve a regular distribution of pores in the crust at the very beginning of frying. The food should not have pores that are too large, connected, or that have been destroyed due to the higher vapor pressure. Also, as moisture loss and

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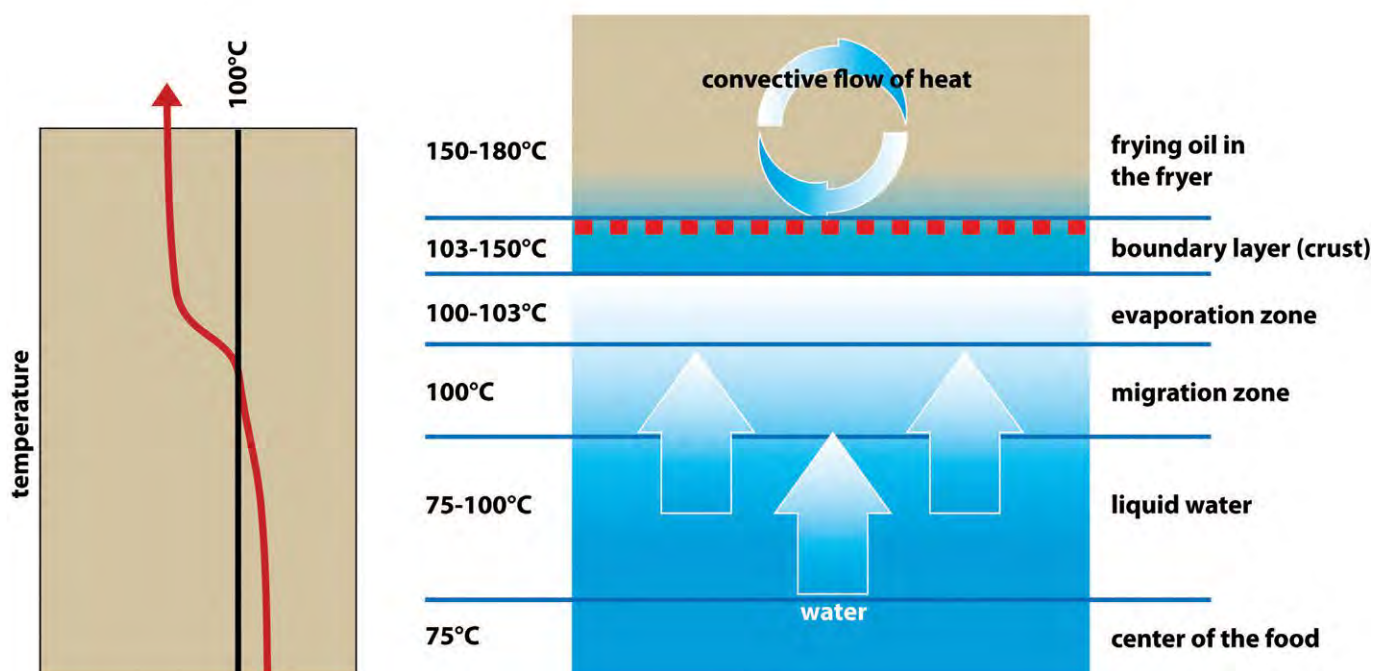


FIG. 1. Heat and mass transfer during frying process.



**TABLE 1.** Physical and chemical parameters to analyze used frying oils

Physical parameter	Changes during deep- frying	Caused by	Correlation with the deterioration of fat (* = dependent on the type of fat)
Refractive index/UV	Increases	Accumulation of conjugated fatty acids	Yes*
Density	Increases	Polymerized Triacylglycerols	Yes*
Dielectric coefficient	Decreases	Polar-oxidized components – disturbed by free fatty acids and water	Yes*
Colour	Becomes more intensive and darker	(Maillard) reaction products of amino acids (protein) and unsaturated Carbonyl compounds	No
Conductivity	Increases	Polar compounds	Yes*
Surface tension	Decreases	Polar compounds	Yes*
Smoke point	Decreases	volatile oxidized decomposition products	Yes*
Specific Heat	Increases	Polar compounds	Yes*
Viscosity	Increases	Polymerized Triacylglycerols (MG>1000)	Yes*
Chemical parameter			
Anisidine value	Increases	Secondary oxidation products	Yes*
Iodine value	Decreases	Formation of oxidized fat products	Yes*
Peroxide value	Increases but can also decrease	Primary oxidation products	No
Petrolether – insoluble oxidized fatty acids	Increases	Oxidized polymerization products	Yes
Polar compounds	Increases	Oxidized and polymerized degradation products including unchanged polar fat components	Yes
Polymerized Triacylglycerols	Increases	Oxidized and not oxidized polymerized Triacylglycerols	Yes
Acid value	Increases	Formation of oxidation products with free carboxyl groups	No

oil uptake are associated, it is important to retard the moisture transfer as long as possible. To meet this goal, a range of coatings that form edible films are now used. These films, which are made of polysaccharides in combination with minerals and special proteins from wheat or egg, form gel matrices to keep the temperature in the boundary zone at the boiling point, thus retarding the loss of moisture.

### Factors affecting frying oil quality

The criteria for the selection of suitable cooking oils have changed substantially in recent years. Concerns about *trans* fatty acids resulted in a switch to liquid oils that are rich in oleic acid and/or palm oil. More recently, an increasing emphasis on health and safety, the environment, and the need for a clean label have again led producers of fried food in Europe to seek alternatives to palm oil. Some have switched to canola oil or sunflower oil containing higher amounts of unsaturated fatty acids, which are potentially less stable. Processors are also looking for oils that are free of synthetic antioxidants, additives, allergens, and genetic modification. Of course, since most oils used for industrial and restaurant frying are refined, bleached, and deodorized, they will not contain allergens and will be free of components that those opposed to genetically modified organisms fear may create problems.

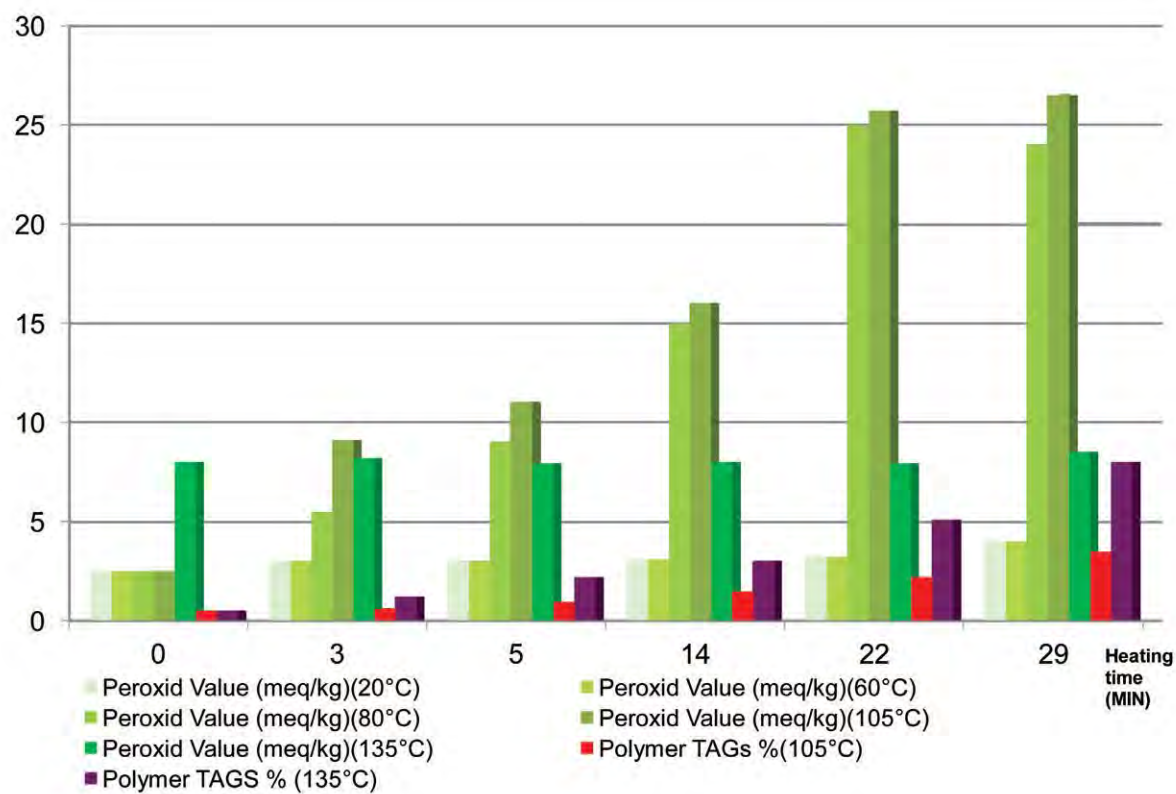
Meanwhile, minimizing the effects of thermal and oxidative degradation of oils to ensure the quality of fried foods has become more important than ever. Unfortunately, for the past two decades research on fat degradation at frying temperature has been stagnant due to

several pervasive beliefs: (i) reactions of lipid oxidation are well understood; (ii) the heat stability of a fat depends on its fatty acid composition as well as on the duration of contact between hot fat and atmospheric oxygen; (iii) used oils that have not been abused are safe and wholesome.

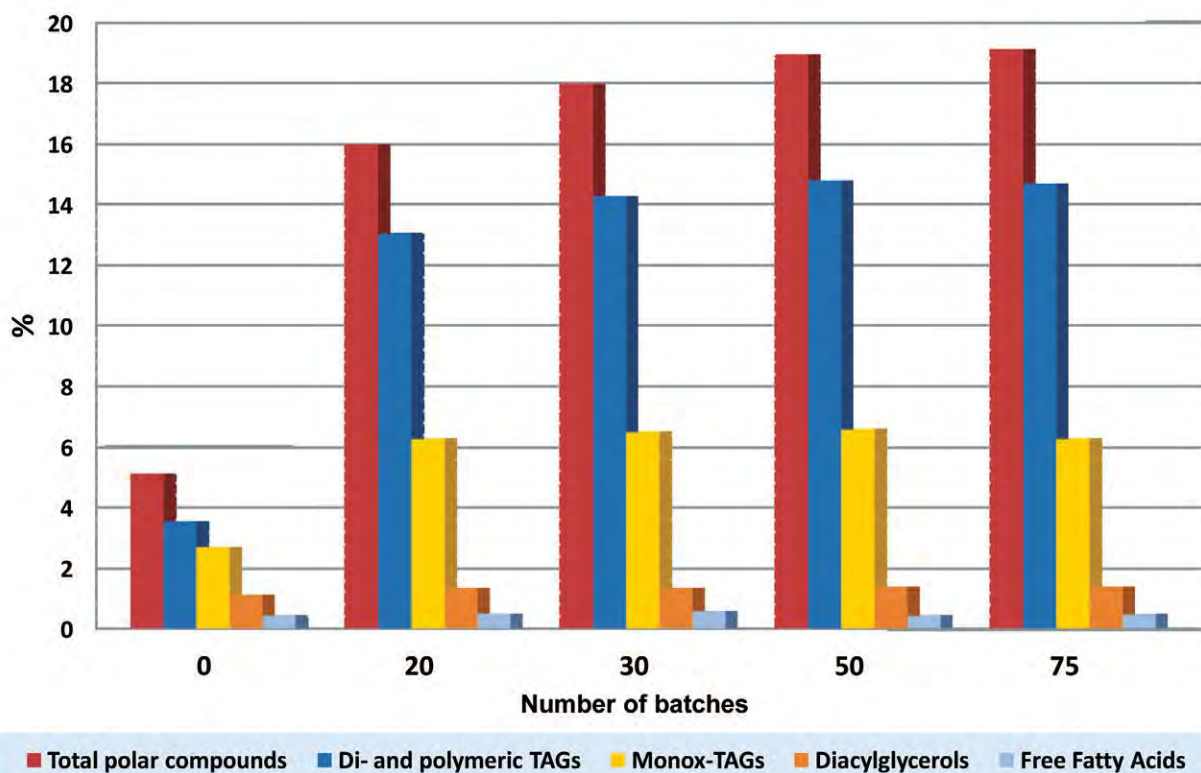
A number of phenomena that occur during the frying of food can be better explained by ignoring the conventional view of fat degradation as a pure radical mechanism. At elevated frying temperatures, triacylglycerols (TAG), the main components in fats and oils (~98% in fresh oils), are affected by oxygen and heat; as a result of oxidation and polymerization, degradation products are formed which are analytically determined as total polar compounds (TPM).

The greatest damage to fats and oils during the frying process occurs at elevated temperatures (160–200°C). The current assumption is that there are two main types of degradation reactions (Fig. 2) due to changing contact with air. The radical mechanism for lipid peroxidation starts above 60°C, and is accelerated until 120–130°C is reached. Above 130°C, a nonradical acid-catalyzed mechanism for the formation of C–C linked dimeric-, polymeric-, or cyclic-triglycerides, dimeric sterols, or oligomerized tocopherols predominates.

Comparing the results of a heating test at 170°C and a Rancimat test at 110 or 120°C demonstrated that tests executed at temperatures lower than 130°C do not allow researchers to estimate the frying behavior of the



**FIG. 2.** Changes in peroxide values and polymerized triacylglycerols during heating of sunflower oil between 20°C and 135°C.



**FIG. 3.** Changing composition of the polar fraction in used frying fats.



oil at 170°C. Many synthetic antioxidants, such as butylated hydroxytoluene (BHT) and butylated hydroxyanisole (BHA), become substantially less effective or even inactive when subjected to elevated temperatures. By contrast, ascorbyl palmitate, sterols, and many other natural ingredients become effective antioxidants at elevated frying temperatures.

There are also some natural ingredients and plant extracts from rosemary and sage that in the presence of acids change into effective antioxidants at deep-frying temperatures.

Understanding the different routes of fat degradation and the effects of changing temperatures during the frying process has been enhanced by a new test that heats oil at 50, 110, and 170°C with and without purging the oil with air (20 L/h) to simulate the different conditions during shallow and deep frying. After each heating period, the amounts of total polar compounds and polymerized TAG, anisidine value, and acid value (free fatty acid) are determined by near-infrared spectroscopy. The data are then analyzed by chemometry to determine the most suitable oil for the job.

Besides autoxidation, the hydrolysis of TAG is often considered as the most important reaction during the deterioration of frying. However, no significant variations of TAG forming free fatty acids (FFA), monoacylglycerols, diacylglycerols, or glycerol are observed (Fig. 3). One end product of hydrolysis is acrolein, an eye irritant. In fats and

oils not containing medium-chain triacylglycerols (< C14), the hydrolytic splitting rises only to a very small extent. Short-chain FFA (octanoic and heptanoic acids) are formed by oxidation of polyunsaturated fatty acids. The remaining glycerol backbone contains the oxidized fatty acid fragments, which have nearly the same molecular weight as diacylglycerols. This can lead to a misinterpretation of a chromatogram of used frying fats, developed by size exclusion–high-pressure liquid chromatography, due to the same molecular mass.

Short-chain fatty acids are volatile and may be distilled off with water vapor so that they are not included in the titration of FFA. Therefore, the acid value, as well as other physicochemical tests, cannot be used as an objective index of fat degradation because it is influenced too much by the nature of the oil and degree of unsaturation. Determinations of the concentrations of polar compounds and polymeric TAG remain the most reliable methods for chemical analysis of frying oils and fats (Table 1 on page 552).

Although the regulatory limits in Europe for restaurant frying oils range between 24% and 27% polar compounds, the levels found in most restaurant operations are much lower since abused oils also produce poor-quality foods. Oils used for industrial frying come nowhere near the regulatory limits for polar materials. The continual replenishment of used frying oils is recommended to keep the amount of total polar compounds below 18–19%, as these

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artifacts accelerate degradation at higher concentration. The formation of undesirable oxidation products has to be controlled, or the foods will have off-flavors and a shorter shelf life.

## Health effects

The demand for fried or pre-fried products remains high despite discussions about obesity and health hazards associated with eating fried foods. It is estimated that globally about 20 million metric tons of frying fats and oils are used each year, with an estimated value of \$100 billion in the United States alone. Abuse of frying oil may create unintended and even toxic volatile and nonvolatile decomposition products. The degraded oil is totally absorbed into fried products, which are then ingested. For many years, the safety of heated/fried oils has been questioned. Long-term feeding studies with rats using frying oils that were not abused did not demonstrate any associated health issues.

In fats and oils subjected to oxidation at low temperature ( $<120^{\circ}\text{C}$ ), rarely more than 4-5% of triacylglycerols would be oxidized. Hydroperoxides are practically absent above  $130^{\circ}\text{C}$  and monomer oxidized triacylglycerols (Monox-TAG) are formed during the early stages of heating at elevated temperatures. Monox-TAG include short-chain fatty acyl and short-chain n-oxo fatty acyl groups as well as different oxygenated groups such as hydroxy, keto and epoxy. Recent work indicates that the presence of high levels of Monox-TAG in the frying oils may be linked with an elevated risk for cardiovascular diseases, hypertension, and diabetes. The new findings confirm the studies on physiological effects described 50 years before. In used frying fats at all states of degradation more oils contain more than 5 to 20% monomer oxidized TAG (Fig. 3, page 553).

For more information on optimal frying, see [www.dgfett.de/material/fritier.php](http://www.dgfett.de/material/fritier.php). ■

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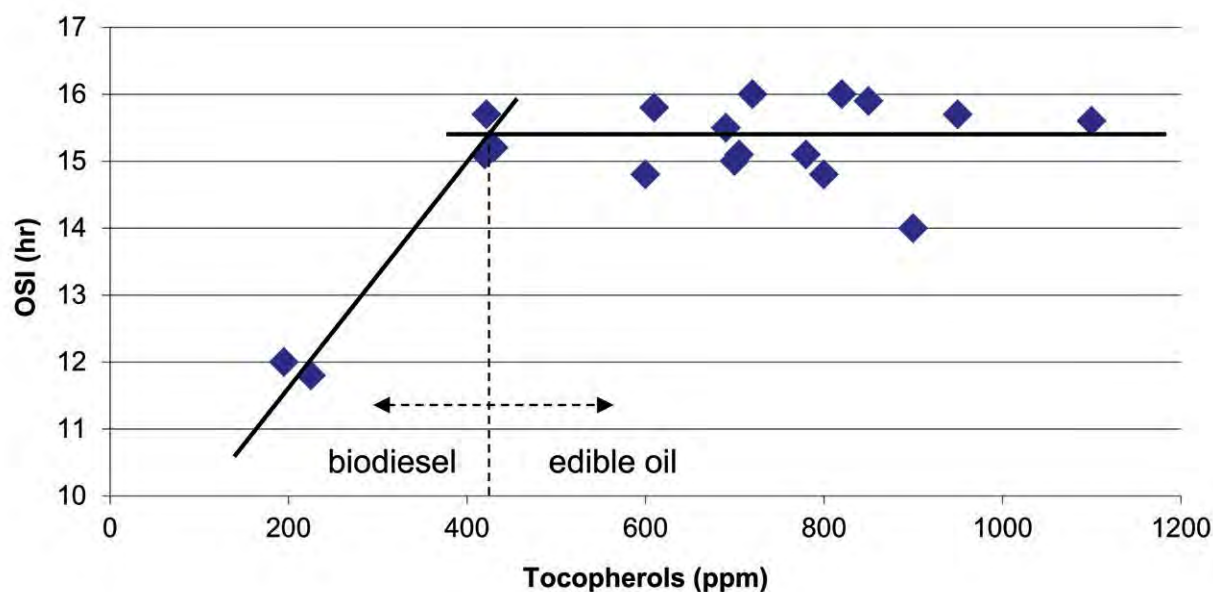


FIG. 1. Effect of tocopherol content on the oxidative stability of soybean oil. OSI, oxidative stability index.

# Efficient recovery of tocopherols from vegetable oils

Wim De Greyt

Growing consumer awareness of nutrition and health has resulted in an increased interest in vegetable oils for their content of several valuable minor components (sterols, carotenoids, ubiquinones, isoflavones, etc.) with known positive nutritional characteristics.

Specifically, vegetable oils are the main natural source of tocopherols (also known as vitamin E), which are proven to have biological activity as antioxidants. Germ oils, such as wheat and corn, have the highest tocopherol levels (1,500–4,000 ppm), but soybean oil is also rich in tocopherols (800–1,200 ppm). Four different isomers ( $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ -tocopherol) can be distinguished.  $\gamma$ -Tocopherol is the most abundant isomer in nearly all vegetable oils, with relative percentages up to 65% in soybean oil and 80% in corn oil. The tocopherol content of sunflower oil consists almost exclusively of  $\alpha$ -tocopherol whereas  $\delta$ -tocopherol is only present in significant amounts in soybean oil. A homologous series of tocotrienols is present mainly in palm oil (DeGreyt and Kellens, 2005).

Natural tocopherols were originally isolated from wheat germ oil but today are mostly derived from soybean oil (>80%) and rapeseed oil (10–15%). Tocopherols are stripped from the oil during the deodorization process and concentrated in the deodorizer distillate.

TABLE 1. Typical tocopherol and free fatty acid (FFA) content of vegetable oil deodorizer distillates<sup>a</sup>

Oil	Tocopherols (%)	FFA (%)
Soybean	7.5–16.5	33–73
Canola	3.5–4.5	40
Sunflower	1.5–5.0	39–70
Corn	1.5–3.5	75–80
Palm		>90

<sup>a</sup>Source: Verleyen *et al.* (2001)

Tocopherol producers are especially interested in deodorizer distillates with more than 15% tocopherols; such concentrations can be obtained during the chemical refining of soybean oil. Physical refining generally yields deodorizer distillates with high free fatty acid (FFA) content and correspondingly much lower tocopherol levels (Verleyen *et al.*, 2001) (Table 1). Owing to the current high demand for tocopherol-rich distillates, their commercial value basically doubled from approximately

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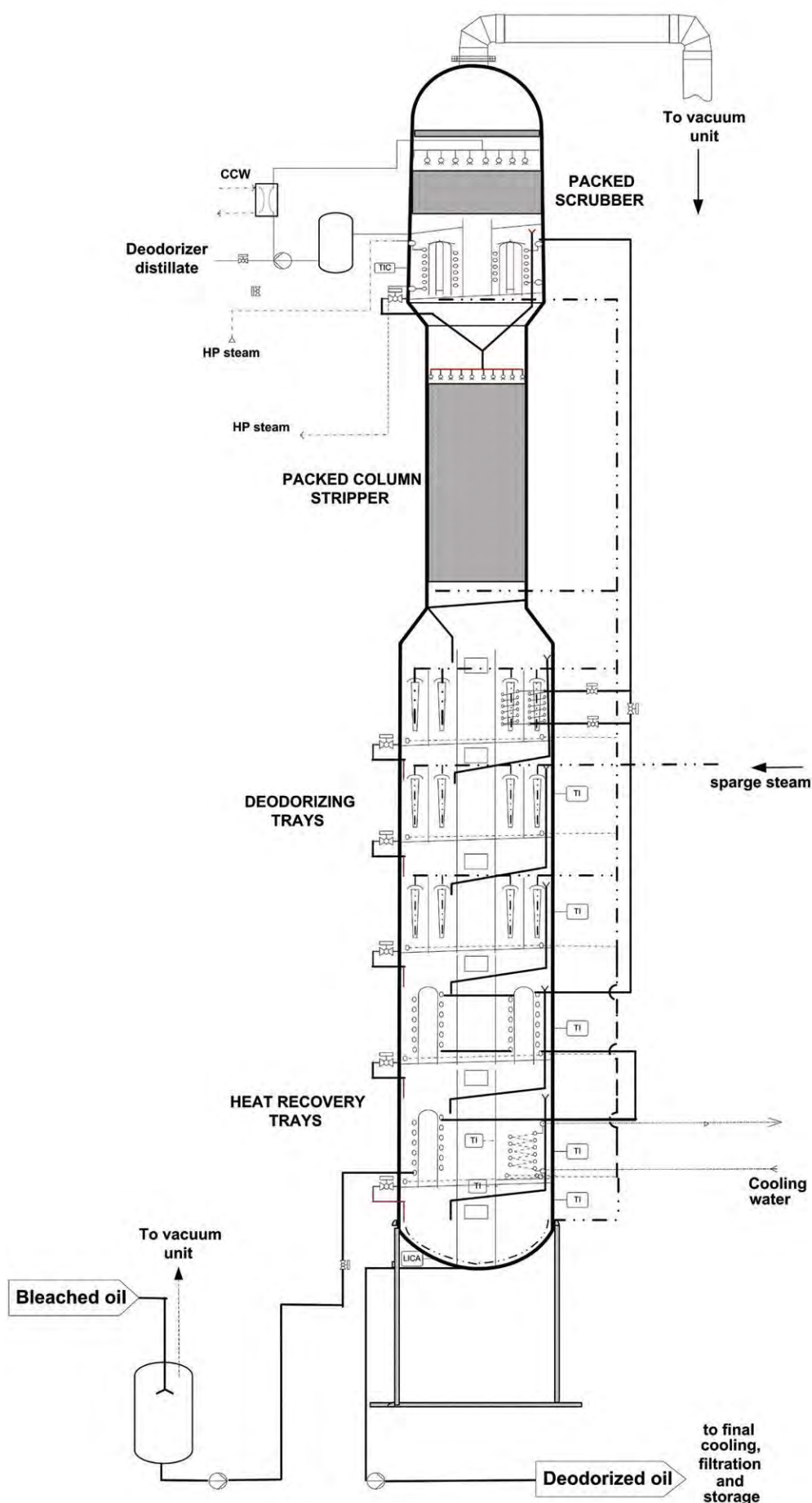


FIG. 2. Stripper-deodorizer with single scrubber.

\$20/kg tocopherol in 1998 (Walsh *et al.*, 1998) to more than \$40/kg tocopherol today. The price will remain high as long as demand exceeds supply, while the availability of natural tocopherol will always be directly related to vegetable (soybean) oil production and refining.

The maximum acceptable degree of tocopherol stripping depends on the type of oil and its application. Refined soybean oil for food applications requires a minimum of 500 ppm tocopherols to guarantee a good oxidative stability (Fig. 1, page 557). More tocopherols can be stripped from partially hydrogenated soybean oil, which is more saturated and therefore requires only 200 ppm tocopherols for oxidative stability, or from soybean oil that is used for biodiesel production. In this latter case, antioxidants can be added to the soy biodiesel to reach the required oxidative stability.

Hence, by taking a global yearly production of 40 million metric tons (MT) of soybean oil and assuming a potential tocopherol recovery of 300–400 ppm during deodorization, the maximum natural tocopherol availability from soybean oil can be estimated at 12,000–16,000 MT per year. The effective production is difficult to estimate but is probably today still less than 5,000 MT (expressed as 100% tocopherols) or less than 30,000 MT of deodorizer distillate (with 15% tocopherols).

## Controlled tocopherol stripping from vegetable oils

A number of features have been introduced in deodorizer design to improve (control) tocopherol stripping without compromising the overall refined oil quality (Fig. 2). Chilled barometric vacuum or dry ice condensing systems allow a lower effective pressure in the deodorizer (<1.5 mbar), which significantly improves the stripping efficiency. Integration of a high-temperature–short-time section in the deodorizer also results in higher tocopherol stripping. By keeping the time short, unwanted thermal side reactions can be minimized. The high-temperature section can either be a classical (shallow bed) deodorizing tray or a packed column. The latter is known to be an efficient stripper, but because of the pressure drop over the structured packing, packed columns are only slightly more efficient than a shallow bed deodorizing tray for stripping of tocopherols and sterols. The main advantage of a packed column (when operated at high

temperature) is the short residence, which is very favorable for temperature-sensitive oils such as soybean. However, this short residence also requires that an additional deodorizing stage be added after packed column stripping to achieve a fully deodorized oil.

## Efficient recovery of tocopherols

Efficient recovery of tocopherols involves more than only an efficient stripping. It also requires minimal degradation and an efficient condensation in the deodorizer distillate. In practice, minor tocopherol losses may occur owing to thermal degradation or oxidation or to incomplete vapor condensation. Thermal degradation will be limited to 5% at normally applied deodorizing temperatures (220–250°C) but may become higher (10% or more) when the oil is deodorized too long at temperatures of 260°C or higher. Tocopherol oxidation can occur by reaction with dissolved air (from minor leakages) or radicals (from peroxides). It is very important to minimize air leakages, otherwise tocopherol losses can be 15–20%. High tocopherol recovery (up to 90% of what is removed during deodorization) is only possible when correct deodorizing conditions are applied in a properly designed deodorizer.

Volatile matter (FFA, tocopherols, sterols, and others) stripped from the oil during deodorization is condensed in a vapor scrubber. Complete condensation is achieved by creating a very good contact between the hot vapor phase and recirculated, cooled deodorizer distillate. This is done either by a series of sprayers integrated in the vapor duct or through a packed bed in the scrubber vessel. Most deodorizers have only one vapor scrubber from which a single deodorizer distillate is obtained. The composition of such deodorizer distillate is mainly determined by the applied refining mode (chemical or physical refining). When applied in physical refining, deodorizer distillates obtained from a single scrubber will have significantly lower tocopherol concentrations due to the diluting effect of the stripped FFA (Tables 1 and 2, pages 557 and 559). However, if the FFA content of the incoming oil is

**TABLE 2.** Tocopherol concentration in deodorizer distillate from single scrubber

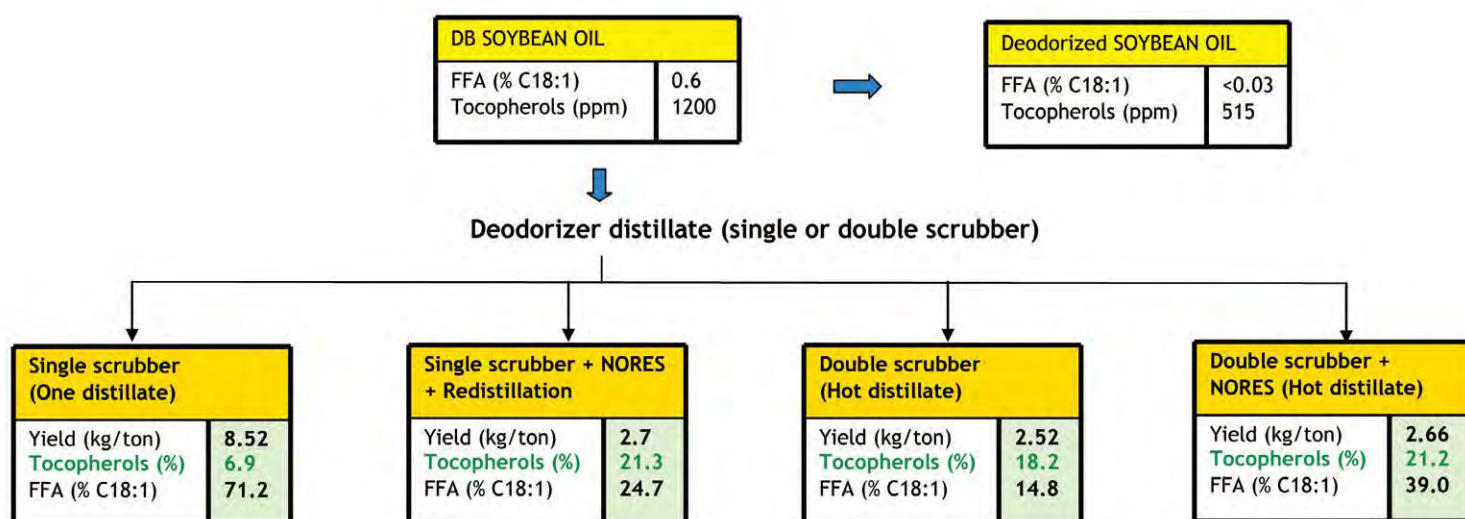
	NB oil <sup>a</sup>		DB oil <sup>a</sup>
FFA (% C18:1)	0.05	0.1	0.6
Tocopherols (ppm)	1240	1200	1200
	Fully refined oil		
FFA (% C18:1)	0.015	<0.03	0.6
Tocopherols (ppm)	900	511	515
	Deodorizer distillate		
FFA (% C18:1)	39.2	31.3	71.2
Tocopherols (%)	<b>20.4</b>	<b>16.2</b>	<b>6.9</b>
Yield (kg/ton)	1.23	3.6	8.52

<sup>a</sup>Neutralized, bleached oil; <sup>b</sup>degummed, bleached oil

## information

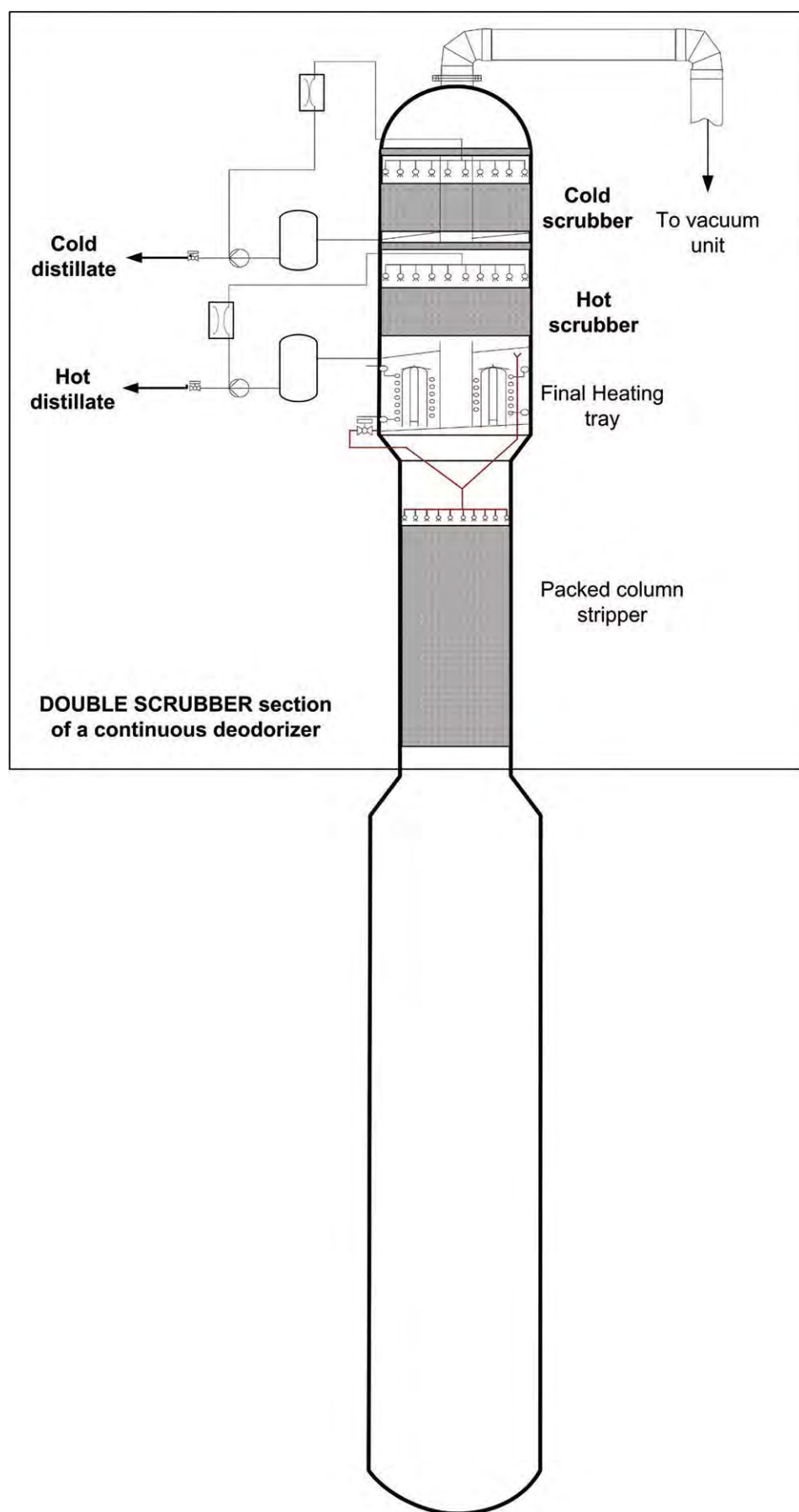
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**FIG. 3.** Overview of different options for the production of tocopherol-rich side streams during physical refining of soybean oil. Abbreviations: FFA, free fatty acids; DB, degummed bleached; NORES, neutral oil recovery system.





**FIG. 4.** Principle of double scrubber for selective condensation of free fatty acids (FFA) and tocopherols.

low ( $<0.05\%$ ) and if enough tocopherols are stripped, a single scrubber can yield deodorizer distillate with higher than 15% tocopherols (Table 2, page 559).

Although soybean oil is still mostly chemically refined, processors seem to have a growing preference for physical refining. Typical FFA content of good-quality degummed, bleached soybean oil is 0.5–0.6%. Since it is not possible to obtain a deodorizer distillate with a high tocopherol content by using a single scrubber setup (Table 2), other strategies have to be applied for that purpose.

One possible option is to redistill the single-scrubber deodorizer distillate. This gives a very sharp separation of tocopherols and FFA. When applied properly, it can yield a side stream with more than 20% tocopherols (Fig. 3, page 559). The main disadvantages of this approach are the required extra heat-treatment of the distillate. This increases the risk of tocopherol degradation and concentrates the tocopherols together with other less volatile impurities in the heavy distillation pitch.

It is better to produce a deodorizer distillate with elevated tocopherol content by integrating two scrubbers in the deodorizer. The double scrubber concept was developed in the late 1990s and has been continually improved since then (Fig. 4). The vapor phase leaving the deodorizer is first partially condensed at a higher temperature. This yields a hot distillate in which the least volatile components (e.g., tocopherols and sterols) are concentrated. Complete condensation of the remaining more volatile substances (mainly FFA) is then achieved in the second cold scrubber, yielding an FFA-rich cold distillate. Provided that the condensation temperatures of the hot and cold scrubber are properly set, the double scrubber concept gives a very good separation between the FFA and tocopherols. Contrary to the redistillation approach where separation is obtained through fractional distillation, separation in the double scrubber is achieved by fractional condensation of the vapor phase. The latter requires no additional heating and may therefore give a higher tocopherol yield and a better overall distillate quality.

Achievable tocopherol concentrations in deodorizer distillates from the physical refining of soybean oil are presented in Figure 3. A single scrubber gives a high distillate yield but with an unacceptably low tocopherol concentration (6.9%). Implementation of a double scrubber gives a hot distillate with much higher tocopherol concentration (18.2%), but the distillate yield is obviously lower. Tocopherol concentration in the hot distillate can be

further increased by integrating a neutral oil recovery system (NORES) into the deodorizer. Such systems recover the mechanically entrained neutral oil (triglycerides and partial triglycerides) from the vapor phase before tocopherol condensation takes place. In this way, NORES leads to a higher concentration of FFA and tocopherols/sterols in the remaining vapor phase entering the scrubber resulting in "hot" distillate with a higher tocopherol concentration (21.2%). A similar tocopherol concentration can be obtained by redistilling a single scrubber distillate obtained after NORES. The current commercial value of such deodorizer distillate is high (>\$8000/MT).

## How tocopherol recovery can enhance the bottom line

Production of tocopherol-rich deodorizer distillates (>15% tocopherols) during the physical refining of soybean oil is possible by combining optimized stripper/deodorization technology with an improved scrubber design. Yields depend on the degree of tocopherol stripping and refining mode (chemical or physical) but typically range from 1.2 kg to 3.6 kg/MT oil (Table 2; Fig. 3). At the current high price of natural tocopherols, this represents a potential extra revenue of between \$15 and \$25/MT oil. Hence, it is not surprising that oil processors are highly interested in technologies that allow them to produce tocopherol-rich deodorizer distillates.

A similar opportunity presented itself during the late 1990s, but processors quickly lost their enthusiasm for recovering natural tocopherols after the demand for them dropped. Today, most tocopherol

## Vitamin E

Most vitamin E is still chemically synthesized by condensation of isophytol and trimethyl hydroxyquinone. Synthetic Vitamin E is a racemic mixture of eight stereoisomers of  $\alpha$ -tocopherol and labeled *d,l*- $\alpha$ -tocopherol. It is a standardized product that is mostly used, either in free or esterified form (acetate), as an additive in animal feed. Semisynthetic natural source vitamin E (= *d*- $\alpha$ -tocopherol) is obtained by chemical conversion (methylation) of natural tocopherols isolated from vegetable oils. Natural source vitamin E and natural mixed tocopherols (mixture of natural  $\alpha$ -,  $\beta$ -,  $\gamma$ -, and  $\delta$ -tocopherols) are preferred for higher added value applications in human food supplements and cosmetics. Global annual production of vitamin E can be roughly estimated at 70,000–75,000 MT, of which only a minor part (<5,000 MT) is of natural origin.

producers expect the natural tocopherols market to grow slowly but steadily. This expectation may positively influence the price still further, making it even more attractive for processors to upgrade their existing deodorizers or invest in new deodorizing/scrubber technologies.

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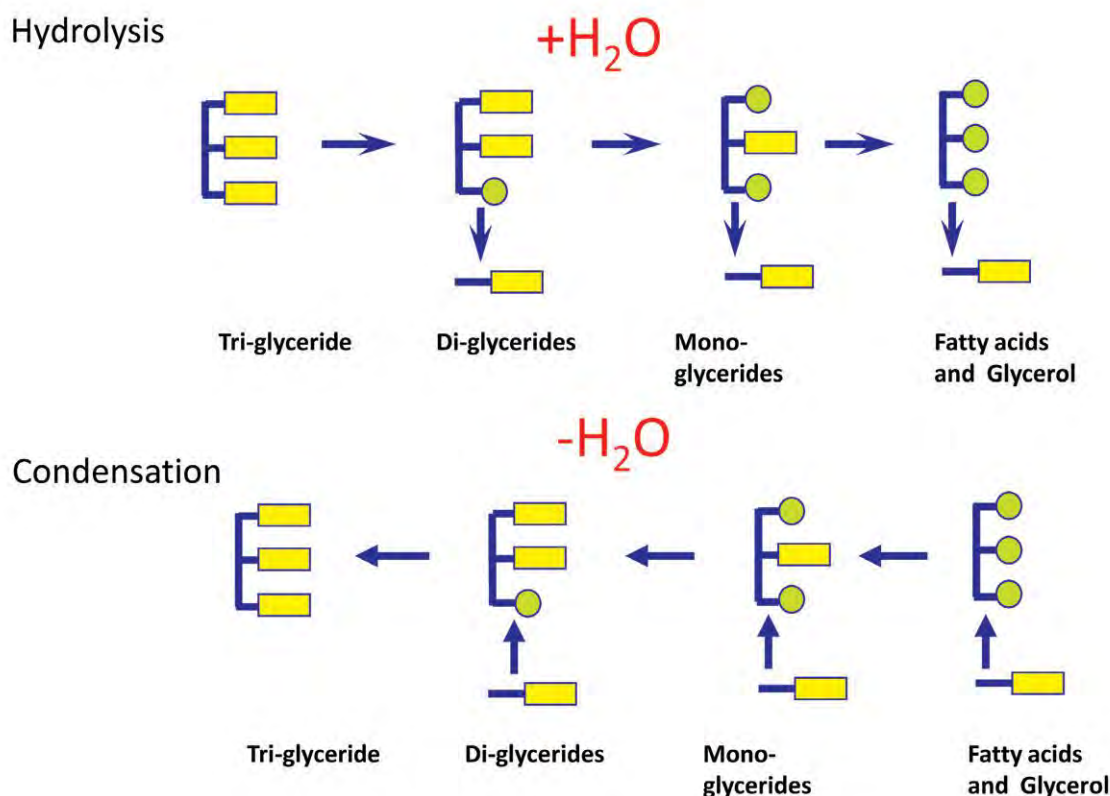


FIG. 1. Hydrolysis and condensation by lipases.

# Improving sustainability in oils processing by fatty acid recovery

David Cowan

Free fatty acids (FFA) are found in all vegetable-, plant-, and animal-derived oils in amounts that vary depending on the handling of the oil sources and their physical location. Typically, crude palm oil contains 3–5% FFA, whereas levels in rice bran oil can be up to 25%. FFA not only occur in oils derived from warm, humid climates but are also widespread in oils from temperate and even cold climates. The FFA component is always removed in refining because of its negative impact on flavor and stability. Methods for removal vary from distillation (deodorization) to alkali neutralization but are always associated with a loss in yield.

As the source of this FFA is often the action of lipases that are either present on the crop before harvesting or extracted together with the oil, we have investigated the possibility of reversing the hydrolysis reaction to regenerate the original triglycerides. Almost total removal of FFA can be achieved this way, but at the cost of long reaction times and enzyme dosages. However, the customer solutions department at Novozymes believes that by accepting a less-than-total reduction, we can convert more than 75% back to triglycerides and thus improve yield and processing sustainability.

## Some background on lipases

Lipases are well known for their ability to hydrolyze fats and to produce fatty acids and partial glycerides as reaction products. Often this is a desired function, as in the removal of fat-based stains when washing clothes and textiles. In other situations, it is not so desirable,

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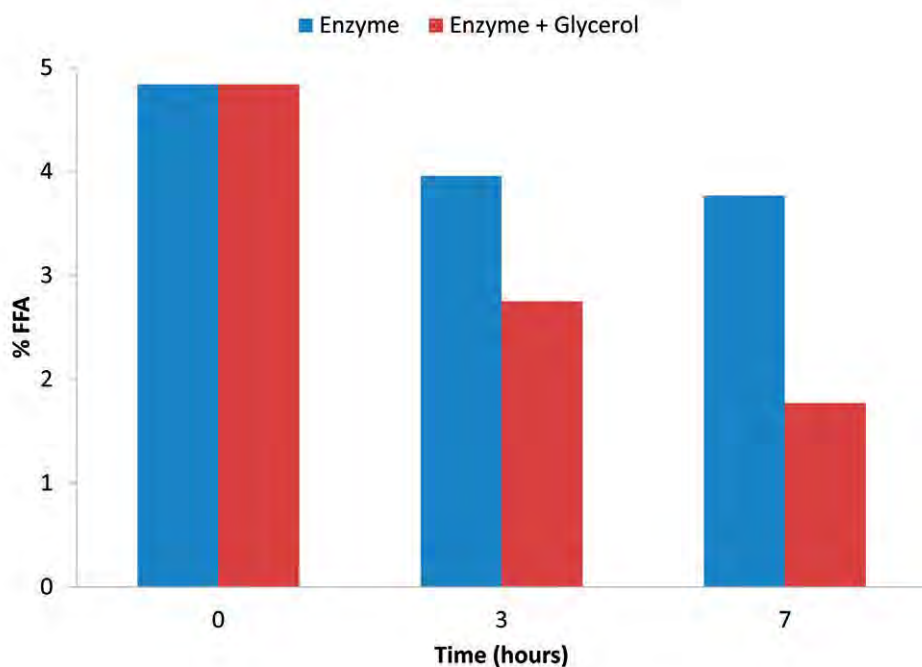


FIG. 2. Reduction in free fatty acids (FFA) following enzyme treatment.

as in the spoilage of milk, in which an off-taste is generated by the hydrolysis of milk fat by microbial lipases.

In oils and fats processing, there are opportunities for lipase to hydrolyze the triglycerides and to increase the level of FFA. Palm and rice bran oils, which are cultivated and harvested in warm, humid climates, are ideal for the growth of fungi. These fungi secrete lipases to hydrolyze fats in the oil and to liberate fatty acids, which the fungi then absorb for their own growth. But as the secreted enzymes continue to liberate fatty acids, the FFA levels generated begin to exceed the growth requirements of the fungi involved. As a result, levels of FFA in the oil climb and eventually must be removed.

Fish oils are a main source of the nutritionally important but unstable long-chain omega-3 fatty acids. Crude fish oil from which these fatty acids are extracted can contain 5–8% FFA. These can be removed by caustic soda neutralization or by an equal amount of entrained oil in the soap that is produced.

Esterases, a type of lipase, have been used in synthesis reactions for a number of years. The esterase from *Candida antarctica*

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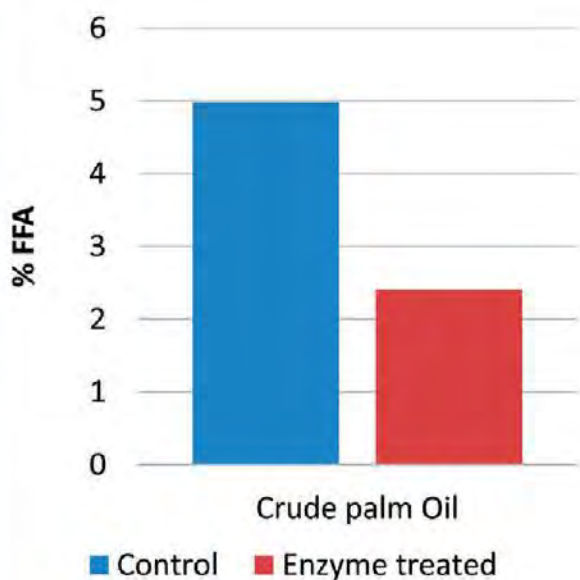
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**FIG. 3.** Reduction in FFA in crude palm oil using immobilized enzymes. For abbreviation, see Figure 2.

has been widely studied and is used today on a large scale to convert fatty acids and fatty acid esters to triglycerides by a condensation reaction with glycerol. Our department at Novozymes recently tried to use that same enzyme to recombine fatty acids with the partial glycerides coming from the original hydrolysis as a way to regenerate the original triglycerides. Palm oil, fish oil, a residue corn oil from ethanol production, and waste food oil were used to test this process.

## Enzyme reactions

The basic enzyme reactions of hydrolysis and condensation are compared in Figure 1 (page S63).

In the presence of excess water, a lipase will hydrolyze a fat and produce a mixture of mono-, di-, and triglycerides and fatty acids. Some lipases will completely hydrolyze fats to a mixture of fatty acids and glycerol, but most enzymes involved in oil spoilage do not have the time or ability to complete the hydrolysis. Reversing the reaction by using an esterase generates water, which needs to be removed for the reaction to continue. The keys to a successful reaction are therefore to choose the right enzyme (one that is not good at hydrolysis) and to efficiently remove the excess water.


## FFA reduction in palm oil

The warm and humid conditions under which the oil palm grows are almost ideal for fungal growth on the fruit bunches, and the delay before processing—particularly during sterilization of the fruit bunches—provides plenty of opportunity for hydrolysis. Because the


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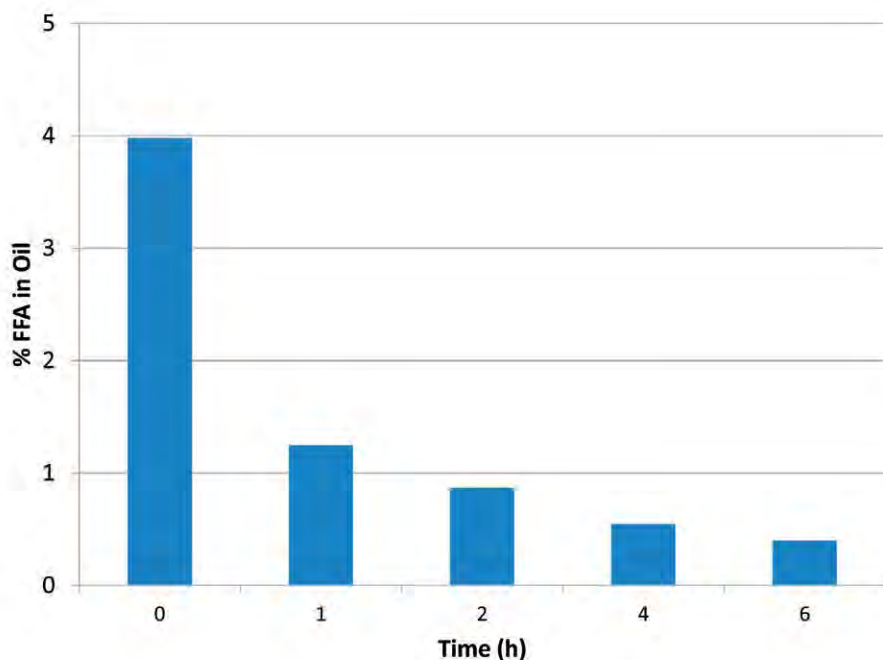
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**FIG. 4.** Reduction by enzymatic condensation reaction of FFA in fish oil. For abbreviation, see Figure 2.

oil is not free of particulates at this stage, we decided to run our screening tests with the *C. antarctica* esterase formulated as a liquid product. Although the enzyme preparation contains water, running the condensation at 70°C and 20 mbar of vacuum can easily remove both the added and produced water. Figure 2 on page 564 shows the reduction in FFA achieved in the presence and absence of glycerol. The role of glycerol is to provide extra sites for the attachment of fatty acids in case there are not enough partial glycerides in the oil.

Normally, immobilized enzymes are contained in a packed bed reactor, but it is also possible to use them in a stirred batch tank reactor. By using them in this way we can avoid any problems with the oil not being finely filtered and the risk of clogging the enzyme column. Figure 3 (page 565) shows the reduction in FFA achieved by the use of immobilized *C. antarctica* B lipase. No glycerol was added in this case because we had noted that some of the FFA could

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OTAI Members	Rs. 5000	Rs. 6000
Students	Rs. 500	Rs. 1000
Overseas Delegates	US \$300	US \$350

**Note:** Registration fee includes the Delegate Kit, Participation in the Conference, Lunches, Tea, Conference Dinner.  
Registration Fee does not include Transport and Accommodation Charges.

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Rs. 1,35,000/= for each 3m x 3m stall	or	USD 3000

**One Free Registration will be offered for each Exhibitor.**

For any specific facility / requirement the exhibitors may contact Conference Convener

**For any further information please contact:**

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Tomato seeds, which processors currently dispose of as waste, could yield a high-value oil relatively rich in antioxidants, according to research in the *Journal of Food Science* (doi:10.1111/j.1750-3841.2012.02804.x, 2012). Researchers from the US Department of Agriculture and the University of California, Davis suggest that the nearly 1.3 million metric tons (MMT), or 60%, of the waste produced in processing tomatoes is seed. Tomato seeds contain 20–36.9% oil (on a dry basis). After assessing four processing parameters, the scientists found that increasing temperature, solvent-to-solid ratio, and extraction time increased oil yield. A larger particle size, however, reduced the oil yield. More than 0.14 MMT of oil could potentially be produced per year in the United States if all tomato seed from pomace were used.



The US National Organic Program has published a final rule that addresses the use of attapulgit as a nonsynthetic processing aid to purify vegetable oils and animal fats. Attapulgit is the product of naturally occurring attapulgus clay that is mined and subsequently dried and pulverized into a fine powder. It removes impurities to improve the appearance, flavor, and stability of plant and animal oils and fats. Details about the ruling are available at <http://tinyurl.com/attapulgit>.



Bunge North America has constructed a full-service culinary center at its Bunge Ingredient Innovation Center (BIIC) in Bradley, Illinois, which is about 60 miles (100 km) south of Chicago. The state-of-the-art facility includes an industrial kitchen and a corporate dining room with extensive video capabilities. During the grand opening of the culinary center on July 24, 2012, Bunge's Corporate Chef Adam Moore and his team prepared several dishes to highlight the kitchen's capabilities and highlight Bunge ingredients in novel formulations.



Archer Daniels Midland Co. (ADM; Decatur, Illinois, USA) announced in late July 2012 that it has agreed to purchase a port terminal in the Brazilian state of Pará. The company currently operates in all of Brazil's major agricultural ports and owns a port terminal in Santos, São Paulo. ■

# News & Noteworthy



## Soy-based tires and soccer boots

Goodyear—the No. 1 tire maker in North and Latin America and No. 2 tire maker in Europe—wants to replace mineral oil with soybean oil in the manufacture of vehicle tires. And it is not just because being “green” is a good marketing ploy.

The addition of soybean oil-based rubber compounds “can potentially increase tread life by 10%,” the company said in announcing its innovation. The compounds also blend more easily with the silica used in making tires, Goodyear said. That, in turn, reduces energy consumption at the factory and cuts down on the emission of greenhouse gases. Using soybean oil in its tire production could reduce Goodyear's use of petroleum-based oil by up to 7 million gallons (about 26 million liters) each year, the company noted.

The tire maker, which is based in Akron, Ohio, USA, produces tires in 22 countries. “If indicators remain positive,” Goodyear expects consumers will be able to purchase tires made with soybean oil as early as 2015.

The United Soybean Board (USB; Chesterfield, Missouri, USA) is partially funding the research with a grant of \$500,000 over two years. USB is supported entirely by

soybean farmers, who make individual contributions of 0.5% of the market price per bushel sold each season. The efforts of the checkoff are directed by the USB, composed of 69 volunteer farmer-leaders.

In related news, Nike, Inc., the sports gear manufacturer, is marketing a new soccer (football) boot it claims is the “most environmentally friendly production boot the company has ever made.”

Conceived and engineered in Italy, the Nike GS features recycled and renewable materials throughout the upper and plate design. The boot has a biobased sole traction plate made primarily from a compound produced from castor oil, as well as a sock liner made from “100% castor beans.” The sole plate is made of 50% renewable Pebax® Renew, a thermoplastic elastomer that is produced from castor oil by France's Arkema specialty chemicals company.

## Consumer Reports rates olive oils

Consumer Reports, a US-based nonprofit that rates brands of everything from automatic dishwashers to toasters, has taken on extra virgin olive oil. A report in its September

CONTINUED ON NEXT PAGE



2012 *Consumer Reports* (CR) magazine made some puzzling suggestions to consumers, including ranking oils with “slight flaws that might not be noticed with food” as “good.”

Tom Mueller, blogger and author of *Extra Virginity: The Sublime and Scandalous World of Olive Oil*, which is reviewed elsewhere in this issue, commended the magazine for contributing to the growing public debate about olive oil quality in the United States. “Still, for an organization whose mission is to protect and empower the consumer, they could have done better,” said Mueller. “While defining the ‘extra virgin’ grade as without defects, the report classified oils with significant taste defects as ‘poor,’ ‘fair’ and even ‘good’—what is a consumer (or an olive oil merchant) to think?”

Mueller also noted that “on the positive side of the ledger,” CR explains the cryptic term “extra virgin” (perceptible fruitiness and absence of sensory defects) and points out that no less than 14 out of the 23 oils the magazine surveyed, including several top brands, failed to qualify as extra virgin because of taste flaws. CR recommends things that a consumer should look for on an olive oil label, such as geographic provenance, and rejects “first cold press” as anachronistic marketing-speak. CR also clarifies some of the characteristics of excellent olive oil, most notably bitterness and pungency, and stresses the importance of organoleptic qualities as an integral part of the international regulations governing the extra virgin grade. “The mere fact that this widely respected organization has written about extra virgin olive oil will further the dialogue about olive oil quality in North America,” Mueller added.

However, the review commits several serious errors, he suggested, “and perpetuates some fundamental, age-old confusions about olive oil.” For one thing, having defined extra virgin as flawless, the report

proceeds to include flawed oils in their “poor,” “fair,” and even “good” categories, “sending a badly mixed message to consumers and oil buyers,” he said. The article also fails to explain several key factors in olive oil quality, he noted.

“Olive oil is closer to fresh-squeezed fruit juice than refined vegetable oil, yet the review doesn’t stress the importance of freshness when buying oil (no word on a harvest date or even ‘best by’ dates on the label). They omit the fact that flawed oils are likely to be short on the therapeutic properties people typically expect in olive oil, and that the positive attributes of true extra virgins—bitterness, pungency, and fruitiness—signal the presence of these very properties,” added Mueller.

The suggestion by the magazine that consumers cook with defective oils, or “nonvirgin” oils, because their flaws “might not be noticed with food” is, simply put, both poor and unhealthy advice, said Mueller. “CR mentions chemical tests for quality, yet gives no indication that they performed any lab tests; their sensory tests were performed by two ‘experts’ whose credentials are nowhere explained.”

Because the taste test is part of the legal definition of extra virgin olive oil, and rigorously defined under international law, these credentials are important.

“CR is right to guard its independence,” Mueller concluded, “but this apparent lack of consultation with olive oil authorities seriously undermines the value of this report for consumers.”

## Glucosinolate-free rapeseed meal?

A team of researchers from the University of Copenhagen has developed a method to hinder glucosinolates from entering the edible parts

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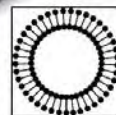
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of oilseeds. Their work increases the potential of rapeseed meal as a commercial animal feed for pigs and chickens.

"We have developed an entirely new technology that we call 'transport engineering.' It can be used to eliminate unwanted substances from the edible parts of crops," explains Barbara Ann Halkier, head of the Center of Excellence for Dynamic Molecular Interactions at the University of Copenhagen's Faculty of Science.

Rapeseed is only one example of a crop whose use will be greatly enhanced thanks to the new technology, the researchers say. Unlike the healthful glucosinolates found in broccoli, oilseed rape produces a glucosinolate that is harmful to most animals when consumed in larger amounts.

"We managed to find two proteins that transport glucosinolates into the seeds of the thale cress plant, a close relative of the oilseed rape. When we subsequently produced thale cress without these two proteins, the remarkable result was that their seeds were completely free of glucosinolates and thus suitable for feed," Halkier said.

Bayer CropScience is now negotiating with the University of Copenhagen's Tech Transfer Unit to collaborate with the research group to deploy the new technology and produce an oilseed rape plant with glucosinolate-free seeds. According to Bayer CropScience project leader Peter Denolf, such seeds will significantly enhance the use of oilseed rape meal as animal feed and bring along a more sustainable oilseed rape processing procedure.

The results are the result of 16 years of basic research, and appeared in *Nature* (doi:10.1038/nature11285, 2012). ■

## Blubber not

In 1768, 4,000 tons of whale oil were exported from Boston to England. The oil was obtained from the blubber in most species, but in sperm whales it came from the head cavity. The oil's primary use as a lamp fuel was eliminated with the development of kerosene during the following century, and petroleum-based waxes and oils later replaced it for most other uses.



# Sustainability watch

## P&G to work with US EPA

The Procter & Gamble Co. (P&G; Cincinnati, Ohio, USA) and the US Environmental Protection Agency (EPA) National Risk Management Research Laboratory (NRMRL) announced in late July 2012 their signing of a five-year Cooperative Research and Development Agreement (CRADA) "to develop new tools to optimize sustainability improvements in manufacturing facilities, and their associated supply chains."

These improvements will directly address the end points of P&G's long-term environmental sustainability vision, announced in September 2010. According to a P&G statement, "this vision includes: powering its plants with 100% renewable energy; using 100% renewable materials or recycle for all its products and packaging; having zero consumer or manufacturing waste going to landfills; and designing products that delight consumers while maximizing the conservation of resources."

To meet this commitment, new methods and tools are needed to help optimize design and decision making across a wide range of operations and supply choices, as well as various environmental sustainability measures. The EPA has developed a comprehensive list of sustainability metrics and performance indicators that can be used to quantify sustainability in a manufacturing and supply chain context, while P&G has a diverse set of manufacturing operations and supply chains that can be leveraged to optimize how such metrics are used to guide improvement choices. The work under this CRADA will combine P&G's manufacturing and supply chain knowledge with the EPA's work on metrics to develop a modeling tool that can be used to assess future product design, material sourcing, and manufacturing options.

P&G will be developing this framework based on metrics associated with its tissue and towel products. "This is a tremendous opportunity for us to be at the leading edge of developing tools to support the entire company's effort to improve the sustainability of our products and our operations," noted Stefano Zenezini, vice-president of product supply for P&G's business unit that makes Charmin, Bounty, and Puffs. "We've made great progress in areas like energy and water use reduction, but really need these new tools to help guide the increasingly complex choices we will be making as we continue to strive to meet the vision the company has committed to."

■ ■ ■

The world's food systems and diets are anything but sustainable, according to a book published in August 2012 by the United Nations Food and Agriculture Organization (FAO) and Bioversity International. Bioversity conducts research on agricultural biodiversity and works to improve the lives of smallholder farmers and rural communities from its headquarters in Rome.

The book defines "sustainable diets" as having "... low environmental impacts [that] contribute to food and nutrition security and to healthy life for present and future generations ... [and that] are protective and respectful of biodiversity and ecosystems, culturally acceptable, accessible, economically fair and affordable, nutritionally adequate, safe and healthy, while optimizing natural and human resources ... ." The 309-page book comprises the proceedings of an FAO-sponsored symposium titled "Biodiversity and Sustainable Diets United Against Hunger" held November 3–5, 2010. The book is available online (PDF) at <http://tinyurl.com/FAO-diet>. ■



# Biofuels News



Aviam Ltd., a joint venture formed by Italian and Mozambican interests, is likely to lose its license to plant jatropha in 10,000 hectares of land in the northern province of Nampula, Mozambique. Aviam had pledged to invest \$20 million in the jatropha plantation, whose products would be used to produce biofuels. However, as of mid-July 2012, the company was not meeting the agreed-on timetable for implementing the project. According to *Notícias*, the government newspaper in Maputo, Mozambique, the company has planted only 150 hectares with jatropha. Additionally, construction has not yet begun on the factory that was promised to process the seeds into biodiesel.

■■■

According to EurObservER ([www.eurobserv-er.org/pdf/baro212.pdf](http://www.eurobserv-er.org/pdf/baro212.pdf)), the governments making up the European Union (EU) no longer view the rapid increase in biofuel consumption as a priority. Instead, the EU is focusing on setting up sustainability systems to verify that the biofuel used in the various countries complies with the sustainability criteria of its Renewable Energy Directive. Between 2010 and 2011 biofuel consumption increased by 3%, or 13.6 million metric tons of oil equivalent (Mtoe) used in 2011 compared with 13.2 Mtoe in 2010.

■■■

Mark Bünger, research director for Lux Research in Boston, Massachusetts, USA, was lead author of a report titled "Pruning the Cost of Bio-Based Materials and Chemicals" (<http://tinyurl.com/LuxResearch-algae>). Among the findings in the report, the model used for computation found that algal cultivation is cost intensive, owing to high capital costs for growing algae at industrial scale. The model estimated that growing algae costs \$202,000 per hectare.

■■■

The São Paulo newspaper *Valor Econômico* reported on August 21 that agricultural commodities trader Cargill would start commercial biodiesel operations by the end of August 2012 in the Tres Lagoas municipality, Mato Grosso do Sul state (Brazil). Cargill has

## Pongamia being established in USA

Since 2009, TerViva Inc. (Oakland, California, USA) has been acquiring acreage in the states of Texas, Florida, Arizona, and Hawaii for the purpose of establishing farms of pongamia (*Milletia pinnata*, formerly known as *Pongamia pinnata*) trees. This nitrogen-fixing legume is native to Australia and India. The trees produce a nut crop that can be harvested annually by using conventional shakers, such as those used by growers of almonds and other nuts. The tree is frost tolerant, but not impervious to freezing. It grows in most soils, is drought tolerant, and has a productive life exceeding 50 years.

The seed has about 40% oil content, which can be converted to biodiesel or aviation fuel. The oil contains as much as 50% oleic acid, another possible and economically important market for the oil. The company claims that at maturity the trees can produce enough nuts to yield 400 gallons of oil per acre (3,700 liters/hectare) per year.

TerViva, along with Mason & Morse Farmland Group (a US real estate company), is promoting pongamia as a replacement crop for Florida citrus crops, which have been severely limited by greening disease. Caused

by the bacterium "*Candidatus Liberibacter asiaticus*," which was first identified in the United States in 2005, greening disease has led to the abandonment of thousands of acres of Florida citrus land.

## Responsible biomass harvesting for ethanol production

Researchers from Iowa State University (ISU; Ames, USA), and the US Department of Agriculture continue to find that their soil data from Emmetsburg, Iowa, show that harvesting crop residue can be a responsible part of good farm management. Data have been collected in conjunction with the scale-up of the Project Liberty cellulosic ethanol plant being constructed by POET-DSM in Emmetsburg.

Stuart Birrell, associate professor of agricultural and biosystems engineering at ISU, said, "Basically at the removal level that POET-DSM recommends, there is no reduction in yield, and removal rates are well within the sustainability limits." The requirement for replacing nutrients is minimal, according to Birrell. Potash may be added at 10–15 pounds per acre (10.3–15.5 kg/hectare) when soil

tests indicate a need, but for nitrogen there has been no evidence of a need.

POET-DSM contracts for about one ton of biomass per acre (2.2 metric tons/hectare) with participating farmers, or less than 25% of the available above-ground biomass. The company has contracted for 85,000 tons (77,000 metric tons) in 2012, and once operational, will require about 285,000 tons per year.

Birrell added, "The natural soil fertility and understanding of that fertility is far more important and has a greater effect on fertilizer recommendation rates than the relatively small amounts you're taking off in stover harvesting"

## Lufthansa continues to investigate biofuel

From July 15 through December 27, 2011, Lufthansa German Airlines fueled an Airbus A321 that flew between Hamburg and Frankfurt with biofuel (*inform* 22:497-499, 2011; 23:149, 2012). One of the two engines ran exclusively on a 50:50 blend of synthetic biofuel and standard aviation fuel, and the other on conventional Jet A1. The plane made the one-hour trip as many as eight times a day, and in that period consumed 1,556 metric tons of biofuel in total (<http://tinyurl.com/Lufthansa-evaluates>).

The company was satisfied with their trial of biofuel. Not only did it find that jatropa-, camelina-, and animal fat-based biofuel had no negative side effects on equipment and operations, it also showed a 1%

improvement in specific fuel consumption owing to the higher energy density of the biobased fuel compared with Jet A1. Lufthansa is now looking at ways to acquire sufficient quantities of biofuel in the future.

In June a representative of Joachim Buse, vice-president of aviation biofuel at Lufthansa, traveled to Russia to inspect camelina fields in the Volga region, where the plant is being tested for its commercial possibilities. Oil mills have not yet been built in the area to process the crops but Lufthansa judged the area as "very promising" (<http://tinyurl.com/Lufthansa-evaluates>).

Lufthansa is not confining itself to one potential area for growing oil-producing plants. FlightGlobal.com reported that Buse said, "Africa still offers very good field availability in the semi-arid, subtropical zone" (<http://tinyurl.com/Lufthansa-evaluates>). Such areas would be best suited to jatropha.

The Malaysian newspaper TheStar.com reported an August 15, 2012, interview with Lufthansa's Daniel Riefer, project manager for aviation biofuel. He was asked whether Lufthansa would consider palm oil from a country such as Malaysia for the airline's needs. He replied, "That could be a possibility. But we have not yet decided on what vegetable oil-based-fuel to use" (<http://tinyurl.com/palm-Lufthansa>).

## Gutter oil as biofuel?

An article in *inform* earlier in 2012 (23:115, 128) discussed the food safety issue involved with China's "gutter oil." This recycled cooking oil, or gutter oil, derives from the refinement of kitchen waste, collected

CONTINUED ON NEXT PAGE

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spent \$64.5 million to get the new facility started. It is capable of producing 700,000 liters of biodiesel per day, all of which will come from soybean oil. Family farmers living in the region will supply a quarter of the raw material.



Petrobras Biocumbustível, the biodiesel subsidiary of Brazil's state-owned oil and gas producer Petrobras, is discussing with the Vale mining company the possibility of setting up a palm oil and biodiesel production partnership. Vale is already extracting palm oil in the Moju municipality of Pará state. Vale's plans before talking with Petrobras had been to produce enough biodiesel (for an 80:20 blend of petrodiesel and biodiesel) to fulfill its own needs as of 2015. Petrobras Biocumbustível is also building a biodiesel plant in Pará, incorporating enough capacity to meet the demand of Brazil's northern region.



The US Environmental Protection Agency (EPA) announced in early August that it had granted registration for Soladiesel RD fuel (Solazyme, South San Francisco, California). This registration enables the commercial sale of SoladieselRD in either blended or unblended forms. "To our knowledge, SoladieselRD is the first 100% algal-based fuel in history to receive an EPA fuel registration for use as a 'neat' or unblended product," said Bob Ames, vice president of fuels commercialization for Solazyme. In tests conducted for the EPA registration, SoladieselRD met or exceeded the ASTM D975 specification for conventional diesel fuel. SoladieselRD performed substantially similar to petroleum diesel and even reduced NO<sub>x</sub> emissions by approximately 10%. ■

## What's new with you?

Retiring? Moving? Celebrating an anniversary? Recent promotion? New child? Won an award? AOCS wants to help you spread the good news. Let us know what's going on. Email us and we'll share your news in the next AOCS member newsletter. Contact Nicole Philyaw at [nicolep@aocs.org](mailto:nicolep@aocs.org)

from gutters and drains; animal fat; and oil that has been repeatedly used to fry foods. With enough cleaning, the oil can meet sanitation standards of cooking oil, even though significant levels of toxic substances remain in the oil.

Now, however, Chinese firms are investigating the possibility of channeling these recycled oils into high-grade fuel for jet aircraft (<http://tinyurl.com/GutterOil-Fuel>). SkyNRG (Amsterdam, Netherlands) originally bought some 20 metric tons of waste oil-derived biofuel from Qingdao's Fresh Bio-Energy Technology Development Company Ltd., in Shandong province, in November 2011. This was shipped to Europe for further testing and refinement.

In July 2012, news appeared of further talks between SkyNRG and authorities and companies in Shanghai to purchase more used cooking oil for refinement into aviation fuel. Dereck Kronemeijer, general manager of SkyNRG, told the *Southern Metropolis Daily* on July 11 in Guangzhou that no deals were officially signed yet (<http://tinyurl.com/SkyERG-China>).

A report appeared in the weekly Chinese newspaper *The Economic Observer* on August 3 that Zhenhai Refining and Chemical Co., a unit of China Petrochemical Corp. (Sinopec Group), the largest oil producer in the country by sales revenue, is also seeking to produce aviation fuel from used cooking oil (<http://tinyurl.com/Zhenhai-gutter-oil>). Zhenhai has already completed production trials and expects a formal review by China's aviation authorities in January 2013. It has the capacity to produce 20,000 metric tons of aviation fuel annually from used cooking oil, according to the report.

*The Economic Observer* also indicated that Zhenhai Refining is talking with restaurant chains such as McDonald's to establish a guaranteed supply of used cooking oil.

To provide some perspective on this information, the *International Business Times* (<http://tinyurl.com/GutterOil-Fuel>) reports that 70% of waste cooking oil in the United States is collected and eventually recycled. In China, only 2% is collected at present.

## Thieves target used cooking oil

*The Philadelphia Inquirer* newspaper (Pennsylvania, USA) reported in late July that thieves are increasingly targeting area restaurants' used cooking oil and selling it to companies that

collect it and convert it into biofuels. The frequency of these thefts has become so great that legitimate companies are communicating with each other to ensure that the oil they buy has not been stolen. The problem may become moot sometime in the future, however, for Chris Moyer, sustainability manager for the National Restaurant Association, was quoted by the *Inquirer* as saying that some restaurants are installing equipment to process old cooking oil into energy for their own use (<http://tinyurl.com/waste-oil-PhilaInquirer>).

## How "green" is rapeseed oil?

A study by Gernot Pehnelt and Christoph Vietze, published jointly by the Friedrich Schiller University and Max Planck Institute of Economics in Jena, Germany, is questioning the claim by the European Commission (EC) that rapeseed oil is sustainable enough to be used in the European Union (EU). The authors, who are economists, have based their enquiry on the criterion of the Renewable Energy Directive (RED) of the European Union: The directive says that, until 2017, greenhouse gas (GHG) emissions associated with production and use of biofuels must be at least 35% lower than those associated with production and use of conventional fuels to be classified as "sustainable." After 2017, the value is scheduled to rise to 50%. Only sustainable biofuels are eligible for the mandatory blending scheme applied within the EU.

The EC's own studies found that rapeseed oil-based biofuel produced reductions in GHG emissions of 38% compared with conventional fuels.

Using the same default values that the EC used, Pehnelt and Vietz found that in most cases their calculations of savings in GHG emissions on locally produced rapeseed biodiesel were under 30% (<http://tinyurl.com/Guardian-sustainability>). According to the journal *Nature*, the authors considered variations in soil quality and fertilizer application during the growing season as well as different efficiencies in fuel production (<http://tinyurl.com/rapeseed-fails>). They also looked at values for emissions found in the literature as well as the default values used by the EC. In the abstract to their study, Pehnelt and Vietz said: "Therefore, the GHG emissions savings values of biodiesel stated by the EU are more than questionable" ([pubdb.wiwi.uni-jena.de/pdf/wp\\_2012\\_039.pdf](http://pubdb.wiwi.uni-jena.de/pdf/wp_2012_039.pdf)). ■

## Briefs

In July 2012, the US Food and Drug Administration approved Amarin Corp.'s Vascepa™ fish oil capsules as a prescription supplement for reducing triglyceride levels in adult patients with severe hypertriglyceridemia (very high triglyceride levels). Vascepa is a purified marine-oil preparation consisting of "not less than 96%" eicosapentaenoic acid, an omega-3 fatty acid found in fatty cold-water fish and some algae. Vascepa joins GlaxoSmithKline's Lovaza as the only by-prescription-only fish oil products on the US market. Citi Investment Research forecast that the drug could reach sales as high as \$2.6 billion and noted that Lovaza currently has annual sales of \$1 billion.

■■■

In the past decade, children and teens aged 6 to 19 in the United States have seen significant reductions in their levels of total and LDL (low-density lipoprotein) cholesterol, according to a study in the *Journal of the American Medical Association* (308:591–600, 2012). The researchers, led by Brian Kit of the Center for Disease Control's National Center for Health Statistics, studied cross-sectional data on 16,116 children and teens who participated in the National Health and Nutrition Examination Survey. Kit told WebMD.com that the emphasis on replacing *trans* fats and other saturated fats with more healthful fats in commercially available foods, as well as public health initiatives to reduce secondhand smoke exposure, may have contributed to the decline.

■■■

A review of more than 100 studies linking chocolate to health benefits suggests that chocolate does indeed improve brain functioning and mood. The review, published in the *British Journal of Pharmacology* (doi:10.1111/j.1365-2125.2012.04378.x, 2012), bolsters the case for cocoa and chocolate being considered as nutraceuticals. (A nutraceutical is a food, or a part of a food, that provides medical or health benefits.) For *inform's* recent article on the secrets of Belgian chocolate, see <http://tinyurl.com/inform-choco>. ■

# Health & Nutrition News



## Fat and flavor perception

A joint study carried out by The University of Nottingham and Anglo-Dutch consumer products giant Unilever has found for the first time that fat in food can reduce activity in several areas of the brain that are responsible for processing taste, aroma, and reward.

This three-year study investigated how the brains of a group of participants in their 20s responded to changes in the fat content of four different fruit emulsions they tasted while undergoing a magnetic resonance imaging test. All four samples were of the same thickness and sweetness, but one contained flavor with no fat, while the other three contained fat with different flavor-release properties.

The research found that the areas of the participants' brains that are responsible for the perception of flavor—such as the somatosensory cortices and the anterior, mid and posterior insulae—were significantly more activated when the nonfatty sample was tested compared to the fatty emulsions despite having the same flavor perception. It is important to note that increased activation in these brain areas does not necessarily result in increased perception of flavor or reward.

Joanne Hort, associate professor in sensory science at The University of Nottingham and one of the researchers, said: "This is the first brain study to assess the effect of fat on the processing of flavor perception and it raises questions as to why fat emulsions suppress the cortical response in brain areas linked to the processing of flavor and reward. It also remains to be determined what the implications of this suppressive effect are on feelings of hunger, satiety, and reward."

Unilever food scientist Johanneke Busch, based at the company's research and development laboratories in Vlaardingen, Netherlands, added: "There is more to people's enjoyment of food than the product's flavor—[such as] its mouthfeel, its texture and whether it satisfies hunger, so this is a very important building block for us to better understand how to innovate and manufacture healthier [*sic*] food products [that] people want to buy."

The UK's Biotechnology and Biological Sciences Research Council co-funded the study. It appeared in *Chemosensory Perception* (doi:10.1007/s12078-012-9130-z, 2012) and was led by Sally Eldeghaidy of The University of Nottingham.

CONTINUED ON NEXT PAGE



## Fat-rich salad dressings bring absorption benefits

Monounsaturated fat-rich dressings may help the body absorb the most carotenoids from a salad, according to a study appearing in *Molecular Nutrition & Food Research* (doi: 10.1002/mnfr.201100687, 2012). In a human trial, researchers fed 29 subjects salads topped off with butter as a saturated fat, canola oil as a monounsaturated fat, or corn oil as a polyunsaturated fat. Each salad was served with 3 g, 8 g, or 20 g of fat from dressing. The participants' blood was tested for absorption of fat-soluble carotenoids—compounds such as lutein, lycopene,  $\beta$ -carotene, and zeaxanthin.

The absorption benefits of soybean oil were the most dependent on dose. The more fat on the salad, the more carotenoids the subjects absorbed. The butter was also dose-dependent but to a lesser extent. Monounsaturated fat-rich dressings such as canola- and olive oil-based dressings promoted the equivalent carotenoid absorption at 3 g of fat as it did 20 g, suggesting that this lipid source may be a good choice for those craving lower-fat

options but still wanting to optimize absorption of health-promoting carotenoids from fresh vegetables.

“Even at the lower fat level, you can absorb a significant amount of carotenoids with monounsaturated fat-rich canola oil,” said Mario Ferruzzi, the study's lead author and associate professor of food science at Purdue University in West Lafayette, Indiana, USA. “Overall, pairing with fat matters. You can absorb significant amounts of carotenoids with saturated or polyunsaturated fats at low levels, but you would see more carotenoid absorption as you increase the amounts of those fats on a salad.”

Ferruzzi and colleagues will next work on understanding how meal patterning affects nutrient absorption. He is trying to determine whether people absorb more nutrients if they eat vegetables at one time or if consumption is spread throughout the day.

## EFSA sets upper intake level for LC-PUFA

In July 2012, the European Food Safety Authority (EFSA) published a Scientific Opinion on the tolerable upper intake level (UL) for eicosapentaenoic acid (EPA), docosahexaenoic acid (DHA), and docosapentaenoic acid omega-3 fatty acids. The opinion stated that available data are insufficient to establish a UL for the omega-3 long-chain polyunsaturated fatty acids (LC-PUFA), individually or combined, for any population group.

IngredientsNetwork.com noted that the decision came after a 2011 study by the German Federal Institute for Risk Assessment (BfR) reported that high consumption of omega-3 fortified foods could exceed the 1.5 g recommended daily intake of DHA and EPA. BfR estimated that between 3.7% and 16.7% of Germans could consume potentially health-damaging levels of DHA and EPA through excessive consumption of omega-3-fortified foods.

The EFSA panel noted: “At observed intake levels, consumption of omega-3 LC-PUFA has not been associated with adverse effects in healthy children or adults.” The panel specified that there was no significant risk with long-term supplemental intakes of EPA and DHA combined up to about 5 g/day; it also qualified that supplemental intakes up to 1.8 g/day of EPA alone do not raise safety concerns for the adult population and up to 1 g/day for

DHA alone does not raise any safety concerns for the general population.

In related news, the Swiss government acted in June 2012 via SR 817.022.104 (Regulation on Special Food) to adopt an upper limit of 250 mg of EPA and DHA intake for the general population and up to 400 mg EPA and DHA for pregnant and lactating women. The Global Organization for EPA and DHA, a trade association based in Salt Lake City, Utah, USA, said it will approach the Swiss government with the EFSA opinion to ask them to reconsider their position, according to a report by NaturalProductsInsider.com.

July 2012 also marked the end of EFSA's massive review of the almost 3,000 potential health claims submitted by industry. In the final group of five opinions, EFSA rejected the application regarding soy isoflavones and positive outcomes in a number of health conditions including menopause, reducing low-density lipoprotein cholesterol, and the delivery of antioxidant benefits.

A month earlier, EFSA upheld its scientific opinion that evidence was lacking that consumption of olive oil polyphenols (standardized by the content of hydroxytyrosol and its derivatives) maintained normal blood HDL (high-density lipoprotein) concentrations.

Also in June 2012, EFSA approved a health claim related to the consumption of cocoa flavanols and maintenance of normal endothelium-dependent vasodilation. EFSA established the following wording as reflecting the scientific data: “Cocoa flavanols help maintain endothelium-dependent vasodilation, which contributes to normal blood flow.” The agency also noted that “in order to obtain the claimed effect, 200 mg of cocoa flavanols should be consumed daily. This amount could be provided by 2.5 g of high-flavanol cocoa powder or 10 g of high-flavanol dark chocolate, both of which can be consumed in the context of a balanced diet. The target population is the general population.”

## Edible stop signs

Once you pop the top of a tube of potato chips, it can be hard to stop munching its contents. But Cornell University researchers may have found a novel way to help: edible serving-size markers that act as subconscious stop signs.

As part of an experiment carried out on two groups of college students (98 students total) while they were watching video clips in class, researchers from Cornell's Food and



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Brand Lab served tubes of potato chips, some of which contained chips dyed red. Researchers found that the red chips served as subconscious “stop signs” that curtailed the amount of food consumed.

In the first study, the red chips were interspersed at intervals designating one suggested serving size (seven chips) or two serving sizes (14 chips); in the second study, this was changed to five and 10 chips.

Unaware of why some of the chips were red, the students who were served those tubes ate 50% less than their peers did.

“People generally eat what is put in front of them if it is palatable,” said Cornell Food and Brand Lab Director Brian Wansink. “An increasing amount of research suggests that some people use visual indications such as a clean plate or bottom of a bowl to tell them when to stop eating.”

Wansink, known for his clever studies on eating cues, was the subject of an *inform* feature in 2005 (see <http://tinyurl.com/Wansink-2005>).

## Almonds in the news

It has been a productive several months for almond-related research. Among the results: Suggestions that almonds contain 20% fewer calories than previously thought have led to a call to overhaul the global calorie system.

A study conducted by scientists from the US Department of Agriculture (USDA) and released in the *American Journal of Clinical Nutrition* (doi: 10.3945/ajcn.112.035782, 2012) provides a new understanding of almonds’ calorie count.

David Baer and his team from USDA’s Agricultural Research Service used a new method of measuring the calories in almonds, which built on traditional methods and allowed

the researchers to determine the number of calories actually digested and absorbed from almonds. Resulting data showed a one-ounce serving of almonds (about 23 almonds) has 129 calories vs. the 160 calories currently listed on the Nutrition Facts Panel. The results may have implications for certain other foods as well.

The new study’s results support previous research indicating that the fat in almonds is not absorbed as easily as the fat in most other foods, due to almonds’ natural cellular structure. This implies that traditional methods of calculating calories overstate those calories coming from almonds because they do not account for the fact that fat digestibility from nuts is less than that from other foods.

In fact, the same research team also recently conducted a similar study using pistachios, finding a 5% decrease in pistachios’ calorie count compared to the 20% decrease in almonds’.

Most often, foods’ calorie counts are calculated based on a system developed by Atwater and colleagues more than 100 years ago. Known as the Atwater general factors, the system assigns calorie values for every gram of protein, fat, and carbohydrate found in a given food (4 kcal/g for protein, 9 kcal/g for fat, and 4 kcal/g for carbohydrate).

However, as the new study notes, “There have been few, if any, studies that looked at the calorie value of whole food within a mixed diet that could confirm . . . Atwater’s coefficients.” So, for this study, the researchers expanded on Atwater’s approach, using a specially designed diet and new method of calculation that allowed them to extrapolate the calories from almonds eaten as part of a full diet.

In the study’s discussion section, the authors considered the potential implications of substituting other foods with almonds in a

calorie-controlled study. Based on the data: “When an 84-g serving of almonds was incorporated into the diet daily, the energy digestibility of the diet as a whole decreased by 5%. Therefore, for individuals with energy intakes between 2,000 and 3,000 kcal/day, incorporation of 84 g almonds into the diet daily in exchange for [the same number of calories from] highly digestible foods would result in a reduction of available energy of 100–150 kcal/d. With a weight-reduction diet, this deficit could result in more than a pound of weight loss per month.”

In related news, a study published in the *Journal of Food Science* (doi: 10.1111/j.1750-3841.2012.02706.x, 2012) looked at the effects of storage conditions on the oxidation of lipids in California almonds. The researchers used whole almonds with or without polyethylene (PE) packaging, blanched whole almonds, and sliced almonds with PE packaging. The almonds were kept in 10 different storage conditions with different temperatures and relative humidity levels. The peroxide values, iodine values, and free fatty acid levels (FFA) were monitored during the storage.

The study found that the peroxide values of the whole almond samples did not change significantly, whereas the blanched and sliced samples changed greatly, suggesting that skins may play an important protective role. Almond skins are rich in antioxidants that can fight against oxidation. Additionally, almond skins act as an oxygen barrier, which also helps minimize oxidation.

The researchers concluded: “FFA alone is not a reliable indicator of rancidity. The skins were very effective in preventing FFAs from further oxidation. On the other hand, blanching, which is believed to inactivate enzymes in the almonds, did not provide much protection against autooxidation of lipids.” ■

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Novozymes has developed a fungus that enables the production of malic acid ( $\text{HO}_2\text{C}-\text{CH}_2-\text{CHOH}-\text{CO}_2\text{H}$ ) from renewable raw materials instead of oil. Malic acid is used as a flavor enhancer in the food industry and can be converted into chemical derivatives used for a variety of plastic, polymer, and resin products. The company is out-licensing the technology to partners who are interested in producing and commercializing malic acid and derivatives made from renewable materials.

■■■

The last remaining application in Turkey for approval to import or use genetically modified (GM) food, made by the ingredients firm Ünak Gıda, was withdrawn in mid-August. This development followed a decision earlier in the month by the Federation of Food and Drink Industry Associations of Turkey (TGDF) to drop an application to import 29 kinds of GM organisms. According to FoodNavigator.com (<http://tinyurl.com/Turkey-GMO>), TGDF specifically cited public opinion and the unfavorable reaction of non-governmental organizations (such as Greenpeace Mediterranean) as the major reason for the decision.

■■■

Argentina has approved the use of Monsanto's genetically modified Roundup Ready 2 soybean seeds in an effort to increase crop production. The approval must appear in the government's official gazette before it can take effect, and Agriculture Secretary Lorenzo Basso told Reuters news service that this would happen before the end of August 2012 (<http://tinyurl.com/RRsoybean-Argentina>). Argentine farmers readily adopted Roundup Ready seeds in the 1990s, but Monsanto never patented the technology in the country, leading to a long legal battle to try to charge royalties. Since then, tensions have eased.

Earlier in the third quarter, Argentina also approved the use of a new variety of GM corn, developed by Dow AgroSciences and Monsanto. In addition, Paraguay announced in August that it will approve the use of Monsanto's Roundup Ready 2 soybean seeds before the end of 2012 as well as new corn technology to increase the country's position as a grains exporter (<http://tinyurl.com/RRsoybean-Paraguay>). ■

# Biotechnology News



*Soil microbes may play a role in the way weeds react to the herbicide glyphosate. The giant ragweed plant growing in sterilized soil void of microbes (left) was barely affected by the herbicide, while the one in unsterilized soil (right) succumbed. (Photo provided by Jessica Schafer)*

## Soil microbes affect glyphosate resistance?

Scientists seeking to understand how “superweeds” acquire resistance to the herbicide glyphosate have reported that soil microbes may play a role.

At Purdue University (West Lafayette, Indiana, USA) weed scientists Steven Hallett and William Johnson and botany graduate student Jessica Schafer have pointed out that most laboratory tests done to understand glyphosate resistance are done in sterile soil (*Weed Sci.* doi: <http://dx.doi.org/10.1614/WS-D-12-00050.1>).

They carried out tests on glyphosate-resistant and -susceptible biotypes of three problematic weeds of the Midwestern United States: giant ragweed, horseweed,

and common lambsquarters. Dose-response experiments were conducted in a greenhouse using sterile and nonsterile field soil. Responses measured were dry weights of roots and shoots.

Soil microbes influenced the response of the susceptible and resistant giant ragweed biotypes and the susceptible common lambsquarters, but not the tolerant common lambsquarters or either horseweed biotype. The different response of the three species to glyphosate in the presence and absence of soil microbes demonstrates that rhizosphere interactions are fundamental to the mode of action of glyphosate.

These findings suggest that the range of tolerance to glyphosate observed in weeds and the evolution of resistance in weed biotypes may also be influenced by rhizosphere interactions.

## Gates Foundation funds GM research

The Bill & Melinda Gates Foundation is providing \$9.8 million to the John Innes Centre (Norwich, UK) to investigate whether a symbiosis can be created between cereal crops and bacteria. The purpose would be to create associations between plant roots and bacteria that fix nitrogen gas from the air, converting it to combined forms ( $\text{NH}_4^+$ ,  $\text{NO}_3^-$ ) that the plants can use to increase yields.

The most immediate benefit would be for subsistence farmers, who cannot afford to purchase nitrogen fertilizer. Without this fertilizer, yields at present are 15–20% of what they could be, according to a statement released by the Innes Centre (<http://tinyurl.com/N2fixation-Gates>).

The new research will investigate the possibility of engineering cereals to associate with nitrogen-fixing bacteria and of delivering this technology through the seed. If it is found to work, farmers would be able to share the technology by sharing seed. Another aspect the researchers hope to address is the use of grasses as rotational crops to enhance soil nitrogen.

The initial focus of the investigation will be corn, the most important staple crop for small-scale farmers in sub-Saharan Africa.

## DuPont Pioneer Hi-Bred loses in Monsanto patent case

In May 2009, Monsanto Co. sued DuPont Pioneer, known also as Pioneer Hi-Bred International, Inc., for patent infringement. Monsanto's purpose was to block DuPont from combining the Roundup Ready trait, for which DuPont already had a license from Monsanto, with its own technology, Optimum GAT. The GAT trait was designed to make soybeans containing it tolerant to so-called ALS-herbicides, on top of tolerance to Monsanto's glyphosate (Roundup Ready) herbicide.

The suit went to trial on July 9, 2012, in US District Court in St. Louis, Missouri (*inform* 23:510, 2012). After a 3 ½-week trial, the eight-person jury deliberated 45 minutes on August 1 and then announced its verdict, which went against DuPont. As part of the decision, Monsanto was awarded \$1 billion, the fourth-largest jury award in a patent trial in US history, according to Bloomberg.com (<http://tinyurl.com/Bloomberg-4thLargest>).

Furthermore, the jury found that DuPont had willfully infringed the Monsanto patent, which means the damages could be increased in the future.

During the trial, Hugh Grant, the chief executive officer of Monsanto, told the jury that DuPont asked Monsanto for the ability to "stack" the GAT technology with the Roundup Ready technology. Monsanto countered by offering to sell the technology for a \$1.5 billion lump sum. DuPont rejected the offer (<http://tinyurl.com/DuPont-stack>). According to Monsanto lawyers, DuPont continued to conduct research on stacking traits anyway.

In a company statement, Monsanto's Executive Vice President and General Counsel David Snively said, "The materials uncovered from DuPont files during this case highlight that DuPont's senior leaders were actively working to hide the fact their OGAT [Optimum GAT] technology had failed and were using elaborate schemes to cover that up with the unlicensed use of our technology" (<http://tinyurl.com/Snively-Monsanto>).

In its company statement (<http://tinyurl.com/DuPont-appeal>), DuPont said, "DuPont believes that the evidence presented during the trial demonstrated clearly that Monsanto's Roundup Ready® soybean patent (RE 39,247) is invalid and unenforceable and that Monsanto intentionally deceived the United States Patent and Trademark Office on several occasions as it sought patent protection. Further, DuPont believes that the damages awarded of \$1 billion are unjustified, particularly considering that Pioneer has never sold a single Optimum® GAT® seed and has no plans to do so in the future."

DuPont said it will "appeal at the earliest possible opportunity and expects to overturn this verdict" (<http://tinyurl.com/DuPont-appeal>).

Monsanto and DuPont are already set to meet again in the same court in 2013 to adjudicate DuPont's accusation in another case that Monsanto has been engaging in anti-competitive behavior (<http://tinyurl.com/DuPont-stack>).

## New method to assess plant drought tolerance

Collaborators from the University of California-Los Angeles (UCLA; USA) and Xishuanbanna Tropical Gardens (Yunnan, China) have developed a new method for quickly assessing the drought tolerance of plants. The method is based on a trait known as "turgor loss

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point.” During drought, the water in leaf cells becomes harder to replace. The turgor loss point is reached when leaf cells become so dehydrated their walls become flaccid. That is, the leaf becomes limp and wilted, and the plant cannot grow.

As described in a statement from UCLA (<http://tinyurl.com/measure-TurgorLoss>) the growth of plants depends on their ability to withstand the evaporative loss of water when they open their stomata to take in CO<sub>2</sub> for photosynthesis. The amount of evaporation a plant can tolerate depends on the water pressure inside of its cells, which in turn depends on (i) its turgor potential; (ii) the pushing force of water against the inside of the cell walls, and (iii) the osmotic potential inside the cell; that is, the pulling force of dissolved salt molecules on the water molecules.

Christine Scoffoni, a UCLA graduate student working on the project, pointed out that plant cells need to maintain their turgor pressure to hold up their cell walls, but with evaporation they lose turgor pressure (<http://tinyurl.com/TurgorLoss-measure>).

At the turgor loss point, saltier cells have a stronger pulling force holding the water molecules inside the cell. Plants with saltier cells can keep their stomata open in drier conditions. Thus, the turgor loss point is a determinant of a plant's drought tolerance.

The UCLA-China team developed a quick method of measuring a pressure-volume (p-v) curve for cell saltiness that involves freezing small discs of leaf tissue to break the cell walls, thawing the samples, and mixing the cell sap. The saltiness of the cell sap can then be measured with an osmometer in 10 minutes. Earlier methods of determining p-v curves required nearly constant attention from a researcher for up to two days.

For further information, see the original online paper: M.K. Bartlett, C. Scoffoni, R. Ardy, Y. Zhang, S. Sun, K. Cao, and L. Sack, Rapid determination of comparative drought tolerance traits: using an osmometer to predict turgor loss point, *Methods in Ecology and Evolution*, doi: 10.1111/j.2041-210X.2012.00230.x.

## Companies funding efforts to defeat GMO labeling in California

On August 15, the *Sacramento Bee* newspaper (<http://tinyurl.com/contributions-Prop37>)

published a list of contributors to a fund being used to defeat California's Proposition 37, which represents an effort to require labeling of genetically engineered food sold within the state. The list came from a news release supplied by the California Right to Know Campaign, which is attempting to pass Proposition 37. The data came from the website of the California Secretary of State Debra Bowen (<http://tinyurl.com/CalSecState>). The issue will come to a vote on November 6, 2012.

As of August 15, Monsanto Co. had contributed \$4.2 million dollars to defeat Proposition 37, or three times more than the next-biggest contributors, E.I. Dupont de Nemours (\$1.3 million) and Dow Agrosciences (\$1.2 million). Altogether, contributions totaled \$25 million. The journal *Nature* reported on August 20 that DuPont's contribution had risen to \$4.0 million (<http://tinyurl.com/Nature-Prop37>).

According to Examiner.com, groups funding efforts to promote passage of Proposition 37 had raised \$2.4 million as of mid-August (<http://tinyurl.com/Funds-for-Prop37>).

## Genetically engineered olive trees destroyed

Eddo Rugini, a plant scientist at the University of Tuscia (Viterbo, Italy), began a research program in 1982 to find varieties of olives, cherries, and kiwifruit that are resistant to pathogens, mainly fungi and bacteria, and that produce shorter trees—which would make cultivation easier in some areas of Italy. In 1998, he received authorization to plant these experimental trees, which were genetically modified with different combinations of the tobacco osmotin gene to resist fungal diseases or with *Root loci (rol) ABC* genes from the soil bacterium *Agrobacterium rhizogenes* to produce shorter trees (*Nature Biotechnology* 30:736, 2012).

In 2009, the researchers attempted to renew the authorization, but were turned down because their plants were not fully contained in a greenhouse. However, there was no official directive to destroy the trees, so the plants continued to grow.

The Genetic Rights Foundation (GRF), an Italian environmental nongovernmental organization, began to push for the destruction of the trees in mid-May 2012. The National Association of Italian Biotechnologists in response launched a petition calling for the research to be given a stay of

execution. Part of their statement said, “The experiments have not yet provided significant results as trees require a long time to grow. For this reason, we request that the plants not be destroyed. . . . Halting research on these trees means a waste of decades of publicly funded research” (<http://tinyurl.com/Olive-Trees-axed>).

The government ordered the university to destroy the trees in mid-June, and the order was carried out.

## Indian parliamentary panel recommends end of GM field trials

The Indian Committee on Agriculture presented the 37th Report of the Committee on Cultivation of Genetically Modified Food Crops—Prospects and Effects to Lok Sabha (the lower house of Parliament) on August 9, 2012. The Committee made a number of recommendations, including:

- A thorough probe into the process by which Bt brinjal (eggplant modified to contain Bt toxin) received government approval
- Reexamination of data that indicated adverse effects on lambs fed with Bt cottonseed
- An in-depth and comprehensive examination of the functioning, composition, powers, and mandate of the Genetic Engineering Appraisal Committee
- An in-depth probe of the Department of Agriculture and Cooperation for its failure to live up to its responsibilities regarding the introduction of transgenic agricultural crops in India
- Investigation into how large quantities of cottonseed oil extracted from Bt cotton have gotten into the human food chain
- Consideration of the possible negative impact of transgenic crops on export markets
- Labeling of all genetically modified products, including food, feed, and food products
- Strict containment of all research and development on transgenics in agricultural crops
- Discontinuation of all field trials

For further information, see <http://tinyurl.com/India-GMdebate> and <http://tinyurl.com/LokSabha-GMFoodCrops>. ■

The Clorox Co. (Oakland, California, USA) introduced concentrated Clorox® regular bleach in August 2012. The change reduces the typical 96-ounce (roughly 3-liter) bleach bottle by a third to 64 ounces.

■■■

The US Environmental Protection Agency (EPA) has released a final report on a case study of engineered nanosilver used in disinfectant sprays. EPA said it used a comprehensive environmental assessment framework that structures available information pertaining to the product life cycle; environmental transport and fate; exposure-dose in receptors, including humans, ecological populations, and the environment; and potential impacts in these receptors. The document does not draw conclusions about potential risks. But, "it is intended to be used as part of a process to identify what is known and unknown about nanosilver in a selected application," the agency noted. The report (PDF) is available at <http://tinyurl.com/EPA-nanosilver>.

■■■

Solazyme, the renewable oils company based in South San Francisco, California, USA, is set to debut lauric oils produced by algae in 2013. According to a presentation from the company at the ICIS World Surfactants Conference in April 2012—as reported by the ICIS Green Chemical blog—Solazyme "can modify the fatty acid composition and saturation of its genetically engineered algae oils to produce lauric acid contents greater than 80%."

■■■

The European Chemicals Agency (ECHA) has published new guidance on the use of certain chemicals introduced under European Regulation No 1907/2006 concerning the Registration, Evaluation, Authorization, and restriction of Chemical substances (REACH). The law entered into force on June 1, 2007. ECHA said that the guidance aims to interpret provisions in Annex XVII of REACH by answering specific questions about the restrictions placed on a number of substances. The agency has emphasized that the document is intended as guidance only and that "only the European Court of Justice can give an authoritative interpretation" of European Union legislative provisions. For more information, visit <http://tinyurl.com/ECHA-guidance>. ■

# Surfactants, Detergents, & Personal Care News



## Multinationals losing market share in China

The headline says it all: "In China, some imports get a local run for the money."

The article below the headline ran in *The Wall Street Journal* (WSJ; see <http://tinyurl.com/WSJ-China>) in August 2012. It details the changing consumer environment in China that has Chinese brands cutting into the market share of the big multinational consumer products companies such as Unilever and Procter & Gamble (P&G).

The WSJ report notes that Guangzhou Liby Enterprise Group Co. and Nice Group are "dominating markets for home-care products like detergents and soaps with a combined market share of 27.6%, compared with P&G's 7.6% and Unilever's 6.6%, according to Euromonitor."

Despite sales that have slowed as China's economy has faltered, the outlook for beauty and personal care products remains strong, Euromonitor told the newspaper, with an expected growth rate of 12% this year. The

home care market is expected to grow by 11% to \$12.2 billion.

The idea that the multinational consumer products giants are losing market share is not true, according to Paul Fox, a P&G spokesperson. Fox told WSJ that "it only works if you pick a subcategory and then make comparisons, but you could equally pick another subcategory and demonstrate that multinationals are gaining share."

## Normal paraffin market expected to recover

The normal paraffin (NP) market is expected to recover substantially by the end of 2012, largely due to the two-phase capacity additions by Shell, according to Beroe Inc. Beroe, a market research firm, is based in Chennai, India.

Normal paraffins in the C10–C13 range are an important precursor for the manufacture of linear alkylbenzene (LAB),

CONTINUED ON NEXT PAGE



## The international top 30

*HAPPI* magazine, which covers the Household and Personal Product Industry, recently published this list of the top 30 international companies in the global household and personal products industry. For a list of the top 50 US companies on the global scene, visit <http://www.happi.com/articles/2012/07/the-top-50>.

1. Unilever: United Kingdom—\$32.9 billion
2. L'Oréal: France—\$28.2 billion
3. Reckitt Benckiser: United Kingdom—\$12.1 billion
4. Henkel: Germany—\$10.7 billion
5. Kao: Japan—\$10.0 billion
6. Shiseido: Japan—\$8.6 billion
7. Beiersdorf: Germany—\$6.5 billion
8. LVMH: France—\$4.2 billion
9. Lion: Japan—\$3.6 billion
10. Natura: Brazil—\$3.3 billion
11. GlaxoSmithKline: United Kingdom—\$2.7 billion
12. AmorePacific: South Korea—\$2.3 billion
13. Bolton: The Netherlands—\$2.3 billion
14. LG: South Korea—\$2.1 billion
15. Kosé: Japan—\$2.0 billion
16. Oriflame: Sweden—\$2.0 billion
17. Pola: Japan—\$2.0 billion
18. Chanel: France—\$1.8 billion
19. Puig: Spain—\$1.7 billion
20. Yves Rocher: France—\$1.7 billion
21. Clarins: France—\$1.6 billion
22. Belcorp: Peru—\$1.5 billion
23. Pierre Fabre: France—\$1.3 billion
24. PZ Cussons: United Kingdom—\$1.3 billion
25. McBride: United Kingdom—\$1.2 billion
26. L'Occitane: Luxembourg—\$1.1 billion
27. Fancil: Japan—\$874 million
28. Lornamead: United Kingdom—\$793 million
29. Sunstar: Switzerland—\$784 million
30. Mandom: Japan—\$757 million

which in turn is used to produce linear alkylbenzene sulfonate (LAS), the workhorse surfactant of the detergent industry.

Before 2009, the NP market remained oversupplied, Beroe said, with average operating rates estimated at approximately 75–80%. Most LAB producers are backward-integrated to NP, and the industry is characterized by a high degree of captive consumption with demand from LAB accounting for a major share of NP production.

However, approximately one-fourth of the demand for NP is satisfied through the merchant market. “In such a market environment, withdrawal of approximately 16% of capacity by ExxonMobil and Sasol tightened the market, with nonintegrated LAB players suffering the most,” Aditya Vikram Kapoor, a Beroe researcher, told *inform*. “This led to a spike in NP prices, fueled by growing demand by the detergent sector for LAS.”

In March 2011, the Pearl GTL (Gas-to-Liquid) plant at Ras Laffan, Qatar, came online. Pearl GTL is a joint venture between Qatar Petroleum and Shell, with the latter taking control of marketing activities

related to NP. Pearl's first commercial shipment of NP was in March 2012, Kapoor said. “Once the Pearl GTL plant reaches full capacity, it is expected to alleviate the tight market by adding 260,000 metric tons of capacity by the end of 2012,” he added.

“GTL-based NP has already been well received in the industry, with Unilever certifying its usage in detergents to be akin to [NP produced via the] kerosene-based route,” Kapoor said. Gulf Petroproducts Co. EC of Saudi Arabia, Tamilnadu Petroproducts Ltd. of India, and P.T. Unggul Indah Cahaya Tbk. of Indonesia have already signed contracts, according to Beroe, and are procuring GTL-based NP from Shell.

While these NP capacity expansions will provide restoration of balance in the next two to three years, further NP capacity will be required in the long term to meet strong growth in the use of LAB by the detergent sector. NP capacity additions are expected to take place in South East Asia and Latin America, Kapoor said, as manufacturers look to shift production to potent demand markets that promise higher growth in the future.

## Self-repairing coating

Researchers at Eindhoven University of Technology (EUT; Netherlands) have developed a coating with a surface that repairs itself after damage. This new coating has numerous potential applications—for example, mobile phones that will remain clean from fingerprints, cars that never need to be washed, and aircraft that need less-frequent repainting.

Functional coatings with highly water-resistant or antibacterial properties have at their surface nano-sized molecular groups that provide specific properties. But these molecular groups are easily and irreversibly damaged by minor contact (such as by scratching), quickly causing their properties to be lost. This has been a big limitation to the possible applications of these types of coatings.

Researchers led by Catarina Esteves of the Department of Chemical Engineering and Chemistry at EUT have now found a solution to this problem. They have done this by developing surfaces with special “stalks” carrying the functional chemical groups at their ends, and mixing these stalks throughout the coating. If the outer surface layer is removed by scratching, the stalks in the underlying layer reorient to the new surface, thereby restoring the function.

This development can be of great importance for many applications, the researchers say. For example, it will be possible to make a self-cleaning car, with a highly water-resistant coating that keeps this self-cleaning property for long periods. The superficial damages to the coating will be self-repaired and the water droplets will simply roll off the car, taking dirt with them. An occasional rain shower is all that will be needed to keep the car clean.

In the same way, products such as mobile phones, solar panels, or even aircraft will remain clean for a longer time. For aircraft, a cleaner surface means less air resistance, which in turn reduces fuel consumption. Other possible applications are contact lenses that self-repair their scratches, and coatings that resist the growth of algae that would be of use in the marine coatings market. A limitation of the new technology is that it only works with superficial scratches that do not completely penetrate the coating, the researchers noted.

Esteves and her team now intend to further develop this coating together with other universities and industrial partners. She expects the first coatings to be ready for production within six to eight years, at prices comparable to those of today's coatings. The results appeared in *Advanced Materials* (doi: 10.1002/adma.201200807, 2012). ■

# People News/ Inside AOCS

## Yeh moves to ZeaChem

In July 2012, AOCS member Bryan Yeh left his position as assistant vice president for biofuels with SAIC (Oakland, California, USA) to move to ZeaChem, Inc. (Menlo Park, California), where he is executive vice president for technology. ZeaChem has developed a cellulose-based biorefinery platform capable of producing advanced



Yeh

ethanol, fuels, and chemicals.

Yeh is a member of the Editorial Advisory Committee for *inform*.

## Olivares heads Bioscience Division at LANL

The National Alliance for Advanced Biofuels and Bioproducts, a consortium of leading scientists and engineers from universities, private industry, and national laboratories led by the Donald Danforth Plant Science Center (St. Louis, Missouri, USA), announced on July 24, 2012 that Executive

Director José Olivares had been selected to serve as division leader for the Bioscience Division, a central hub for biotechnology development at Los Alamos National Laboratory (LANL). Olivares, who has considerable experience in algal biofuels research, will lead a team of 180 researchers and staff within disciplinary groups that include genome science, biosecurity and public health, bioenergy and environmental science, and advanced measurement science. Olivares will hold this position until the end of the currently funded consortium term, which is April 2013.

## Darzins joins GTI

Al Darzins has joined Gas Technology Institute (GTI; Des Plaines, Illinois, USA), a research, development, and training organization addressing energy and environmental challenges, as a research and development director. He will focus GTI's biological-based research efforts in alternative energy, renewable fuels development, and microbiologically influenced corrosion.

Darzins comes to GTI from the Biochemical Sciences & Engineering area at DuPont, where he led the Protein Engineering and Enabling Technologies groups within Biofuels. Before that, he was with the National Renewable Energy Laboratory

(NREL), where he managed the Applied Sciences group in the National Bioenergy Center. He provided leadership to this multidisciplinary research team responsible for developing and integrating chemical and biological technologies for the conversion of biomass to transportation fuels. Darzins' major research interests at NREL included the development of microalgae as a potential feedstock for a variety of biofuel applications.

## Werpy VP for R&D at ADM

Archer Daniels Midland Co. (ADM; Decatur, Illinois, USA) appointed Todd Werpy as vice president (VP) for research and development (R&D) in mid-August. He was previously the company's VP for chemicals and advanced biofuels. He will have responsibility for all corporate R&D functions, and will oversee ADM's efforts to expand its product portfolio and strengthen research partnerships with government agencies, academic institutions, and corporations.

Werpy joined ADM in 2007 following a 15-year tenure at the US Department of Energy's Pacific Northwest National Laboratory, where he was responsible for research and business development for new chemicals and chemical intermediates from renewable feedstocks. He was part of the team that developed the catalytic technology ADM uses to convert glycerin and sorbitol to propylene glycol.

Werpy holds more than 20 patents in the area of catalysis and chemical conversion of biomass to chemicals. ■

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## IN MEMORIAM

### LINCOLN DOUGLAS METCALFE

Lincoln D. Metcalfe died at the age of 91 on April 17, 2012, in LaGrange, Illinois, USA. His wife of 63 years, Evelyn, survives, as well as his daughter Lynne Kornecki, two grandsons, and two great-grandsons.



Metcalfe

He attended Wilson Junior College and received a bachelor's degree in meteorology in 1943 from the University of California-Los Angeles (USA). Metcalfe served three years of active duty overseas with the US Air Force in meteorology and air traffic control, then went on to the University of Chicago (Illinois, USA) to earn a B.S. in chemistry in 1947.

Metcalfe's first position after graduation from the University of Chicago was with Armour and Co. in the Chicago area, where he started as an analytical chemist. He continued with the company in its various transformations until his retirement. The company recognized his work with its Armour Creative Science Award.

He was widely known for his work on the analytical chemistry of fatty acids and their derivatives, particularly in gas chromatography, and published a number of articles in the *Journal of the American Oil Chemists' Society*. He also developed a number of patents.

Metcalfe joined AOCS in 1959, and was an active participant in the North Central Section. He served on the Governing Board as a Member-at-Large in 1980–1981.

The AOCS North Central Section recognized Metcalfe's work in 1990 by naming him to receive the A.E. Bailey Award. In his acceptance speech, he told a revealing tale on himself: In the 1950s Metcalfe paid \$200 for what was only the fifth gas chromatograph built by the company that evolved into Hewlett Packard—but then accidentally burned out the instrument. He said, "I was afraid to tell management that I destroyed their expensive gas chromatograph—the only one in the company. I then secretly enlisted the lab machine and instrument shops to help me design and build a gas chromatograph that would separate fatty acid methyl esters and all the fatty acid derivatives. The instrument we built was successful beyond our wildest hopes" (*INFORM* 1:404–406, 1990). The machines, he added, produced "excellent chromatographs with the methyl ester, nitriles, amine, quaternary ammonium compounds, ketones, aldehydes, amino acids, and just about anything else we could find to inject into the instruments."

Metcalfe also received the AOCS Award of Merit in 1997 and was named an AOCS Fellow in 1999.

### CECILIA R. GILMORE

AOCS has received word of the death of Cecilia R. Gilmore on April 20, 2009, at the age of 90. She was an emeritus member of AOCS, having joined in 1951.

Gilmore received a B.A. from Rosary College, River Forest, Illinois, USA, in 1940 (the school later became Dominican University). She began with Durkee Famous Foods in its Chicago location as a chemist in 1941, and stayed with the company through its various

transformations until her retirement from its Strongsville, Ohio, location in the early 1990s.

She is survived by 21 nieces and nephews, and many great- and great-great nieces and nephews.

### POUL MØLLER

The daughter of Poul Møller notified AOCS in July 2012 of his death. Møller received his M.Sc. in chemical engineering in 1949, and when he joined AOCS in 1962 he was head of the research department for AB Karlshamn Oljefabriker in Karlshamn, Sweden. He later moved to Denmark, where he pursued his interests in recovering trace nutrients from oils and in using supercritical conditions as a means to hydrogenate fats and oils.

### JOHN BERNARD BRAUNWARTH

John Braunwarth, of Janesville, Wisconsin, USA, died on November 11, 2011, at the age of 82. Three sisters survive, and three other siblings preceded him in death. He joined AOCS in 1976.

He received his B.S. from Milton College (Wisconsin) in 1951. His first position was with Pure Oil (later purchased by Union Oil) as a research chemist. He then was research manager for Varney Chemical in Janesville, a division of Northern Petrochemical Co. He joined Armstrong Chemical Co. in Janesville in 1972 as a research chemist and retired from the company as chief executive officer. Armstrong (which is now part of ABITEC Corp.) manufactured ammonium chloride quaternary compounds, fabric softeners, and industrial chemicals.

## AOCS Board Petition to Nominate

For each annual election of AOCS Governing Board officers, the membership may nominate up to four additional member-at-large candidates by petition. Petitioned candidates receiving at least 50 AOCS member signatures will be added to the ballot approved by the Governing Board. Preference will be given to the first four petitioned candidates meeting the eligibility requirements as outlined here. Petitioned nominations must be received at the AOCS Headquarters no later than **October 30, 2012.**

Petition forms can be obtained by visiting <http://aocs.files.cms-plus.com/AboutUsPDFs/2012Petition.pdf>.

Please mail completed petitions with at least 50 AOCS signatures to: AOCS Nominations and Elections Committee, P.O. Box 17190, Urbana, IL 61803-7190 USA or fax to: Amy Lydic, +1 217-693-4852.

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# Extracts & Distillates

## The effect of coffee consumption on serum lipids: a meta-analysis of randomized controlled trials

Cai, L., *et al.*, *Eur. J. Clin. Nutr.* 66:872–877, 2012.

Numbers of epidemiological studies assessing coffee consumption and serum lipids have yielded inconsistent results. We aimed to evaluate the effects of coffee intake on serum lipids by searching several English and Chinese electronic databases up to September 2011 for randomized controlled trials of coffee on serum lipids. Weighted mean effect size was calculated for net changes in serum lipids by using random-effect models or fixed-effect models. Subgroup and meta-regression analyses were conducted to explore possible explanations for heterogeneity among trials. Twelve studies conducted in Western countries with a total of 1017 subjects were identified. Meta-analyses showed, on average, drinking coffee for 45 days was associated with an increase of 8.1 mg/dl (95% confidence interval (CI): 4.5, 11.6;  $P < 0.001$ ) for total cholesterol (TC), 5.4 mg/dl (95% CI: 1.4, 9.5;  $P = 0.009$ ) for low-density lipoprotein cholesterol (LDL-C), and 12.6 mg/dl (95% CI: 3.5, 12.6;  $P = 0.007$ ) for triglyceride (TG). The increase in TC was greater in trials using unfiltered coffee and caffeinated coffee as the treatment group. Those who had hyperlipidemia were more sensitive to the cholesterol-raising effect of coffee. Meta-regression analysis revealed a positive dose-response relation between coffee intake and TC, LDL-C and TG. The intake of coffee, especially unfiltered coffee, contributed significantly to the increase in TC, LDL-C, and TG, and the changes were related to the level of intake. Studies of coffee intake on serum lipids in Asian populations should be performed.

## Oxidation of cod liver oil during gastrointestinal *in vitro* digestion

Larsson, K., *et al.*, *J. Agric. Food Chem.* 60: 7556–7564, 2012.

Oxidation of cod liver oil rich in long-chain n-3 polyunsaturated fatty acids (LC n-3 PUFA) was investigated during a gastrointestinal (GI) *in vitro* digestion. The digestion

stimulated TBA-reactive substances (TBARS) formation in both the gastric and intestinal steps, whereas levels of lipid hydroperoxides remained nearly constant. The presence of digestive compounds was decisive for the TBARS development because TBARS did not change when the cod liver oil was subjected only to the temperature and pH gradient of the GI model. Preformed oxidation products in the cod liver oil resulted in further elevated TBARS levels during the digestion. Addition of hemoglobin (11.5  $\mu$ M) to emulsified cod liver oil dramatically increased TBARS and lipid hydroperoxide levels during GI digestion, whereas 1 mg  $\alpha$ -tocopherol/g oil did not show any protection against oxidation. Specific concern thus needs to be taken in the design of foods containing LC n-3 PUFA to preserve these lipids and avoid harmful oxidation, both before and after consumption.

## Administration of omega-3 fatty acids and Raloxifene to women at high risk of breast cancer: interim feasibility and biomarkers analysis from a clinical trial

Signori, C., *et al.*, *Eur. J. Clin. Nutr.* 66:878–884, 2012.

The antiestrogen, Raloxifene (Ral) is an effective breast cancer chemopreventive agent. Omega-3 fatty acids (n-3FA) may inhibit mammary carcinogenesis. On the basis of their mechanisms of action, we test the hypothesis that a combination of n-3FA and Ral may be superior in reducing select biomarkers of breast cancer risk in women. Postmenopausal women at increased risk for breast cancer (breast density 25%) were randomized to: (1) no intervention; (2) Ral 60 mg; (3) Ral 30 mg; (4) n-3FA (Lovaza) 4 g, and (5) Lovaza 4 g+Ral 30 mg for two years. Reduction in breast density is the primary end point of the study. We report preliminary data on feasibility, compliance, and changes in secondary end points related to IGF-I signaling, estrogen metabolism, oxidative stress, and inflammation in the first group of 46 women who completed one year of the study. All interventions were well tolerated with excellent compliance ( $96 \pm 1\%$  overall) by pill count and also supported by the expected rise in both serum n-3FA and n-3FA/omega-6 fatty acids (n-6FA) ratio in women randomized to groups 4 and 5 ( $P < 0.05$ ). Lovaza decreased serum triglycerides and increased high-density lipoprotein (HDL) cholesterol compared with control ( $P$

$< 0.05$  for both). Ral reduced serum IGF-1 in a dose-dependent manner ( $P < 0.05$ ) while Lovaza did not. Lovaza had no effect on IGF-1 or IGFBP-3. None of the other biomarkers were affected by our treatment. The combination of Lovaza and Ral is a feasible strategy that may be recommended in future breast cancer chemoprevention trials.

## Isolation and identification of a potent radical scavenger (canolol) from roasted high erucic mustard seed oil from Nepal and its formation during roasting

Shrestha, K., *et al.*, *J. Agric. Food Chem* 60: 7506–7512, 2012.

Roasting of high erucic mustard (HEM) seed has been reported to give a typical flavor and increase the oxidative stability of the extracted oil. A potent radical scavenging compound was successfully isolated from roasted HEM seed oil in a single-step chromatographic separation using an amino solid-phase extraction column. Nuclear magnetic resonance and mass spectrometry spectra revealed the compound as 2,6-dimethoxy-4-vinylphenol (generally known as canolol), and its identity was fully confirmed by chemical synthesis. The formation of canolol during roasting was compared among HEM varieties (*Brassica juncea*, *B. juncea* var. *oriental*, *B. nigra*, and *Sinapis alba*) together with a low erucic rapeseed variety. HEM varieties were shown to produce less than one-third of canolol compared to rapeseed at similar roasting conditions. This observation was linked to a lower free sinapic acid content together with a lower loss of sinapic acid derivatives in the HEM varieties compared to rapeseed. Around 50% of the canolol formed in the roasted seed was shown to be extracted in the oil. Roasting of HEM seed before oil extraction was found to be a beneficial step to obtain canolol-enriched oil, which could improve the oxidative stability.

## Commercialization of high oleic canola oils

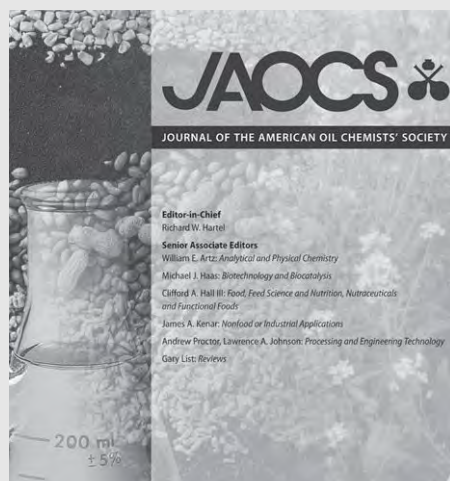
DeBonte, L., *et al.*, *Lipid Technol.* 24:175–177, 2012.

High oleic canola oils were developed through plant breeding in the 1980s as a trans-fat solution. More than 25 years after its initial launch, high oleic canola oils include a series of products with fatty acid profiles tailored for both foodservice and food processing. High

CONTINUED ON PAGE 588



## AOCS Journals

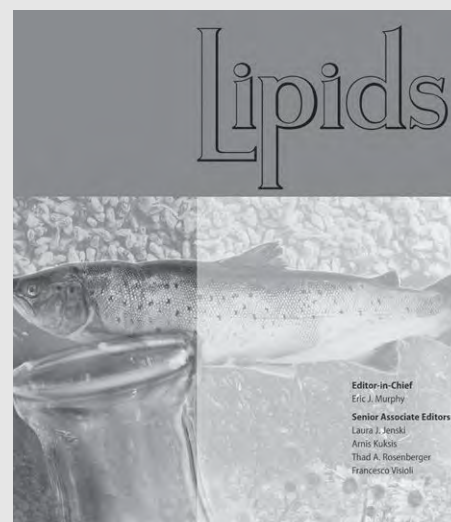


### Journal of the American Oil Chemists' Society (September)

- Studies on mango (*Mangifera indica*, L.) kernel fat of some Kenyan varieties in Meru, Muchiri, D.R., S.M. Mahungu, and S.N. Gitunja
- Ultrahigh pressure liquid chromatographic determination of tocopherols in B100 biodiesel, Pauls, R.E.
- Characterization of the solvent properties of glycerol using inverse gas chromatography and solubility parameters, Vincent, J.D., K. Srinivas, and J.W. King
- Identification of the unsaturated heptadecyl fatty acids in the seed oils of *Thespesia populnea* and *Gossypium hirsutum*, Dowd, M.K.
- Proximate analysis of *Adenanthera pavonina* L. seed oil, a source of lignoceric acid grown in Pakistan, Sultana, R., and T. Gulzar
- Biodiesel production from soybean oil catalyzed by  $\text{Li}_2\text{CO}_3$ , Wang, J.X., K.T. Chen, S.T. Huang, K.T. Chen, and C.C. Chen
- Enzyme-catalyzed synthesis of monoacylglycerols citrate: kinetics and thermodynamics, Huang, J., Y. Liu, Q. Jin, X. Wu, X. Wang, and Z. Song
- Concentration of docosahexaenoic acid (DHA) by selective alcoholysis catalyzed by lipases, Martín Valverde, L.M., P.A. González Moreno, A. Rodríguez Quevedo, E. Hita Peña, M.J. Jiménez Callejón, L. Esteban Cerdán, E. Molina Grima, and A. Robles Medina

- Esterification of free fatty acids in *Zanthoxylum bungeanum* seed oil for biodiesel production by stannic chloride, Zhang, J., J. Liu, and H. Ma
- Concentration of stearidonic acid in free fatty acids form from modified soybean oil by selective esterification with dodecanol, Vázquez, L., L. Kleiner, and C. C. Akoh
- Microbial conversion of arachidonic acid to arachidonyl alcohol by a new *Acinetobacter* species, Nagao, T., Y. Watanabe, S. Tanaka, M. Shizuma, and Y. Shimada
- The effect of temperature and moisture content of stored rapeseed on the phytosterol degradation rate, Gawrysiak-Witulska, M., M. Rudzińska, J. Wawrzyniak, and A. Siger
- Degradation of phytosterols during near-ambient drying of rapeseeds in a thick immobile layer, Gawrysiak-Witulska, M., and M. Rudzińska
- Specialty fats enriched with behenic and medium chain fatty acids from palm stearin by lipase acidolysis, Mounika, C., S. Yella, and S. Reddy
- Chemical and sensory stability of fried-salted soybeans prepared in different vegetable oils, Jáuregui, M.P., C. Riveros, V. Nepote, N.R. Grosso, and M.F. Gayol
- Use of an adsorption process for purification of pollock-oil-based biodiesel comprises methyl esters, Mis Solval, K., and S. Sathivel
- Synthesis and characterization of polyols derived from corn oil by epoxidation and ozonolysis, de Souza, V.H.R., S.A. Silva, L.P. Ramos, and S.F. Zawadzki
- Bonding quality of chemically-modified soybean protein concentrate-based adhesives in particleboards from rice husks, Cianname, E.M., J.F. Martucci, P.M. Stefani, and R.A. Ruseckaite
- Evaluation of Siberian apricot (*Prunus sibirica* L.) germplasm variability for biodiesel properties, Wang, L.
- Investigation of conjugated soybean oil as drying oils and CLA sources, Chintareddy, V.R., R.E. Oshel, K.M. Doll, A. Yu, W. Wu, G. Zhang, and J.G. Verkade
- Chemical composition and oxidative stability of roasted and cold pressed pumpkin

- seed oils, Neđeral, S., D. Škevin, K. Kraljić, M. Obranović, S. Papeša, and A. Bataljaku
- (In)correct use of statistics, Dijkstra, A.J.



### Lipids (September)

- Correcting the effects of  $-20^{\circ}\text{C}$  storage and aliquot size on erythrocyte fatty acid content in the Women's Health Initiative, Pottala, J.V., M.A. Espeland, J. Polreis, J. Robinson, and W.S. Harris
- Myocardial infarction changes sphingolipid metabolism in the uninfarcted ventricular wall of the rat, Knapp, M., M. Žendzian-Piotrowska, K. Kurek, and A. Błachnio-Zabielska
- *trans*-10, *cis*-12 Conjugated linoleic acid enhances endurance capacity by increasing fatty acid oxidation and reducing glycogen utilization in mice, Kim, J.H., J. Kim, and Y. Park
- Free fatty acid derivative HUHS2002 potentiates  $\alpha 7$  ACh receptor responses through indirect activation of CaMKII, Kanno, T., T. Shimizu, A. Tanaka, T. Nishimoto, and T. Nishizaki
- Macrophages alter the differentiation-dependent decreases in fibronectin and collagen I/III protein levels in human preadipocytes, Gagnon, A., M.N. Yarmo, A. Landry, and A. Sorisky
- Dynamics of the lipid content and biomass of *Calanus finmarchicus* (copepodite V) in a Norwegian fjord, Bergvik, M., Ø. Leiknes, D. Altin, K.R. Dahl, and Y. Olsen

- Sesamin modulates gene expression without corresponding effects on fatty acids in Atlantic salmon (*Salmo salar* L.), Schiller Vestergren, A., L. Wagner, J. Pickova, G. Rosenlund, A. Kamal-Eldin, and S. Trattne
- Isolation of a  $\Delta 5$  desaturase gene from *Euglena gracilis* and functional dissection of its HPGG and HDASH motifs, Pollak, D.W., M.W. Bostick, H. Yoon, J. Wang, D.H. Hollerbach, H. He, H.G. Damude, H. Zhang, N.S. Yadav, S.-P. Hong, P. Sharpe, Z. Xue, and Q. Zhu
- Serum autotaxin is not a useful biomarker for ovarian cancer, Nakamura, K., K. Igarashi, R. Ohkawa, H. Yokota, A. Masuda, S. Nakagawa, T. Yano, H. Ikeda, J. Aoki, and Y. Yatomi
- Kinetic study of sodium cocoyl sarcosinate synthesis and factors affecting the reaction on bench and pilot scales, Hassanzadeh, M., M. Kambarani, L. Tayebi, and F. Yazdian
- Highly efficient adsorption of anionic dyes from aqueous solutions using sawdust modified by cationic surfactant of cetyltrimethylammonium bromide, Ansari, R., B. Seyghali, A. Mohammad-khah, and M.A. Zanjanchi
- Physicochemical studies of 4-substituted *N*-(2-mercaptophenyl)-salicylideneimines: corrosion inhibition of mild steel in an acid medium, Kesavan, D., M.M. Tamizh, M. Gopiraman, N. Sulochana, and R. Karvembu
- Corrosion inhibition by some cationic surfactants in oil fields, Tawfik, S.M., A. Sayed, and I. Aiad
- Synthesis, characterization, and conductivity of quaternary nitrogen surfactants modified by the addition of a hydroxymethyl substructure on the head group, Jordan, D., E. Tan, and D. Hegh
- Properties of the quaternary ammonium salts with novel counterions, Yan, H., Q. Li, T. Geng, and Y. Jiang
- Properties of sodium methyl ester alpha-sulfo alkylate/trimethylammonium bromide mixtures, Wong, S.P., W.H. Lim, S.-F. Cheng, and C.H. Chuah
- Effects of inorganic salts and polymers on the foam performance of 1-tetradecyl-3-methylimidazolium bromide aqueous solution, Zhang, Q., X. Wei, J. Liu, D. Sun, X. Zhang, C. Zhang, and J. Liu
- Preparation and surface activities of modified double-tailed anionic surfactants, Lin, L.-H., H.-J. Liu, H.-C. Chu, M.-Y. Dong, M.-C. Hwang, C.-F. Wang, and K.-M. Chen
- Synthesis, characterization, and surface activity of alkyl *m*-xylene sulfonate isomers, Luan, H., W. Wu, J. Cheng, T. Yu, G. Qu, Y. Xu, G. Li, X. Gao, A. Li, D. Yuan, W. Tong, Z. Li, K. Liu, G. Liu, and W. Ding
- Critical micelle concentration of poly(oxy-1,2-ethanediyl),  $\alpha$ -nonyl phenol- $\omega$ -hydroxy ethers ( $C_9H_{19}C_6H_4E_{i=6,10,5,12,17,5}$ ) by surface equations of state, Viades-Trejo, J., D.M. Abascal-González, and J. Gracia-Fadrique
- Composition of multicomponent surfactant systems at the water-air interface, Szymczyk, K.
- Synthesis and surface activities of amido-betaine surfactants with ultra-long unsaturated hydrophobic chains, Feng, D., Y. Zhang, Q. Chen, J. Wang, B. Li, and Y. Feng



### *Journal of Surfactants and Detergents* (September)

- Modeling of precipitation phase boundaries in mixed surfactant systems using an improved counterion binding model, Maneedaeng, A., A.E. Flood, K.J. Haller, and B.P. Grady
- Synthesis and properties of a series of CO<sub>2</sub> switchable surfactants with imidazoline group, Qiao, W., Z. Zheng, and Q. Shi
- Effect of temperature, salts and ureas on the association behavior of an amphiphilic phenothiazine drug promethazine hydrochloride, Kabir-ud-Din, M.A. Rub, and A.Z. Naqvi

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oleic canola oil has taken most of the market for trait-enhanced oils while other specialty oils have either seen significant market share reduction or failure. High oleic canola oils with 23%–27% linoleic acid are ideal for fried foods, while those with less than 20% are most suitable in the manufacture of shelf-stable foods. In addition to improved nutrition, the next generation of high oleic canola oils will significantly reduce packaging costs.

### Measurement of single soybean seed attributes by near-infrared technologies. A comparative study

Agelet, L.E., *et al.*, *J. Agric. Food Chem.* 60:8314–8322, 2012.

Four near-infrared spectrophotometers, and their associated spectral collection methods, were tested and compared for measuring three soybean single-seed attributes: weight (g), protein (%), and oil (%). Using partial least-squares (PLS) and four preprocessing methods, the attribute that was significantly most easily predicted was seed weight (RPD > 3 on average) and protein the least. The performance of all instruments differed from each other. Performances for oil and protein predictions were correlated with the instrument sampling system, with the best predictions using spectra taken from more than one seed angle. This was facilitated by the seed spinning or tumbling during spectral collection as opposed to static sampling methods. From the preprocessing methods utilized, no single one gave the best overall performances but weight measurements were often more successful with raw spectra, whereas protein and oil predictions were often enhanced by SNV and SNV + detrending.

### Phytosterols and sterol esters in diverse *Cucurbita*, *Cucumis* and *Citrullus* seed oils

Oelschlägel, S., *et al.*, *Lipid Technol.* 24:181–184, 2012.

$\Delta 7$ -Phytosterols present in pumpkin seed oil are significant for the prevention of prostate disorders. Herbal medicines are increasingly offered as dried kernels or concentrated ethanolic extracts of *Cucurbita pepo* seeds. Until now, the pumpkin seeds of *C. pepo* have almost exclusively been used for this purpose. Only a few data concerning the sterol content of other Cucurbitaceae seeds are available. Therefore, we isolated, identified, and quantified the free and esterified phytosterols of 12 *Cucurbita*, 3 *Cucumis*, and 3 *Citrullus* seed oils. The total sterol content of these seeds ranged from 297 mg per 100 g oil in *Cucurbita maxima* ‘Turk’s Turban’ to 814 mg per 100 g oil in *Citrullus lanatus* ‘Sugar Baby Watermelon,’ equivalent to 64 to 193 mg per 100 g seeds respectively. These were mainly  $\Delta 7$ -sterols (~82%) with the sterol esters accounting for ~32% of the total sterol content.

### Lipidomics: when apocrypha becomes canonical

Brown, H.A., *Curr. Opin. Chem. Biol.* 16:221–226, 2012.

Lipidomics is a branch of the field of metabolomics. Although only about a decade since its inception, lipidomics has already had a major influence on the way in which questions about lipid metabolism and signaling are posed. The field is intertwined in the culture and rich history of mass spectrometry. Early

work emphasized analytical issues such as limits of detection and numbers of molecular species quantitated in single injections. Increased sophistication in applications of lipidomic analysis and emerging technologies, such as imaging mass spectrometry, are facilitating the study of lipid metabolism and signaling species in cellular functions and human diseases. In the coming years we anticipate a richer understanding of how specific lipid species mediate complex biological processes and interconnections between cellular pathways that were thought to be disparate.

### Degradation of edible oil during food processing by ultrasound: electron paramagnetic resonance, physicochemical, and sensory appreciation

Pingret, D., *et al.*, *J. Agric. Food Chem.* 60:7761–7768, 2012.

During ultrasound processing of lipid-containing food, some off-flavors can be detected, which can incite depreciation by consumers. The impacts of ultrasound treatment on sunflower oil using two different ultrasound horns (titanium and pyrex) were evaluated. An electron paramagnetic resonance study was performed to identify and quantify the formed radicals, along with the assessment of classical physicochemical parameters such as peroxide value, acid value, anisidine value, conjugated dienes, polar compounds, water content, polymer quantification, fatty acid composition, and volatiles profile. The study shows an increase of formed radicals in sonicated oils, as well as the modification of physicochemical parameters evidencing an oxidation of treated oils. ■

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# Patents

## Published Patents

### Water-based primer composition and coating method of plastic shaped articles using the composition

Ishikura, M., *et al.*, Kansai Paint Co., Ltd., US8173744, May 8, 2012

This invention provides a water-based primer composition characterized by comprising an aqueous dispersion formed by dispersing unsaturated carboxylic acid- or acid anhydride-modified polyolefin (i) having a melting point of 50–120°C and a weight-average molecular weight within a range of 30,000–200,000, and unsaturated carboxylic acid- or acid anhydride-modified polyolefin (ii) having a melting point of 30–100°C and a weight-average molecular weight within a range of 30,000–200,000, in an aqueous medium, the melting point of the modified polyolefin (i) being higher than that of the modified polyolefin (ii) by at least 10°C.

### Method of making fatty acid ester derivatives

Erhan, S.Z., *et al.*, United States of America/Secretary of Agriculture; Penn State Research Foundation, US8173825, May 8, 2012

Fatty acid ester derivatives and a process for their production from unsaturated fatty acids are disclosed. The process comprises: (i) reacting an unsaturated fatty acid or an ester thereof having one or more sites of unsaturation, with an epoxidation reagent to form a fatty acid epoxide wherein at least one of the sites of unsaturation of the fatty acid or fatty acid ester is converted to an oxirane ring; and (ii) reacting the fatty acid epoxide produced in (i) with a carboxylic acid to form a hydroxy fatty acid ester derivative wherein the oxirane ring is opened and converted to a hydroxy ester comprising a hydroxyl group at one carbon of the opened oxirane ring and an ester of the carboxylic acid at the other carbon of the opened oxirane ring.

### Non-aqueous pigment ink

Endo, T., Riso Kagaku Corp., US8177899, May 15, 2012

A non-aqueous pigment ink comprising a pigment, a pigment dispersant, and a non-aqueous solvent, wherein the non-aqueous solvent comprises an alcohol solvent, a fatty acid ester solvent, and a hydrocarbon solvent, the alcohol solvent comprises a saturated branched alcohol containing 14 to 18 carbon atoms and having one branch, and an amount of the saturated branched alcohol is within a range from 3 to 40% by mass relative to a total mass of the ink.

### Surfactant materials and coatings for weighting agents for use in oil based drilling fluids

Ballard, D.A., M-I LLC, US8178475, May 15, 2012

A wellbore fluid that includes an oleaginous continuous phase; a nonoleaginous phase; and a polymeric additive formed by mixing at least one lipophilic monomer and at least one crosslinking agent, wherein the at least one lipophilic monomer is at least one of an

epoxide-functionalized derivative of at least one selected from soybean oil, linseed oil, rapeseed oil, cashew nut shell oil; perilla oil, tung oil, oiticia oil, safflower oil, poppy oil, hemp oil, cottonseed oil, sunflower oil, high-oleic triglycerides, triglycerides of euphorbia plants, peanut oil, olive oil, olive kernel oil, almond oil, kapok oil, hazelnut oil, apricot kernel oil, beechnut oil, lupine oil, maize oil, sesame oil, grapeseed oil, lallemantia oil, castor oil, herring oil, sardine oil, menhaden oil, whale oil, tall oil, and synthetic aliphatic or aromatic ethers, and the at least one crosslinking agent includes at least one selected from amines, alcohols, phenols, thiols, carbanions, carboxylates, and mixtures thereof is disclosed.

### Method and apparatus for drying carboxylic acid

Parker, K.R., and P.E. Gibson, Grupo Petrotex, S.A. DE C.V., US8173835, May 8, 2012

Disclosed is a method and apparatus for drying a wet cake in a carboxylic acid production process. The method comprises employing a contact dryer for drying solid particles of carboxylic acid, where the solid particles can have a residence time of less than about 7 minutes in the dryer and an exit temperature of less than about 250°C upon exiting the dryer.

### Tread for tire

Lopitiaux, G., and D. Vasseur, Michelin Recherche et Technique S.A., US8178605, May 15, 2012

Tire tread comprising a rubber composition, the said composition comprising at least one diene elastomer, one reinforcing filler and one plasticizing system, characterized in that the said plasticizing system comprises, in combination: a plasticizing hydrocarbon resin, the glass transition temperature of which is greater than 0°C; and a carboxylic acid diester corresponding to the formula (I):  $R-OOC-(CH_2)_n-COO-R$  in which  $n$  is included within a range from 1 to 15 and the R radicals, which are identical or different, represent a hydrocarbon radical. The invention also relates to the use of such a tread in the manufacture or the retreading of tires exhibiting a substantially improved compromise in properties with regard to the wear resistance and the wet grip.

### Fatty acid acetylated salicylates and their uses

Milne, J.C., *et al.*, Catabasis Pharmaceuticals, Inc., US8173831, May 8, 2012

The invention relates to fatty acid acetylated salicylate derivatives; compositions comprising an effective amount of a fatty acid acetylated salicylate derivative; and methods for treating or preventing an inflammatory disorder comprising the administration of an effective amount of a fatty acid acetylated salicylate derivative.

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



## FATTY ACID RECOVERY (CONTINUED FROM PAGE 566)

be transferred to this material, leading to an increase in mono- or diglycerides.

## FFA reduction in other oils

Fish oil is in great demand because of the need for long-chain omega-3 fatty acids as nutritional supplements, but the levels in these oils are normally around 20% of the total fatty acid composition. Extraction by esterification and short-path distillation followed by concentration by an enzymatic condensation step using immobilized *C. antarctica* B lipase is the normal route to high omega-3 content products. But fish oils typically contain approximately 5% FFA, which are removed by neutralization, and this process results in an overall yield loss of approximately 10%. If the FFA component can be masked by recombination with mono- and diglycerides, then this loss can largely be avoided. By using the same process as we have applied to crude palm oil, FFA levels can be considerably reduced (Fig. 4, page 566).

Two other oils, coming from the ethanol produced from corn and waste collected from the food industry, have also been studied. These oils are typified by high levels of FFA as well as other forms of acidity that can interfere with the condensation reaction. So for optimal enzyme performance, a small amount of alkali is added to generate about 50 ppm of soap in the oil. This increases the pH and improves the enzyme reaction. The results are summarized in Table 1.

## Improving sustainability

The use of enzymatic processing to improve sustainability of oils and fats processing has been reported upon before in *inform* (Cowan, D., K.M. Oxenbøll, and H.C. Holm, *inform* 19:210–212, 2008). The main benefits in terms of reduction of environmental indicators such as greenhouse gases (CO<sub>2</sub>), acidification, and smog formation come from reduced energy requirements, lower losses, and lowered chemicals consumption. Therefore, if we can reduce losses overall in the production of vegetable oils, we can achieve a reduction in the environmental impact of these production processes.

Our department, which is involved with determining the sustainability of enzymatic processes, has developed some simple tools for the study of how a single environmental impact indicator (CO<sub>2</sub> production) is affected by changing process conditions. We have modeled the production of margarine using palm stearine and palm kernel oil as raw materials and applying either enzymatic (EIE) or chemical (CIE) interesterification as the process for achieving the correct melting profile of the hard stock. If the raw materials used in this process are derived from a palm oil process in which the overall yield has been increased by converting the fatty acids back to triglycerides, then a new calculation can be carried out. In Table 2, the potential benefits of switching from CIE to EIE are compared to those when fatty acid conversion is used.

The calculation of CO<sub>2</sub> reduction is based on a reduction in FFA of 3% and an equal increase in yield. Overall, there will be a small reduction in fatty acid distillate, and this is not included in the calculation. However, despite this small reduction in fatty acid distillate, there will be a significant improvement in yield and reduction in greenhouse gas production associated with the reconversion of fatty acids back to triglycerides. The same calculations can also be applied to other cases indicated in the article for which the benefits will be even larger as they are directly related to the starting level of FFA in the oil. New enzyme

**TABLE 1.** Free fatty acid (FFA) reduction in waste corn and food oils

Source	Starting FFA (%)	FFA after reaction (%)	Time (h)
Corn oil from ethanol plant	12	2	8
Waste oil from the food industry	17	4	6

**TABLE 2.** Reduction in environmental impact<sup>a</sup>

	CO <sub>2</sub> saving (kg)/MT hard stock
CIE to EIE	22.7
FFA recovery	80.6
Process CO <sub>2</sub>	-5.8
Overall balance	74.8

<sup>a</sup>CIE, chemical interesterification; EIE, enzymatic interesterification; MT, metric tons. For other abbreviation, see Table 1.

formulations better suited to the operating conditions are under development and full-scale testing is planned for the near future.

*David Cowan is a senior customer solutions scientist at Novozymes, where he works in the field of enzyme applications within vegetable oil processing and manages the lipase applications laboratory. He can be contacted at dc@novozymes.com.*

## One thing leads to another

**1880:** The manufacture of lard substitutes from cottonseed oil is developed. **1902:** The fermentation process for hydrolyzing fats using the enzyme lipase is discovered. **1910:** The Sabatier-Norrmann-Kaiser process for hydrogenating vegetable oils is introduced. **1911:** Procter & Gamble launches the cottonseed oil-based shortening, Crisco. **1922:** Englishman Thomas McCulloch Fairborn patents a miniature golf course surface made from crushed cottonseed hulls mixed with oil, dyed green, and rolled on top of a sand foundation. The “artificial green” eliminates the need for real grass care, which allows mini golf to grow.



# Book Review

## ***Palm Oil: Production, Processing, Characterization and Uses***

**Oi-Ming Lai, Chin-Ping Tan, and Casimir C. Akoh (eds.)**

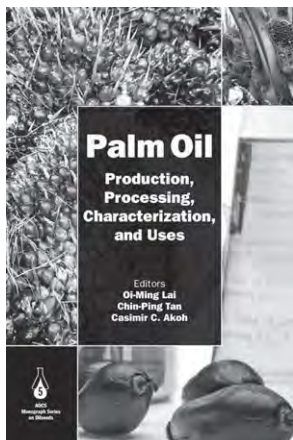
**AOCS Press, 2012, 838 pages**

**\$270 (nonmembers) or \$195 (members)**

**ISBN 978-098189369-3**

**Albert J. Dijkstra**

AOCS Press introduced *Palm Oil: Production, Processing, Characterization and Uses*—the fifth book in the AOCS Monograph Series on Oilseeds—just in time for the 2012 AOCS Annual Meeting & Expo. According to the invitation sent to prospective authors in December 2010, the book set out to serve as a “concise and well-documented source of information on major conventional and latest technological advances and/or emerging technologies in the production and processing, characterization,



and strategies used in the utilization of palm oil and its components from upstream to downstream in Africa, South America, and South East Asia. It also examines the current state of knowledge on genomics, tissue culture, and genetic engineering of oil palm. Current hot topics that are garnering attention and becoming globally relevant such as palm oil as an alternative to *trans* fats in food, waste, and environmental management and issues relating to sustainable development of [the] palm oil industry are highlighted and addressed. Special chapters on palm biomass in various wood-based products, bioenergy, and biofuels are also included.” This is a very ambitious aim, indeed, but I think that the book as published succeeded in achieving this aim.

This book will be of great help to the growing number of people whose work involves palm oil or palm kernel oil. Even if they are not directly interested in all the aspects of palm oil covered by this book, it will put their interest in perspective by providing them with background information on historical, botanical, economic, and technological aspects of palm oil production and processing; the chemical, physicochemical, and nutritional properties of palm oil; and its use

in food and the oleochemical industry. It also covers waste management, by-product utilization, and sustainability aspects.

The chapters are invariably up to date and provide a wealth of literature references; the combined references cover 145 pages! Most journal references are readily accessible, but some local journals may be more difficult for those living elsewhere, since many have not been listed in scholar.google.com. When governmental reports are referred to, a website is often mentioned, including the date it was accessed. Relatively few patents have been included in the references.

The editors are to be complimented for producing this book, to which more than 60 different authors living in a dozen or so different countries contributed 25 chapters. Their task must have been daunting, especially since the provisional list of contributors shows many names of authors who did not actually contribute. Accordingly, new authors had to be invited at short notice, which put the editors under even greater pressure. Consequently, I have the impression that there was just too little time to carry out an exhaustive editing and copy-editing job and as a result, the book shows occasional overlaps.

Are these overlaps a serious drawback? Far from it. The field covered by the book is so wide that most readers will probably study only part of the book in depth and, therefore, will hardly notice that part of what they study has also been explained elsewhere. Accordingly, this duplication may well come in handy since what has been duplicated usually forms an integral part of the chapter concerned. In fact, the editors should be congratulated that the three chapters describing palm and palm kernel oil production in Malaysia, Brazil, and Nigeria look at the same subject in totally different ways and in fact complement each other.

Having edited several books myself after my career in industrial R&D, I can envisage the huge task faced by the editors of inviting authors, trying to keep to deadlines, suggesting changes, trying to agree on them, and the like. Moreover, for many of the 60+ authors, English is not their mother tongue. Consequently, some chapters are definitely easier to read than others. Sometimes I had to read a sentence several times to grasp what the author meant, and I did not always succeed.

Personally, I would not be surprised if the first print run of this book were to be exhausted fairly soon. I therefore advocate that the editors start preparing subsequent print runs by some careful and judicious editing while there is still time and, it is to be hoped, also edit the index. This editing will make the book far more accessible, easier to read; it will eliminate inconsistencies and increase its value to the reader even further. In my opinion, the book is certainly worth this additional effort.

*Although officially retired, Albert J. Dijkstra remains active as an author, editor, inventor, and scientific consultant with more than 30 years experience in food oil chemistry and processing. He can be contacted at [albert@dijkstra-tucker.be](mailto:albert@dijkstra-tucker.be).*

## Did you know?

The first processing of soybeans in the United States occurred in Seattle, Washington, in 1911. Sesame, linseed, and castor oils were pressed in Egypt as early as 259 BCE. Europe first started importing palm oil in 1850.

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## **Aflatoxin Corn Meal Test Kit First Place (tie)**

Janet Smith  
Fieldale Farms Corp.  
Baldwin, Georgia 30511  
USA

## **Aflatoxin Corn Meal Test Kit First Place (tie)**

Wayne Adcock  
State Chemical Laboratory  
Auburn, Alabama 36832  
USA

## **Aflatoxin Corn Meal Test Kit Honorable Mention**

Tuyen Mai  
Intertek Agricultural Services  
St. Rose, Louisiana 70087  
USA

## **Aflatoxin Corn Meal Test Kit Honorable Mention**

Tami Brown  
A&L Plains Agricultural Labs Inc.  
Lubbock, Texas 79408  
USA

## **Aflatoxin in Almonds First Place**

Alex Kostin  
Neogen Corp.  
Lansing, Michigan 48912  
USA

## **Aflatoxin in Corn Meal First Place**

Paul Thionville, Shani Jolly,  
Andre Thionville, Nancy  
Trosclair, and Boyce Butler  
Thionville Laboratories, Inc.

New Orleans, Louisiana 70123  
USA

## **Aflatoxin in Corn Meal Honorable Mention**

Dr. Jim Balthrop Jr.  
Office of the Texas State Chemist  
College Station, Texas 77843  
USA

## **Aflatoxin in Cottonseed First Place**

John Wetmore  
Wetmore Enviro Lab Ltd.  
Casa Grande, Arizona 85122  
USA

## **Aflatoxin in Milk First Place**

Martha J. Chinakwe  
Alabama Department of  
Agriculture & Industries  
Montgomery, AL 36107-1123  
USA

## **Aflatoxin in Peanut Butter First Place**

Simone Staiger  
Eurofins WEJ Contaminants  
GmbH  
Hamburg 21079  
Germany

## **Aflatoxin in Peanut Butter Honorable Mention**

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Edenton Analytical Team  
JLA USA  
Edenton, North Carolina  
27932  
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IEH Laboratories & Consulting  
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Lost Hills, California 93249  
USA

## **Aflatoxin in Peanut Paste Honorable Mention**

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Specialty Lab  
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USA

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Buenos Aires C11130 DNN  
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## **Aflatoxin in Peanut Paste Honorable Mention**

Angela Turville  
NSW Forensic & Analytical  
Laboratory  
Lidcombe NSW 2141  
Australia

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Alex Kostin  
Neogen Corp.  
Lansing, Michigan 48912  
USA

## **Aflatoxin Peanut Paste Test Kit First Place**

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Algood Food Co.  
Louisville, Kentucky 40258-1896  
USA

## **Aflatoxin Peanut Paste Test Kit Honorable Mention**

Brantley Freeman  
Algood Food Co.  
Louisville, Kentucky 40258-1896  
USA

## **Aflatoxin Peanut Paste Test Kit Honorable Mention**

Andrea Pando  
Brownfield Analytical Team  
JLA USA  
Brownfield, Texas 79316  
USA

## **Cholesterol First Place (tie)**

Ann Lindgren, Paulette  
Manemann  
Hormel Foods LLC  
Austin, Minnesota 55912  
USA

## **Cholesterol First Place (tie)**

Sonia Bouchard  
CFIA Food Lab  
Longueuil PQ J4K 1C7  
Canada

## **Cholesterol Honorable Mention**

Covance Labs  
Madison, WI 53704  
USA

CONTINUED ON NEXT PAGE



**Cholesterol****Honorable Mention**

Analytical Team  
Nutreco Canada Inc.  
St Hyacinthe, Québec J2R 1S5  
Canada

**Cottonseed****First Place**

Donald Britton  
Mid-Continent Laboratories  
Memphis, Tennessee 38101  
USA

**Cottonseed Oil****First Place**

Paul Thionville, Shani Jolly,  
Andre Thionville, Nancy  
Trosclair, and Boyce Butler  
Thionville Laboratories, Inc.  
New Orleans, Louisiana  
70123  
USA

**Edible Fat****First Place**

Beth Miller  
Ag Processing Inc.  
St. Joseph, Missouri 64504  
USA

**Edible Fat****Honorable Mention**

Eddie L. Baldwin, Helen  
Cianciolo, Howard Payne  
Stratas Foods RDI Center  
Bartlett, Tennessee 38133  
USA

**Edible Fat****Honorable Mention**

James Houghton  
AAK USA  
Louisville, Kentucky 40208  
USA

**Edible Fat****Honorable Mention**

Deborah McRoberts  
AAK USA  
Louisville, Kentucky 40208  
USA

**Edible Fat****Honorable Mention**

Linda S. McLaren  
Loders Croklaan

Channahon, Illinois 60410  
USA

**Feed Microscopy****First Place**

Elizabeth Krzykwa  
Canadian Food Inspection  
Agency  
Ottawa, Ontario K1A 0C6  
Canada

**Feed Microscopy****Second Place**

Dr. Jim Balthrop Jr.  
Office of the Texas State Chemist  
College Station, Texas 77843  
USA

**Feed Microscopy****Honorable Mention**

Piotr Czajkowski  
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Poland

**Feed Microscopy****Third Place**

Yuan-te Fu  
NCDA & CS Food & Drug  
Protection  
Raleigh, North Carolina 27607  
USA

**Fish Meal****First Place**

Carl W. Schulze  
New Jersey Feed Laboratory Inc.  
Trenton, New Jersey 08638  
USA

**Fish Meal****Honorable Mention**

Paul Thionville, Shani Jolly,  
Andre Thionville, Nancy  
Trosclair, and Boyce Butler  
Thionville Laboratories, Inc.  
New Orleans, Louisiana 70123  
USA

**Fumonisin in Corn****First Place**

Jerome J. King, Daniel Jabs  
Midwest Laboratories Inc.  
Omaha, Nebraska 68144  
USA

**Fumonisin in Corn Test Kit****First Place**

Gary Coleman  
University of Kentucky  
Department of Regulatory  
Services  
Lexington, Kentucky  
40546-0275  
USA

**Gas Chromatography****First Place**

Analytical Team Bunge  
Bradley, Illinois 60915  
USA

**Gas Chromatography****Honorable Mention**

Linda S. McLaren  
Loders Croklaan  
Channahon, Illinois 60410  
USA

**Gas Chromatography****Honorable Mention**

Paul Thionville, Shani Jolly,  
Andre. Thionville, Nancy  
Trosclair, and Boyce Butler  
Thionville Laboratories, Inc.  
New Orleans, Louisiana 70123  
USA

**Gas Chromatography****Honorable Mention**

Kim Jennings-Wilson  
Stratas Foods  
Quincy, Illinois 62306  
USA

**Gas Chromatography****Honorable Mention**

Randall Hoffman  
ADM Valdosta  
Valdosta, Georgia 31601  
USA

**Gas Chromatography****Honorable Mention**

Hazimah Abu Hassan and  
Hajar Musa  
Malaysian Palm Oil Board  
Bandar Baru Bangi, Selangor  
43650  
Malaysia

**Gas Chromatography****Honorable Mention**

Jocelyn Alfieri  
Silliker Canada Co.  
Markham, Ontario L3R 5V5  
Canada

**GOED/AOCS****Nutraceutical Oils****First Place**

Mark Arsenault  
Ocean Nutrition Canada  
Mulgrave, Nova Scotia B0E 2G0  
Canada

**GOED/AOCS****Nutraceutical Oils****Honorable Mention**

Pete Cartwright  
New Jersey Feed Laboratory Inc.  
Trenton, New Jersey 08638  
USA

**Marine Oil****First Place**

Analytical Services POS  
Bio-Sciences  
Saskatoon, Saskatchewan  
S7N 2R4  
Canada

**Marine Oil****Honorable Mention**

Otelia Robertson  
Omega Protein Inc.  
Reedville, Virginia 22539  
USA

**Marine Oil Fatty Acid Profile****First Place**

Pete Cartwright  
New Jersey Feed Laboratory Inc.  
Trenton, New Jersey 08638  
USA

**Marine Oil Fatty Acid Profile****Honorable Mention**

Cecilia Palomino  
SGS Del Peru S A  
Callao 1 27-0125  
Peru

**Marine Oil Fatty Acid Profile****Honorable Mention**

Analytical Services POS  
Bio-Sciences  
Saskatoon Saskatchewan  
S7N 2R4  
Canada

**Mixed Seeds Canola****First Place**

Sovena Oilseeds Laboratory  
Sovena Oilseeds  
Almada 2801-801  
Portugal

**Mixed Seeds Safflower****First Place**

Sovena Oilseeds Laboratory  
Sovena Oilseeds  
Almada 2801-801  
Portugal

**Mixed Seeds Sunflower****First Place**

Sovena Oilseeds Laboratory  
Sovena Oilseeds  
Almada 2801-801  
Portugal

**NIOP Fats and Oils****First Place**

Renato M. Ramos  
Admiral Testing Services  
Luling, Louisiana 70070  
USA

**NIOP Fats and Oils****Honorable Mention**

Melanie Greer  
Dallas Group of America  
Jeffersonville, Indiana 47130  
USA

**Nutritional Labeling****First Place**

Sonia Bouchard  
CFIA Food Lab  
Longueuil PQ J4K 1C7  
Canada

**Nutritional Labeling****Honorable Mention**

Analytical Team  
Nutreco Canada Inc.  
St Hyacinthe, Québec J2R 1S5  
Canada

**Oilseed Meal****First Place**

Lynn Hawkins, Michael Hawkins  
Barrow-Agee  
Memphis, Tennessee  
38116-3507  
USA

**Oilseed Meal****Honorable Mention**

Mike White, Brian Eskridge  
ATC Scientific  
North Little Rock, Arkansas  
72114  
USA

**Oilseed Meal****Honorable Mention**

Trevor Meredith  
Solbar Hatzor  
Ashdod 77121  
Israel

**Oilseed Meal****Honorable Mention**

Frank Tenent, Edgar Tenent  
K-Testing Lab Inc.  
Memphis, Tennessee 38116  
USA

**Oilseed Meal****Honorable Mention**

Frank Fuentes  
Southern Cotton Oil Co.  
Lubbock, Texas 79404  
USA

**Oilseed Meal****100% Crude Fiber****First Place (tie)**

Frank Fuentes  
Southern Cotton Oil Co.  
Lubbock, Texas 79404  
USA

**Oilseed Meal****100% Crude Fiber****First Place (tie)**

Ardin Backous, Anders Thomsen,  
Keith Persons, and Kent Karsjens  
Eurofins Scientific  
Des Moines, Iowa 50321-3157  
USA

**Oilseed Meal****100% Crude Fiber****Honorable Mention**

Frank Tenent, Edgar Tenent  
K-Testing Lab Inc.  
Memphis, Tennessee 38116  
USA

**Oilseed Meal****100% Crude Fiber****Honorable Mention**

Brad Newton Beavers,  
Jennie Stewart  
Carolina Analytical Services  
Bear Creek, North Carolina  
27207  
USA

**Oilseed Meal****100% Crude Fiber****Honorable Mention**

Lynn Hawkins, Michael Hawkins  
Barrow-Agee  
Memphis, Tennessee  
38116-3507  
USA

**Oilseed Meal 100% Moisture****First Place**

John Reuther, Eric de Ronde  
Eurofins Central Analytical Labs  
Metairie, Louisiana 70001  
USA

**Oilseed Meal 100% Moisture****Honorable Mention**

Sandra Holloway  
Intertek  
Memphis, Tennessee 38113  
USA

**Oilseed Meal 100% Moisture****Honorable Mention**

Trevor Meredith  
Solbar Hatzor  
Ashdod 77121  
Israel

**Oilseed Meal 100% Moisture****Honorable Mention**

Renato M. Ramos  
Admiral Testing Services  
Luling, Louisiana 70070  
USA

**Oilseed Meal****100% Moisture****Honorable Mention**

Gordon Whitbeck, John Dillard  
A&A Laboratories Inc.  
Springdale, Arizona 72764  
USA

**Oilseed Meal****100% Moisture****Honorable Mention**

Frank Fuentes  
Southern Cotton Oil Co.

Lubbock, Texas 79404  
USA

**Oilseed Meal 100% Moisture****Honorable Mention**

Chin Chaothaworn  
SGS Thailand Ltd.  
Bangkok 10120  
Thailand

**Oilseed Meal 100% Moisture****Honorable Mention**

Renato M. Ramos  
Admiral Testing Services  
Luling, Louisiana 70070  
USA

**Oilseed Meal 100% Nitrogen****Ba 4d-90****First Place**

Trevor Meredith  
Solbar Hatzor  
Ashdod 77121  
Israel

**Oilseed Meal 100% Nitrogen****Ba 4d-90****Honorable Mention**

Garlon J. Beckwith  
Mid-Continent Laboratories  
Greenwood, Mississippi 38930  
USA

**Oilseed Meal 100% Nitrogen****Ba 4e-93****First Place**

Mike White, Brian Eskridge  
ATC Scientific  
North Little Rock, Arkansas  
72114  
USA

**Oilseed Meal 100% Nitrogen****Ba 4e-93****Honorable Mention**

Frank Tenent, Edgar Tenent  
K-Testing Lab Inc.  
Memphis, Tennessee 38116  
USA

**Oilseed Meal 100% Nitrogen****Ba 4e-93****Honorable Mention**

Lynn Hawkins, Michael  
Hawkins  
Barrow-Agee

CONTINUED ON NEXT PAGE



Memphis, Tennessee  
38116-3507  
USA

**Oilseed Meal 100% Oil  
First Place**

Melinda Graham  
Hartsville Oil Mill  
Darlington, South Carolina  
29540-1027  
USA

**Oilseed Meal 100% Oil  
Honorable Mention**

Ardin Backous, Anders  
Thomsen, Keith Persons,  
and Kent Karsjens  
Eurofins Scientific  
Des Moines, Iowa  
50321-3157  
USA

**Oilseed Meal 100% Oil  
Honorable Mention**

Mike White, Brian Eskridge  
ATC Scientific  
North Little Rock, Arkansas  
72114  
USA

**Oilseed Meal 100% Oil  
Honorable Mention**

Carmina Jinga  
SGS Canada  
Vancouver, British Columbia  
V6P 6T7  
Canada

**Olive Oil Part A  
First Place**

Giorgio Cardone  
Chemiservice s.a.s.  
Monopoli, Bari 70043  
Italy

**Olive Oil Part A  
Honorable Mention**

Ioanis Falagas  
Soya Hellas S.A.  
Athens 10673  
Greece

**Olive Oil Part B  
First Place**

Claudia Guillaume  
Modern Olives Laboratory  
Services

Lara, Victoria 3212  
Australia

**Olive Oil Part C  
First Place**

Giorgio Cardone  
Chemiservice s.a.s.  
Monopoli, Bari 70043  
Italy

**Palm Oil  
First Place**

Ricardo Arevalo  
Numar Company  
San Jose  
Costa Rica

**Palm Oil  
Honorable Mention**

James Houghton  
AAK USA  
Louisville, Kentucky 40208  
USA

**Palm Oil  
Honorable Mention**

Deborah McRoberts  
AAK USA  
Louisville, Kentucky 40208  
USA

**Peanut Seed  
First Place**

Laboratory Staff  
USDA AMS S&T Science  
Specialty Lab  
Blakely, Georgia 39823-2785  
USA

**Peanut Seed  
Honorable Mention**

Edenton Analytical Team  
Tina Harrell  
JLA USA  
Edenton, North Carolina  
27932  
USA

**Phosphorus in Oil  
First Place**

Analytical Services POS  
Bio-Sciences  
Saskatoon, Saskatchewan  
S7N 2R4  
Canada

**Solid Fat Content by NMR  
First Place**

Bill Zubrinic  
Bunge Canada  
Hamilton, Ontario L8N 3K7  
Canada

**Solid Fat Content by NMR  
Honorable Mention**

Randy Kruger  
Bunge North America  
Council Bluffs, Iowa 51503  
USA

**Solid Fat Content by NMR  
Honorable Mention**

Ricardo Arevalo  
Numar Company  
San Jose  
Costa Rica

**Soybean Oil  
First Place**

Carl Whitney  
ConAgra Grocery Products  
Memphis, Tennessee 38104  
USA

**Soybean Oil  
Honorable Mention**

Carmina Jinga  
SGS Canada  
Vancouver, British Columbia  
V6P 6T7  
Canada

**Soybeans  
First Place**

Tuyen Mai  
Intertek Agri Services  
St. Rose, Louisiana 70087  
USA

**Soybeans  
Honorable Mention**

Ardin Backous, Anders Thomsen  
Keith Persons, and Kent Karsjens  
Eurofins Scientific  
Des Moines, Iowa  
50321-3157  
USA

**Specialty Oils  
First Place**

John Reuther, Chris Lirette  
Keith Barnhill  
Eurofins Central Analytical Labs

Metairie, Louisiana 70001  
USA

**Tallow & Grease  
First Place (tie)**

Carmina Jinga  
SGS Canada  
Vancouver, British Columbia  
V6P 6T7  
Canada

**Tallow & Grease  
First Place (tie)**

Adalberto Coronado  
National Beef Packing  
Company  
Liberal, Kansas 67901  
USA

**Tallow & Grease  
Honorable Mention**

Montréal Analytical Team  
Sanimax San Inc.  
Montréal PQ H1C 1G2  
Canada

**Tallow & Grease  
Honorable Mention**

Jose Garcia  
National Beef Packing  
Company  
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USA

**Trace Metals  
First Place**

John Reuther, Marvin Boyd  
Eurofins Central Analytical  
Labs  
Metairie, Louisiana 70001  
USA

**Trace Metals  
Honorable Mention**

Mark Ochs  
CHS  
Mankato, Minnesota 56001  
USA

**trans by GC  
First Place**

Paul Thionville, Shani Jolly,  
Andre Thionville, Nancy  
Trosclair, and Boyce Butler  
Thionville Laboratories Inc.  
New Orleans, Louisiana 70123  
USA

**trans by GC****Honorable Mention**

Devika Srinivasan,  
Yasuhiro Fujii, and Matthew Paul  
Weston Food Laboratories  
Enfield NSW 2136  
Australia

**trans by GC****Honorable Mention**

Marilyn Lowe  
Stratas Foods  
Decatur, Illinois 62526  
USA

**trans by GC****Honorable Mention**

Linda S. McLaren  
Loders Croklaan  
Channahon, Illinois 60410  
USA

**trans by GC****Honorable Mention**

Marcos Rogerio Da Silva  
ADM do Brasil Ltda  
Rondonopolis MT 78745-800  
Brazil

**trans by GC****Honorable Mention**

Saiko Sera  
The Nisshin Oillio Group Ltd.  
Yokosuka 239-0832  
Japan

**trans by GC****Honorable Mention**

Wakako Tsuzuki  
National Food Research Institute  
Tsukuba, Ibaraki 305-8642  
Japan

**trans by IR****First Place**

QA/QC Laboratory  
ADM Mankato Refinery  
Mankato, Minnesota 56001  
USA

**Unground Soybean Meal**  
**First Place**

Ardin Backous, Anders Thomsen,  
Keith Persons, and Kent Karsjens  
Eurofins Scientific  
Des Moines, Iowa 50321-3157  
USA

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Renato M. Ramos

**ATC Scientific**

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North Little Rock, AR 72114 USA  
+1 501-771-4255  
Mike White, Brian Eskridge

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Michael Hawkins, Lynn Hawkins

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Jennie Stewart, Brad Beavers

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Frank M. Hahn

**Intertek Agri Services**

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Tuyen Mai

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Edgar Tenent, Frank Tenent

**Minnesota Valley Testing Lab**

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Carmina Jinga

**SGS North America**

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Angela Carlson

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+1 504-733-9603  
Paul Thionville, Shani Jolly, Boyce Butler,  
Andre Thionville

**Unground Soybean Meal**  
**Honorable Mention**

Sharon Spicer  
Ag Processing Inc.  
St. Joseph, Missouri 64504  
USA

**Unground Soybean Meal**  
**Honorable Mention**

Sherry Muse

Ag Processing Inc.  
St. Joseph, Missouri  
64504  
USA

**Unground Soybean Meal**  
**Honorable Mention**

Mike White, Brian Eskridge  
ATC Scientific

North Little Rock, Arkansas  
72114  
USA

**Vegetable Oil Color Only**  
**First Place**

Melanie Greer  
Dallas Group of America  
Jeffersonville, Indiana 47130  
USA

# Active packaging materials to inhibit lipid oxidation: US regulatory framework

John Koontz

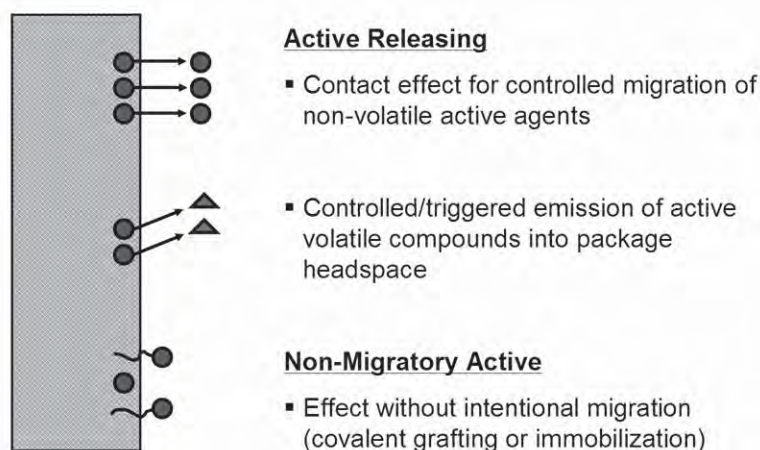
Food packaging has traditionally been defined as a passive barrier to delay the adverse effect of the environment on the product that is contained. More recently, packaging materials have been brought to market that interact with both the environment and the food, playing an active role in preservation. Such active packaging is primarily designed to prolong shelf life, improve safety, or enhance sensory properties of foods.

In polymer packaging, many strategies are currently used in the marketplace to inhibit oxidation in foods. These include modified atmosphere packaging, ultraviolet (UV) light absorbers, high oxygen barrier polymer films, and polymer-based oxygen scavenging systems. Meanwhile, researchers have laid the foundation for innovative active packaging technologies that may aid the food industry in inhibiting lipid oxidation, maintaining nutritional quality, and extending shelf life. In some cases, polymer packaging may serve as a reservoir or matrix from which active ingredients, such as antioxidants, are delivered in a controlled manner into a food product (Fig. 1). Antioxidants and metal-chelating agents have also been covalently grafted or immobilized onto polymer surfaces to provide benefits to the food. As the packaging industry contemplates moving toward direct incorporation or immobilization of active agents onto polymer packaging materials, it is important to review how current US Food and Drug Administration (FDA) regulations apply to these novel technologies.

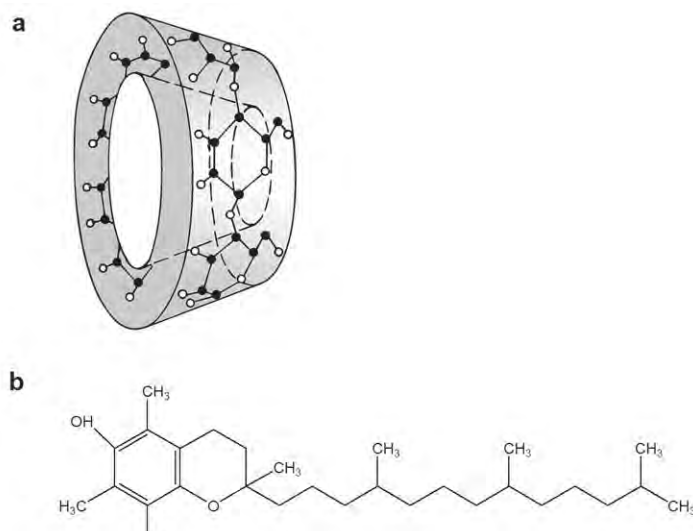
## Controlled-release antioxidant packaging

Antioxidants have traditionally been used as direct food additives in a single initial dose to protect food against generation of free radicals and lipid oxidation. Natural antioxidants have been shown to lose activity and become pro-oxidants at high concentrations; therefore, a need exists to develop active packaging that can gradually deliver antioxidants in a controlled manner. Cyclodextrins (CD) can be considered as empty capsules of molecular size that form inclusion complexes with guest molecules, resulting in an encapsulation process on the molecular scale (Fig. 2).

Researchers have (i) formed and characterized CD inclusion complexes with the natural antioxidants  $\alpha$ -tocopherol and quercetin; (ii) incorporated CD inclusion complexes of natural antioxidants into linear low-density polyethylene (LLDPE) films; and (iii) measured the release rates of inclusion complexes of natural antioxidants from LLDPE into a food simulant. Solid inclusion complexes of  $\alpha$ -tocopherol: $\beta$ -CD and quercetin: $\gamma$ -CD were formed by a co-precipitation method, and their antioxidant



**FIG. 1.** (Adapted from Dainelli, D., N. Gontard, D. Spyropoulos, E. Zonder-van-van den Beuken, and P. Tobback, *Active and intelligent food packaging: legal aspects and safety concerns*, Trends Food Sci. Technol 19(Suppl. 1): S103–S112, 2008).



**FIG. 2.** (Fig. 2a adapted from Loftsson, T., and M.E. Brewster, *Pharmaceutical applications of cyclodextrins 1. Drug solubilization and stabilization*, J. Pharm. Sci. 85:1017–1025, 1996).

contents were determined to be 18.1% and 13.0% (w/w), respectively. Free and CD-complexed antioxidant additives were compounded with a twin-screw mixer into two LLDPE resins followed by compression molding into films. Release of  $\alpha$ -tocopherol and quercetin from LLDPE films into coconut oil at 30°C was quantified by high-performance liquid chromatography. After four weeks, the total release of  $\alpha$ -tocopherol from



LLDPE was 70% from the free form and 8% from the CD-complexed form. The mechanism by which  $\alpha$ -tocopherol was released was modified due to its encapsulation inside the  $\beta$ -CD cavity within the LLDPE matrix as indicated by its diffusion coefficient decreasing by two orders of magnitude. Molecular encapsulation of natural antioxidants using CD may be used as a controlled-release mechanism within polymer food packaging to gradually deliver an effective antioxidant concentration to foods.

## What are food additives?

The US FDA is the primary federal agency responsible for ensuring the safety of food additives. The Office of Food Additive Safety within the Center for Food Safety and Applied Nutrition is responsible for ensuring the safety of all substances deliberately added to food and substances that may become a part of food as a result of migration from food packaging. A food additive is any substance added to food, directly or indirectly (food packaging or processing), unless the substance is generally recognized as safe (GRAS) for its intended use or is otherwise excluded. Food additives are generally classified by whether they impart an intended technical effect to the food and can be categorized into three groups: direct food additives, secondary direct food additives, and indirect food additives (Fig. 3).

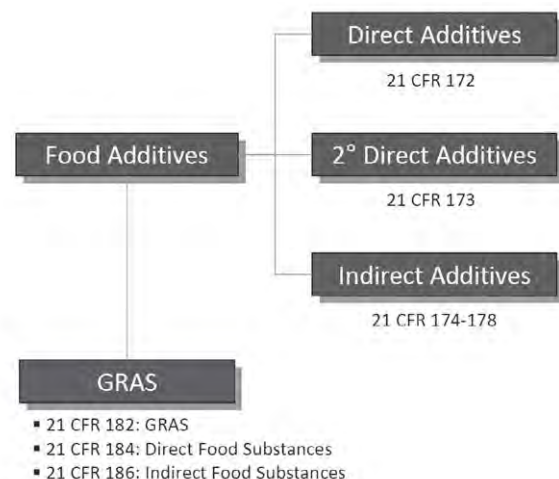
Direct additives are substances deliberately added to a food to accomplish an intended technical effect. The secondary direct additives are a sub-class of direct additives, primarily processing aids, intended to have a technical effect on food during the processing but not on the finished food. Indirect additives are substances that unintentionally become components of food, such as components of packaging materials, and have a technical effect in a food contact material, but not in the food itself.

GRAS compounds are generally recognized as safe for their intended use based on evaluations by qualified experts, and are exempt by law from the "food additive" definition. Both food additives and GRAS compounds may be used as food contact substances unless there is any intended effect on or in food. A food contact substance (FCS) is "any substance intended for use as a component of materials used in manufacturing, packing, packaging, transporting, or holding food if such use is not intended to have a technical effect in such food." Antioxidants are defined in the US Code of Federal Regulations as "substances used to preserve food by retarding deterioration, rancidity, or discoloration due to oxidation" and may be classified as either food additives or GRAS compounds.

## FDA regulatory programs

Premarket authorization is required for food additives as set forth in Section 409 of the Federal Food, Drug, & Cosmetic Act. FDA's current regulatory programs that the industry should follow to market new packaging materials include the food additive petition (FAP) program, which is the traditional petition process that is very time intensive. Direct food additives are authorized via the petition process, resulting in publication of regulations. The exceptions to the FAP program include: the threshold of regulation (TOR) program, the GRAS notification program, and the FCS notification (FCN) program. The TOR program is limited to only substances used in food packaging or processing equipment that do not show any intended effect on the food itself and have a dietary concentration below 0.5 ppb.

Substances whose use is GRAS by qualified experts are not required by law to receive FDA approval before marketing. A manufacturer may determine that use of a substance is GRAS without formally submitting to FDA as a so-called GRAS-self determination.



**FIG. 3.** US regulatory classification of food additives and GRAS (generally recognized as safe) substances in the Code of Federal Regulations.

## Global market for active packaging

The global market for active food and drink packaging was valued at \$11.7 billion in 2011 by Visiongain, a market research firm based in London, UK. Demand should rise by about 8% per year to reach \$17 billion by 2016 and to more than \$24 billion by 2021, the company forecast in a 2011 report.

The largest market for active packaging will be the United States, where Visiongain expects the market will reach \$3.6 billion by 2021. Japan—which produced the first active packaging materials in the mid-1970s—currently is the second-largest market, Visiongain said. There, the market value in 2021 likely will reach \$2.4 billion. Australia is forecast to be third, with a market worth of \$1.7 billion by 2021.

Market penetration in Europe has been slower, by comparison, owing to a regulatory framework that "could not keep up with technological innovations in the food packaging sector," according to Dario Dainelli and colleagues (*Trends in Food Science & Technology*, doi:10.1016/j.tifs.2008.09.011, 2008). Passage of regulations in 2009 should see the EU-27 become the second-largest market overall by 2021, according to the Visiongain report (see other sidebar).

Gas scavengers presently account for 36% of the total market and will see "solid growth," the report notes. The market for oxygen absorbers is also expected to increase, driven by the removal of *trans* fats from processed products and rising demand for packaged organic foods.

## EC regulates active packaging

The European Commission (EC) acted in May 2009 to regulate active and intelligent packaging via EC Regulation 450/2009. Under the Framework Regulation 1935/2004 governing food contact materials in general, the new regulation lays down specific rules for active and intelligent food contact materials.

According to the 2009 regulation, a list of authorized substances that can be used to manufacture active or intelligent packaging must be established after the European Food Safety Authority (EFSA) has performed a risk assessment and issued an opinion on each substance. The EFSA reviews focus on risks related to the dietary exposure to chemicals because of the migration of the active/intelligent substances, the migration of their degradation and/or reaction products, and their toxicological properties. Guidance on the regulation is available at <http://tinyurl.com/EC-Guidelines>. Further assistance for small- and medium-sized enterprises is available via a database of active and intelligent packaging innovations at <http://kb.activepackaging.eu>.

Despite the passage of the 2009 regulation, a packaging industry group is calling for further legislation. The Active & Intelligent Packaging Industry Association (AIPIA), based in Utrecht, was formed in February 2012 and includes Nestlé, Dow Performance Packaging, DSM, and Bayer among its 40-plus members. According to an article on AIPIA on the FoodManufacture.co.uk website, the group feels further legislation is needed “to clarify which active and intelligent packaging technologies and materials can be used, and how they can be applied.” The group did not respond to requests for an interview.

## information

- Koontz, J.L., J.E. Marcy, S.F. O’Keefe, and S.E. Duncan, Cyclodextrin inclusion complex formation and solid-state characterization of the natural antioxidants  $\alpha$ -tocopherol and quercetin, *J. Agric. Food Chem.* 57:1162–1171 (2009).
- Koontz, J.L., J.E. Marcy, S.F. O’Keefe, S.E. Duncan, T.E. Long, and R.D. Moffitt, Polymer processing and characterization of LLDPE films loaded with  $\alpha$ -tocopherol, quercetin, and their cyclodextrin inclusion complexes, *J. Appl. Polym. Sci.* 117:2299–2309 (2010).
- Koontz, J.L., R.D. Moffitt, J.E. Marcy, S.F. O’Keefe, S.E. Duncan, and T.E. Long, Controlled release of  $\alpha$ -tocopherol, quercetin, and their cyclodextrin inclusion complexes from linear low-density polyethylene (LLDPE) films into a coconut oil model food system, *Food Addit. Contam. Part A Chem. Anal. Control Expo. Risk Assess.* 27:1598–1607 (2010).
- Song, Y.S., and M.S. Hepp, US Food and Drug Administration approach to regulating intelligent and active packaging components, in *Innovations in Food Packaging*, edited by J.H. Han, Elsevier, San Diego, California, USA, 2005, pp. 475–481.
- U.S. Food and Drug Administration, Food Ingredients & Packaging. [www.fda.gov/Food/FoodIngredientsPackaging/default.htm](http://www.fda.gov/Food/FoodIngredientsPackaging/default.htm) (accessed May 2012).

However, if a manufacturer wants FDA affirmation of its determination, any person may notify FDA of a claim that a particular use of a substance is exempt from the premarket approval requirements based on the notifier’s determination that such use is GRAS. FDA issues a letter that the agency has no questions at this time regarding the manufacturer’s conclusion that the compound is GRAS under the intended conditions of use. An absence of questions from FDA does not denote the agency’s agreement. Among the 428 notifications in the current GRAS notice inventory, 15 relate to antioxidants as food ingredients including quercetin, carotenoids,  $\alpha$ -tocopherol, lycopene, catechins, *trans*-resveratrol, grape seed extract, and  $\alpha$ -glycosyl isoquercitrin. However, none of the current GRAS notifications are related to antioxidants in food packaging applications.

The FCN program was intended to replace the FAP and TOR programs as the primary means by which FDA authorizes the use of food additives that are FCS. Indirect food additives are authorized for use via the FCN program, unless circumstances warrant use of the petition process. Among the 891 notifications in the current FCS inventory, 45 antioxidants are related to polymer or adhesive packaging applications. FCS notifications are effective only for the specific manufacturer, the specific FCS, and the conditions of use. Other manufacturers cannot market products containing the same FCS without filing their own notification.

## FDA perspective on active packaging materials

FDA’s premarket safety review focuses on the safety of the chemicals that may be expected to migrate from packaging into food. Therefore, it is irrelevant whether packaging materials are active, passive, or intelligent. Active releasing packaging for controlled migration of antioxidants into food for a technical effect may be considered either a direct additive or GRAS substance and should follow the FAP program or GRAS notification program. Nonmigratory active packaging (covalent grafting or immobilization) for a technical effect in the food without intentional migration may be considered an indirect additive and should follow the FCN program. Polymer-based oxygen-scavenging films and UV light absorbers are considered indirect additives and should likewise follow the FCN program.

The FDA encourages innovation in active packaging applications that may limit oxidation, maintain nutritional quality, and extend shelf life. FDA reminds developers that such materials are subject to the FAP process if components of the package are reasonably expected to become components of food or affect the characteristics of food as a result of their intended use. Several food ingredients and packaging guidance documents for industry are available on the FDA website (see the *information* box). FDA recommends that developers meet with the agency before submitting a FAP or FCN to prevent an expenditure of resources on experiments that may not provide adequate data. Before initiating studies for a submission, consultation with FDA regarding the design of experimental protocols that are likely to address FDA’s concerns will result in faster time to market for active packaging technologies.

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# Call for Papers and Posters

## XV Latin American CONGRESS and Exhibition on Fats and Oils

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### Call for Papers and Posters

The organizing committee welcomes abstract submissions for oral and poster presentations. The technical program will feature invited presentations by leading experts as well as by volunteer presenters.

Abstract submissions are encouraged on the following topics:

- ▶ Analytical techniques and applications
- ▶ Biotechnology of lipids
- ▶ Chemistry and lipid synthesis
- ▶ Detergents and soaps
- ▶ Lipid oxidation and antioxidants
- ▶ Norms, regulations, and food safety
- ▶ Oils, fats, and lipids in human and animal health and nutrition
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# CALL FOR NO



## A. Richard Baldwin Distinguished Service

This is the Society's highest service award. It recognizes long-term, distinguished service to AOCS in positions of significant responsibility.

*Nature of the Award:* \$2,000, a travel-and-expense allowance, and a plaque provided by Cargill.

**Deadline:** November 1

## AOCS Award of Merit

This award recognizes productive service to AOCS: leadership in committee activities; service that has advanced the Society's prestige, standing, or interests; and service not otherwise specifically recognized.

*Nature of the Award:* A plaque.

**Deadline:** November 1

## AOCS Fellow

The status of Fellow is awarded to members of AOCS whose achievements in science entitle them to exceptionally important recognition or to those who have rendered unusually important service to the Society or to the profession.

*Nature of the Award:* Fellow membership status and a plaque.

**Deadline:** December 1

## CALL FOR NOMINATIONS

Each award has its own specific and unique nomination requirements. For award consideration, it is essential that all paperwork be complete and received at AOCS by the nomination deadline. Self-nominations are welcomed and encouraged. Please refer to the website for the nomination requirements and submission deadlines.

## ELECTRONIC SUBMISSIONS ONLY!

AOCS is accepting nomination material only by electronic communication. Window based programs (WORD) and PDF material emailed to AOCS must include the award name and candidate name in the email subject line.



## Supelco/Nicholas Pelick-AOCS Research

This award recognizes outstanding original research of fats, oils, lipid chemistry, or biochemistry. The recipient must have published the research results in high-quality technical papers regarding fats, oils, lipid chemistry, or biochemistry.

*Nature of the Award:* \$10,000, a travel-and-expense allowance, and a plaque. The award is sponsored by Supelco, a subsidiary of Sigma Aldrich Corp, and Nicholas Pelick, past president of AOCS.

**Deadline:** November 1

## Stephen S. Chang

This award recognizes a scientist, technologist, or engineer whose distinguished accomplishments in basic research have been used by industries for the improvement or development of products related to lipids.

*Nature of the Award:* A cash prize and a jade horse, provided by the Stephen and Lucy Chang endowed fund.

**Deadline:** October 15

## AOCS Young Scientist Research

This award recognizes a young scientist who has made a significant and substantial research contribution in one of the areas represented by the Divisions of AOCS.

*Nature of the Award:* \$1,000, a plaque, and a travel-and-expense allowance provided by the International Food Science Center A/S.

**Deadline:** November 1

## Corporate Achievement

This award recognizes industry achievement for an outstanding process, product, or contribution that has made the greatest impact on its industry segment.

*Nature of the Award:* A plaque.

**Deadline:** November 1



## ACI/NBB Glycerine Innovation

The Industrial Oil Products Division initiated this award to recognize outstanding achievement for research in new applications for glycerine with particular emphasis on commercial viability.

*Nature of the Award:* \$5,000 and a plaque provided by the American Cleaning Institute and the National Biodiesel Board.

**Deadline:** November 1

## Samuel Rosen Memorial

Milton Rosen and the Surfactants and Detergents Division initiated this award to recognize a surfactant chemist for significant advancement or application of surfactant chemistry principles.

*Nature of the Award:* \$2,000 and a plaque.

**Deadline:** November 1

## Herbert J. Dutton

The Analytical Division initiated this award to recognize an individual who has made significant contributions to the analysis of fats and oils and related products or whose work has resulted in major advances in the understanding of processes utilized in the fats and oils industry.

*Nature of the Award:* \$1,000 and a plaque.

**Deadline:** November 1

## Timothy L. Mounts

The Edible Applications Technology Division initiated this award to recognize research relating to the science and technology of edible oils or derivatives in food products, which may be basic or applied in nature.

*Nature of the Award:* \$1,000 and a plaque provided by Bunge North America.


**Deadline:** November 1

# DOMINATIONS

## Edible Applications Technology Outstanding Achievement

This award recognizes a scientist, technologist, or leader who has made significant contributions to the Division's field of interest, or made contributions to the advancement of edible oils.

*Nature of the Award:* \$500 and a plaque.

**Deadline:** November 1 

## Ralph Holman Lifetime Achievement

The Health and Nutrition Division established this award to annually recognize an individual who has made significant contributions to the Division's field of interest, or whose work has resulted in major advances in health and nutrition.

*Nature of the Award:* \$500, a travel-and-expense allowance, and a signed orchid print.

**Deadline:** November 1 



## Alton E. Bailey

This award is supported by the USA Section and recognizes research and/or service in the fields of fats and oils and related disciplines.

*Nature of the Award:* \$750 and a plaque.

**Deadline:** November 1

## AAOCS Award for Scientific Excellence in Lipid Research

This award recognizes a scientist from within the Australasian region that has made a significant research contribution towards fats and oils research, either cumulative or one major advancement.

*Nature of the Award:* Travel-and-expense allowance to attend the 2013 AAOCS Section meeting and a plaque.

**Deadline:** Varies



## Thomas H. Smouse Fellowship

This award was established by the Archer Daniels Midland Foundation and the family and friends of Thomas H. Smouse. The purpose of this graduate fellowship is to encourage and support outstanding research by recognizing a graduate student pursuing an M.S. and/or Ph.D. degree in a field of study consistent with the areas of interest of AOCs.

*Nature of the Award:* The Fellowship level is up to \$15,000 (\$10,000 Fellowship, \$5,000 for travel and research expenditures related to the student's graduate program).

**Deadline:** February 1

## Ralph H. Potts Memorial Fellowship

This award recognizes a graduate student working in the field of chemistry of fats and oils and their derivatives. Qualifying research will involve fatty acids and their derivatives, such as long-chain alcohols, amines, and other nitrogen compounds.

*Nature of the Award:* \$2,000, a plaque, and travel-and-expense allowance. The award is supported by AkzoNobel, Inc.

**Deadline:** October 15 

## Honored Student

This award recognizes graduate students in any area of fats and lipids. To receive the award, a candidate must remain a registered graduate student and must not have received a graduate degree or have begun career employment prior to the Society's Annual Meeting.

*Nature of the Award:* Travel-and-expense allowance to attend and present a lecture at the Society's Annual Meeting.

**Deadline:** October 15 


## Kalustian and Manuchehr Eijadi

Each award recognizes outstanding merit and performance of one Honored Student award recipient and includes a scholarship of \$1,000.

## Hans Kaunitz

This award is supported by the USA Section and encourages studies in the sciences relating to fats, oils, and detergent technology. This award is open to graduate students within the geographical boundaries of the USA Section.

*Nature of the Award:* \$1,000, travel-and-expense allowance, and a certificate.

**Deadline:** February 1 

## AOCS Division Awards for Students

These awards recognize students at any institution of higher learning, who are studying and doing research towards an advanced degree in fats, oils, proteins, lipids, surfactants, detergents, and related materials.

The following student awards are currently being offered by these AOCS Divisions:

- Analytical Division Student Award
- Biotechnology Student Excellence Award
- Edible Applications Technology Division Student Award
- Health and Nutrition Division Student Excellence Award
- Industrial Oil Products Division Student Award
- Lipid Oxidation and Quality Division Student Poster
- Processing Division Student Excellence Award
- Protein and Co-Products Division Student Poster
- Surfactants and Detergents Division Student Travel Award

*Nature of the Award:* Awards can consist of \$100 to \$1,000 and a certificate.

**Deadline:** Varies from October 15 to January 15 

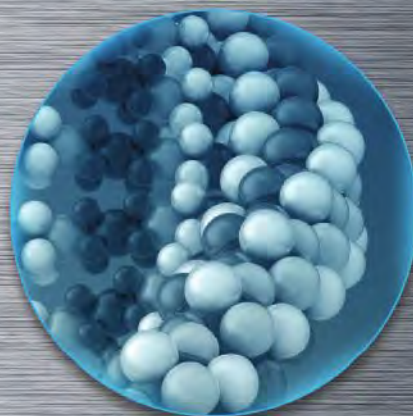


The award recipient must agree to attend the AOCS Annual Meeting & Expo and present an award address. The 104th AOCS Annual Meeting & Expo will be held in Montréal, Québec, Canada from April 28–May 1, 2013.

**AOCS Awards contact** ➤ [awards@aocs.org](mailto:awards@aocs.org) • [www.aocs.org/awards](http://www.aocs.org/awards)



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# Can you determine fatty acid composition in minutes instead of hours?

This article describes the rapid (<5 min) determination of the fatty acid composition of edible oils and fats by Fourier transform near-infrared (FT-NIR) spectroscopy and an evaluation of the transferability of calibration models among different FT-NIR spectrometers.

Magdi M. Mossoba, Hormoz Azizian,  
and John K.G. Kramer

Interest in determining the fatty acid (FA) composition of fats, oils, and other foods has increased dramatically since the introduction of regulations in the United States, Canada, and many other countries that require the total *trans* fat and total saturated fat contents of food to be stated on nutrition facts labels (total *cis* monounsaturated fat and total *cis* polyunsaturated fat contents are optional) [1–3]. Such regulations were issued in response to an emerging body of literature showing that the risk of cardiovascular disease was positively associated with the intake of isolated *trans* fat [4].

Currently, gas chromatography (GC) is the officially recognized method used to provide the complete FA composition for labeling purposes [5]. A rapid Fourier transform mid-infrared (FTIR) spectroscopy method based on second-derivative spectra was developed using attenuated total reflection cells [6,7]. Although this method allows for the rapid measurement of total *trans* fat content, it does not provide the total contents of saturated fatty acids (SFA), monounsaturated fatty acids (MUFA), or polyunsaturated fatty acids (PUFA), which limits its usefulness.

In recent years, a novel Fourier transform near-infrared (FT-NIR) spectroscopic procedure capable of determining the complete FA composition of fats and oils was developed using partial least squares calibration models [8,9]. Model development was based on accurate FA composition data obtained by a primary GC reference method [8,9]. These

**Table 1.** Fatty acid composition of six different fats and oils determined by gas chromatography and Fourier transform near-infrared spectroscopy

Product	Method*	Σ SFA	Σ <i>trans</i> FA	Σ MUFA	Σ PUFA
Partially hydrogenated soybean oil	GC	<b>18.7</b>	<b>28.6</b>	<b>27.9</b>	<b>20.4</b>
	FT-NIR Mean	18.1	28.0	29.6	22.1
	FT-NIR SD	0.8	1.5	1.9	0.5
	FT-NIR RMSECV	1.6	1.6	3.0	1.4
Soybean oil	GC	<b>16.0</b>	<b>1.6</b>	<b>21.5</b>	<b>61.6</b>
	FT-NIR Mean	16.7	1.5	22.7	60.5
	FT-NIR SD	0.7	0.2	1.8	0.8
	FT-NIR RMSECV	1.0	1.3	2.3	2.0
Canola oil	GC	<b>8.0</b>	<b>1.7</b>	<b>63.3</b>	<b>25.8</b>
	FT-NIR Mean	8.4	1.2	63.3	25.5
	FT-NIR SD	1.0	1.3	2.3	2.0
	FT-NIR RMSECV	1.6	1.6	3.0	1.4
Sunflower oil	GC	<b>11.7</b>	<b>0.8</b>	<b>24.5</b>	<b>62.7</b>
	FT-NIR Mean	11.5	1.1	24.3	62.8
	FT-NIR SD	0.3	0.2	1.0	1.6
	FT-NIR RMSECV	1.0	1.3	2.3	2.0
Coconut oil	GC	<b>89.1</b>	<b>1.5</b>	<b>7.3</b>	<b>1.8</b>
	FT-NIR Mean	89.4	1.7	7.3	1.7
	FT-NIR SD	0.7	0.5	0.4	0.2
	FT-NIR RMSECV	1.2	0.6	2.3	0.6
Palm oil	GC	<b>52.9</b>	<b>2.1</b>	<b>36.4</b>	<b>8.9</b>
	FT-NIR Mean	53.0	3.2	37.1	8.9
	FT-NIR SD	0.8	0.5	1.4	0.1
	FT-NIR RMSECV	1.2	0.6	2.3	0.6

\*GC, gas chromatography. All oils were analyzed using a 100 m SP 2560 column; see details in [8,9]. FT-NIR, Fourier transform infrared spectroscopy.

Mean of 9 independent measurements (5 instruments, 2 modes, 3 different pathlengths)

SD, standard deviation

RMSECV, root mean square error of cross validation

CONTINUED ON NEXT PAGE

models were developed by NIR Technologies Inc. ([www.nirtechnologies.com](http://www.nirtechnologies.com)) by using spectra measured on a Bruker Optics FT-NIR spectrometer ([www.brukeroptics.com](http://www.brukeroptics.com)) equipped with a fiber optic probe and Bruker OPUS operating software [8,9]. The question that needed to be addressed has been whether these commercially available models could be used to rapidly and accurately determine FA composition using data collected on any FT-NIR or dispersive (including portable, hand-held) NIR spectrometer under different measurement modes and experimental conditions. To help answer that question, three separate laboratories conducted a limited validation study to evaluate the transferability of these calibration models among five same-make FT-NIR spectrometers.

Although commonly accepted and highly dependable, a thorough GC analysis [10] could take hours and would require the use of organic solvents and chemicals and derivatization of the fat and oil to their FA methyl esters prior to separation by GC. A successful GC determination is dependent on the correct identification, accurate quantification, and sorting out of overlapping peaks, much of which is subject to the

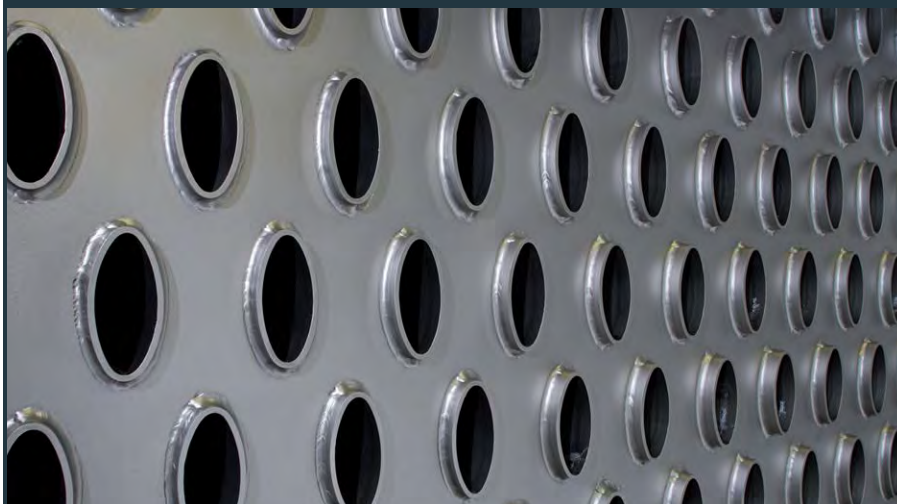
analyst's expertise. Compared to GC, FT-NIR offers several advantages [1,2,11]. There is no requirement for sample preparation of fats and oils, no need for chemicals and solvents, and no solvent disposal. The method is nondestructive to oil or melted fat samples; FT-NIR spectra can be collected in the transmission mode using glass tubes or in the transfection mode using fiber optic probes. Most importantly, it is much faster (<5 min per test sample) and less costly than GC. The collection of an FT-NIR spectrum takes less than a minute, and the application of calibration models (data analysis) to determine the FA composition is fully automated and independent of analyst bias. Because calibration models have been previously developed [8,9], their subsequent application to FA profiling is cost effective. FT-NIR is potentially an ideal screening tool for the rapid determination of total SFA, *trans* FA, *cis* MUFA, and *cis* PUFA, and it should greatly facilitate verification of compliance with food labeling regulations.

In a more extensive three-laboratory FT-NIR collaborative study, model transferability was evaluated on five different spectrometers from a single vendor, Bruker Optics, Inc. This study was designed to

explore the possibility of evaluating calibration models under different transfection and transmission measurement modes and different pathlengths, which included 2- and 4-mm fiber optic probes, and 3- and 6-mm transmission tubes by using 5- and 8-mm outer diameter (OD) glass tubes, respectively. Whereas the number of laboratories was only three, the number of independent determinations (data sets) collected under these various experimental conditions totaled nine. Six representative edible oils and fats were selected for this study. These products were typical oils that covered the type and range of FA one would find in edible oils on the market. For example, canola was selected for the high content of oleic and linolenic acids, soybean oil for the high content of linoleic and linolenic acids, palm and coconut oils for their high content of the saturated FA 12:0, 14:0, and 16:0, and the partially hydrogenated (PH) soybean oil for its high content of *trans* FA. All the oils were obtained from local grocery stores, and the PH soybean oil was a gift from Bunge Canada (Toronto, Ontario). Data analysis was obtained by applying pre-developed calibration models [8,9]. Specifically, the following calibration models were used: a very

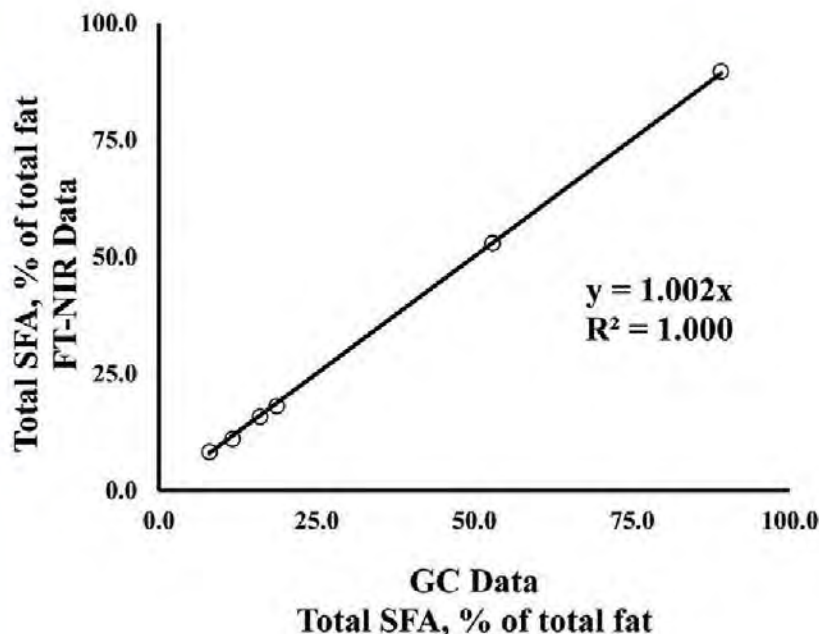
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**FIG. 1.** Pearson correlations of the saturated fatty acid (SFA) content (as % of total fat) determined by Fourier transform near-infrared (FT-NIR) and gas chromatography (GC) for each of the six fats and oils analyzed.

low *trans* FA (<3% *trans* FA) model or a low-to medium-*trans* FA (<20% *trans* FA) model for low-*trans* fat edible oils such as soybean, canola, and sunflower oils; a high-saturated FA model for coconut and palm oils; and a high-*trans* FA model for PH soybean oil. See Table 1, page 605, for a comparison of results for the six fats and oils analyzed by FT-NIR and GC. The FT-NIR results are presented as the mean  $\pm$  standard deviation (SD) of nine independent data sets obtained using five different FT-NIR spectrometers, two different modes (transfection and transmission), and three different pathlengths. The root mean square errors of cross validation (RMSECV) values were low (Table 1 on page 605) indicating a satisfactory measure of confidence in the FT-NIR determinations.

Figure 1 shows an example of excellent correlation between the FT-NIR and GC determinations with respect to the total SFA content for all six products investigated.

A paired *t*-test indicated that there was no significant statistical difference between the results of these two methodologies. Accuracy expressed as RMSECV was previously generated by the calibration models and reported to be 1% of total fat for SFA. In addition, a graphical presentation of the analysis of variance based on a unique approach

developed by Bland and Altman [12] was carried out in which the differences between GC and FT-NIR determinations are plotted against their means. A comparison of the results generated for the sums of SFA for the six products is shown in Figure 2 (page 608).

The limits of agreement between GC and FT-NIR are defined as the mean difference  $\pm 2$  SD of the differences. The Bland-Altman plot in Figure 2 was satisfactory and indicated that all values fell within these limits of agreement. By using these same comparative evaluation tests, satisfactory accuracy was also obtained for total *trans* FA, total *cis* MUFA and total *cis* PUFA.

A detailed discussion of accuracy and precision data obtained in this study was recently published in the *Journal of the American Oil Chemists' Society* [H. Azizian, J.K.G. Kramer, M.M. Mossoba, Evaluating the transferability of FT-NIR calibration models for fatty acid determination of edible fats and oils among five same-make spectrometers using transmission or transfection modes with different pathlengths; doi: 10.1007/s11746-012-2116-9, 2012].

Regarding the evaluation of data at different pathlengths, the results obtained using

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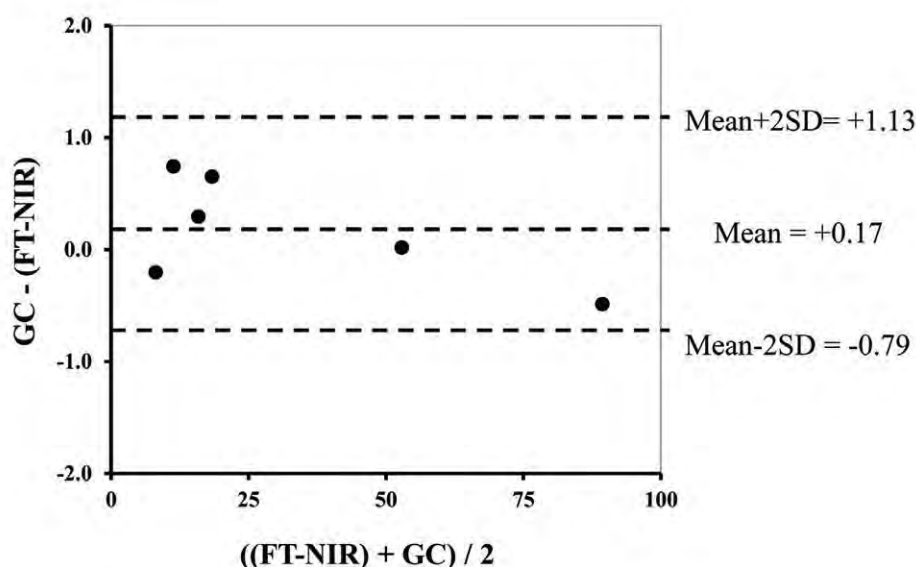
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**FIG. 2.** The mean difference between the SFA content determined by GC and FT-NIR for the six fats and oils investigated are presented as described by Bland and Altman [12]. Values outside the mean difference of  $\pm 2$  standard deviations are considered significant [12]. For abbreviations, see Figure 1.

the 8-mm OD (6-mm pathlength) transmission tubes led to large SD relative to those obtained with pathlengths of 2-, 3-, or 4-mm under the experimental conditions used. Figure 3 shows significant discrepancies in the quantification of total SFA and total PUFA using the 8-mm OD tubes (Fig. 3).

The absorption values for the 8-mm OD tubes were larger than 3.0 absorbance units, which is significantly outside the linear range of Beer-Lambert's law. Therefore, it is recommended that these calibration models be applied to FT-NIR spectra collected only with pathlengths that are equal to or less than 4 mm. The use of long-pathlength transmission tubes would have dictated the exclusion from the calibration models of an important wavelength range (near  $5800 \text{ cm}^{-1}$ ) where saturation occurred. This range was critically needed for the development of some of our models for accurate FA profiling. By opting to use smaller pathlengths, we eliminated the need to exclude such an important spectral range from our calibration models. Research is ongoing to examine the transferability of these calibration models to FT-NIR spectrometers from other vendors.

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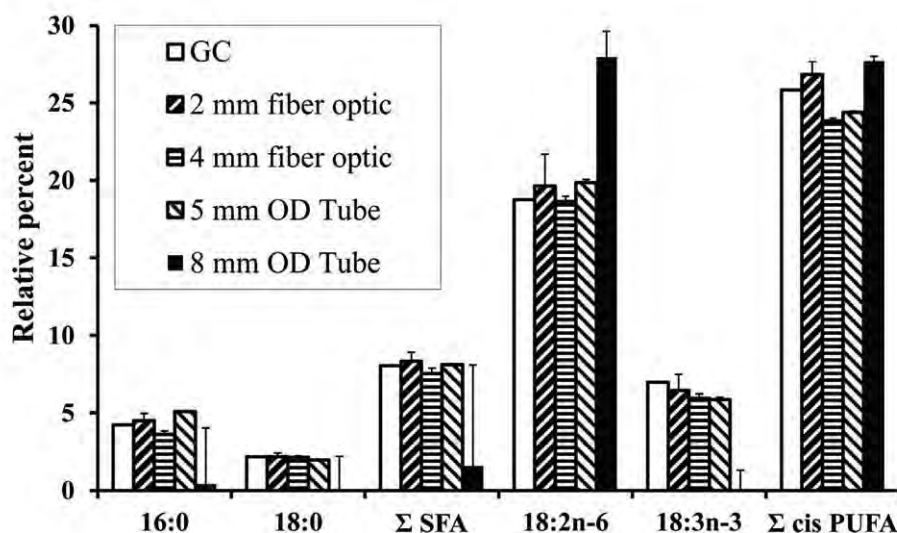
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For labeling purposes, the total *trans* FA content does not need to be reported unless it exceeds the legal definition of 0 grams *trans* fat/serving which is <0.5 grams/serving in the United States [1] and <0.2 grams/serving in Canada [2]. Owing to differences in serving size declared on labels for edible fats and oils, these limits correspond to means of <3.6% *trans* fat (as percentage of total fat) in the United States and <2.2% *trans* fat (as percentage of total fat) in Canada. Both the FT-NIR and GC results of all the unhydrogenated processed edible fats and oils in this study met the legal definition of 0 grams *trans* fat/serving in both countries. On the other hand, the total *trans* FA content of the PH soybean oil averaged 28% of total fat (Table 1).

In addition, as already mentioned, the declaration of the contents of total *cis* MUFA and total *cis* PUFA is optional in the current US and Canadian regulations. This information would be needed to distinguish between several vegetable oils such as soybean, corn, and olive oils since their SFA content is similar (14–16%); only sunflower (11%) and

CONTINUED ON NEXT PAGE



**FIG. 3.** Comparison of fatty acid (FA) content (as % of total fat) determined by FT-NIR and GC for the two major saturated FA (16:0 and 18:0), total SFA, selected polyunsaturated fatty acids (PUFA: 18:2n-6 and 18:3n-3), and total PUFA for canola oil. The following FT-NIR measurement modes and pathlengths were compared: fiber optic probes at 2 mm and 4 mm, and transmission tubes of 5 mm (equivalent to 3-mm outside diameter [OD]) and 8 mm (equivalent to 6 mm OD), respectively. For other abbreviations, see Figure 1.

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canola oils (7%) have slightly lower levels of SFA. We also found three different types of sunflower oils in the marketplace; some were high in linoleic acid, others high in oleic acid, and others had intermediate levels of linoleic and oleic acids [www.sunflowernsa.com/oil, accessed June 14, 2012]. Based only on their SFA and *trans* FA contents, these oils would not be readily distinguishable. Knowledge of the *cis* MUFA and n-6 and n-3 *cis* PUFA contents of fats and oils would be particularly valuable to assess their nutritional value based on the presence of essential FA, and to evaluate their suitability and stability for use as frying oils.

In conclusion, calibration transfer among the three participating laboratories equipped with five different same-vendor FT-NIR spectrometers was successful. The application of pre-developed calibration models gave satisfactory precision and accuracy under the experimental conditions used. Pathlengths of 2- and 4-mm (fiber optic probes) and approximately 3-mm transmission tubes (5-mm OD) are recommended. Unlike GC, FT-NIR spectroscopy requires no sample preparation for the FA determination of edible fats and oils. The FT-NIR protocol for calculating FA composition is fully automated and independent of analyst skill or bias because calibration models are pre-developed and cannot be subjectively modified. FT-NIR is a rapid (<5 min) and viable alternative method to the time-consuming and labor-intensive GC methodology that takes hours. The potential for applying FT-NIR to the routine screening of edible fats and oils for their total SFA, *trans* FA, MUFA and PUFA contents for regulatory compliance monitoring in both the United States and Canada will be further investigated.

*Magdi M. Mossoba is a research chemist at the US Food and Drug Administration (FDA) in College Park, Maryland, USA. An authority on the application of FTIR spectroscopy (and hyphenated GC-FTIR techniques) to problems of characterization and quantification of food constituents (including lipids), contaminants, and additives, Mossoba has co-authored over 100 peer-reviewed publications, is the chair of the AOCS Books and Special Publications Committee, and is a co-chair of the Research Scientist Peer Review Committee at the FDA. He was the recipient of the AOCS Herbert J. Dutton Research Award in 2008. He can be contacted at Magdi.Mossoba@fda.hhs.gov.*

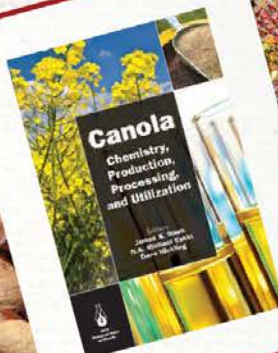
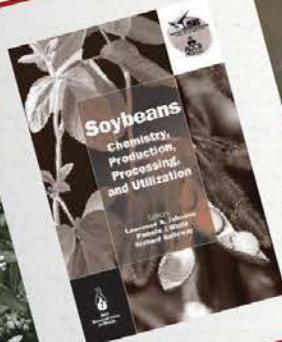
*Hormoz Azizian is the founder of NIR Technologies Inc., Ontario, Canada, specializing in the nondestructive characterization of materials using Fourier transform near-infrared spectroscopy. His current focus has been on the development of rapid FT-NIR methodologies for the analysis of food products including fats and oils, particularly trans-fat content, as well as for the noninvasive determination of body fat content. He can be contacted at hazizian@nirtechnologies.com.*

*John K.G. Kramer was a research scientist with Agriculture and Agri-Food Canada from 1970 to 2010, where he led a team to evaluate the safety of canola oil for human consumption. That work culminated in the successful award of GRAS [generally recognized as safe] status for canola oil in the United States in 1985. He is the author of 230 refereed papers, 25 chapters, and four books; has been an associate editor of Lipids since 1988; has served on the National Research Council Expert Committee on Fats and Oils and other Lipids, National Standards Board of Canada, Canola Council of Canada, and Codex Alimentarius Committee on Fats and Oils (1984–1998); and received the Canada Merit Award in 1983, the CSP Canola Research Award in 1984, and the AOCS Herbert J. Dutton Research Award in 1999.*





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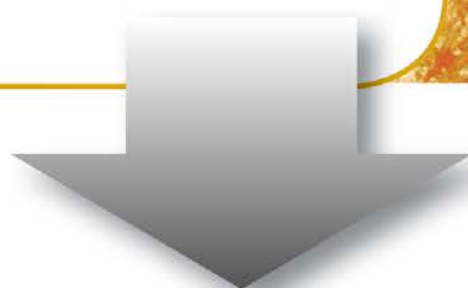
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# Advances in field-portable mass spectrometers for on-site analytics

Christopher C. Mulligan  
and Kyle e. Vircks

We live in an information-driven society deluged by advances in wireless communications, the Internet and integrated computer networks, and various other technologies. When compared to previous generations, we are spoiled by the way information has become available at the push of a button or click of a mouse. Gone are the days of using a card catalog or *Chemical Abstracts* to search for scientific literature (or even of setting foot into a library), as it is quicker and easier to get the information we need through e-journals and web resources such as *SciFinder* and *Web of Knowledge*.

We are just as impatient for chemical information. Identifying unknown species and determining physical properties, quantities, and specific interactions of target analytes is essential for many fields of science, so it is not surprising that the pursuit of higher-performing chemical instrumentation and associated methods that allow chemical information to be acquired “on demand” drives the fields of analytical chemistry and engineering. Realistically, however, the speed of chemical analysis is typically limited by sample preparation time and the throughput of the chemical instrumentation that is used. Following are some strategies that are being used to overcome these limitations.

One of the greatest increases in sample throughput could come from reducing or eliminating the time and resources required to transport samples from the field to the analytical laboratory, a necessity for most instrumentation classes that can add minutes to several days to an overall analysis, depending on the target of the study. As previously

discussed (*inform* 20: 625–627, 676, 2009), mass spectrometry (MS) is highly amenable to miniaturization, allowing analysts to take portable MS systems into the field. This effectively brings the chemistry lab to the sample and eliminates sample shipment/transport.

## Commercially available portable MS systems

One of the most mature categories of portable MS systems are those that use gas chromatographic separation (GC/MS), brought on by the need for rapid, on-site analysis of volatile and semivolatile species important to human health, homeland security, and environmental monitoring. Several rugged instruments are commercially available. Torion Technologies, Inc. (Tridion-9) and Smiths Detection (Guardion) both offer suitcase-style GC/MS systems that use solid-phase microextraction (SPME) fibers for sample

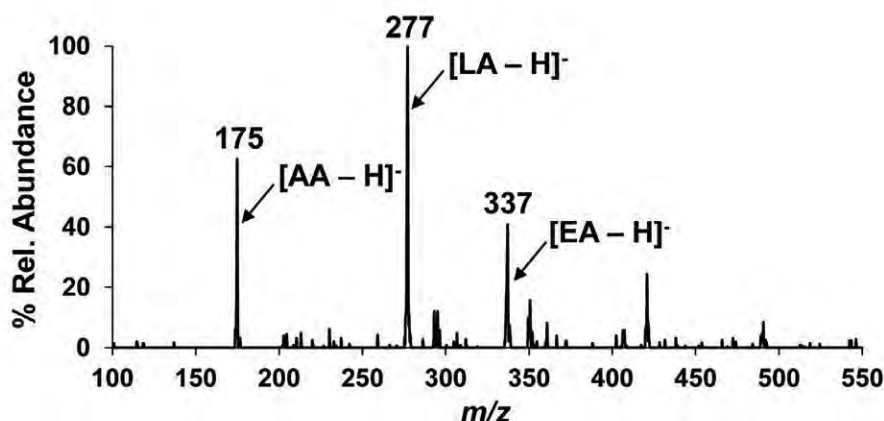
introduction and low thermal mass GC assemblies to expedite chromatographic separation. Flir Systems, Inc. has developed the Griffin 460, one of the most versatile portable GC/MS products available, which allows sample introduction via direct syringe injection, direct air sampling, purge-and-trap and headspace methods, and SPME. The Griffin 460 is also compatible with Flir’s portable, sorbent tube air sampler, the X-Sorber. These handheld devices allow both time- and volume-based collection in harsh environments, record both operator and sample information to preserve chain of custody, and store sample location via an on-board global-positioning system. Analysis of volatile species without the need for GC separation has also been demonstrated, as is the case for the 1st Detect MMS-1000, which uses selective membrane introduction, and the Syft Technologies Ltd.



**FIG. 1.** Photograph of the Microsaic Systems 3500 MiD miniature mass spectrometer, showing the small footprint of the instrument. Photo courtesy of Microsaic Systems PLC.

CONTINUED ON NEXT PAGE





**FIG. 2.** Negative-ion desorption electrospray ionization (DESI) mass spectrum of ascorbic acid (AA) and the fatty acids linolenic acid (LA) and erucic acid (EA) collected on the Flir Systems Atmospheric Inlet MS (AIMS) portable mass spectrometer. Each analyte was deposited onto a Teflon slide at a mass of 165 ng. Even at trace levels, the deprotonated molecule of each analyte is clearly seen at high intensity.

Voice 200, which uses mass-selected reagent ions to ionize and detect target species and is robust to high-moisture samples.

Miniaturized systems have also been developed to target semi- and nonvolatile species, using ionization methods such

as electrospray ionization (ESI) and atmospheric pressure chemical ionization (APCI). ESI and APCI, which produce ions at atmospheric pressure, offer a significant engineering challenge to instrument development because of the need to sample externally generated ions with reduced-size vacuum systems. There are fewer commercial instruments of this type, but those that are available offer high performance and flexibility in application despite their small form factor. The Advion, Inc. *expression* is a compact quadrupole MS system that has been designed specifically for use in fume hoods and can be coupled to high-performance (HPLC) and ultra-high performance liquid chromatography separation through both ESI and APCI ionization sources. Microsaic Systems offers one of the smallest footprint ESI-capable MS systems available. The Microsaic 3500 MiD, as seen in Figure 1 on page 613, utilizes “plug and play” chip-based components produced via microelectromechanical systems fabrication techniques, reducing the size of the ionization source, vacuum interface, and quadrupole mass analyzer. While applicable as a detector for HPLC, this system has also been implemented in continuous reaction

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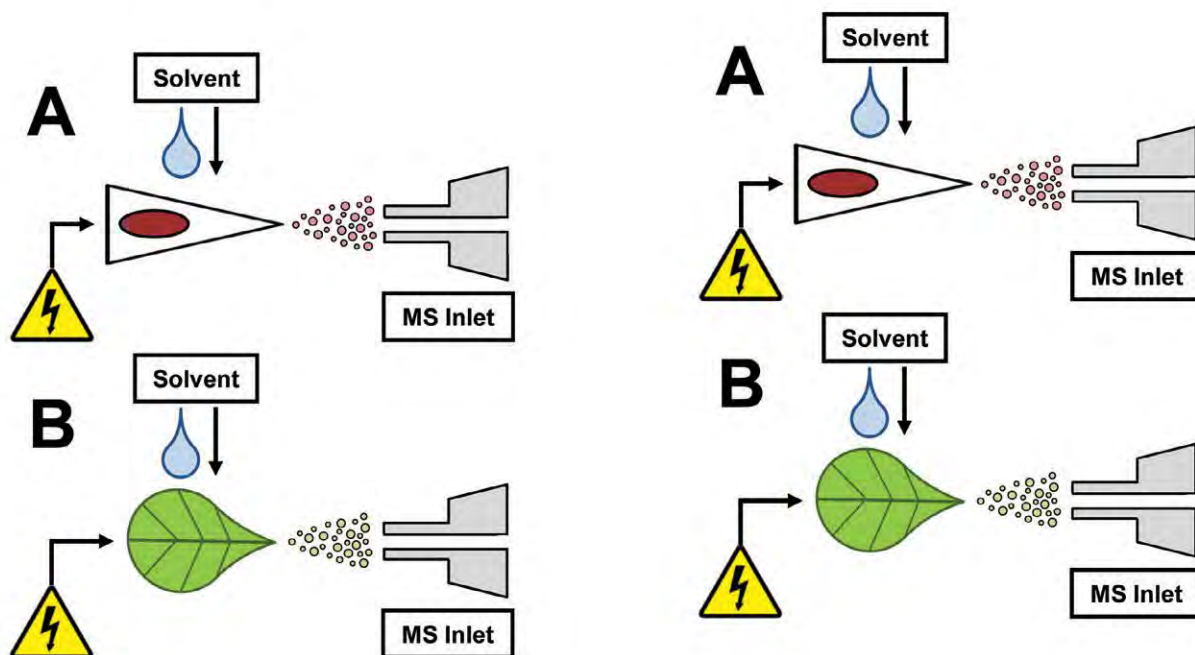
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**FIG. 3.** Schematics of the newly-reported ionization methods (a) paper spray ionization and (b) leaf spray ionization. Both methods offer advantages in terms of minimal sample preparation and flexibility in implementation.

monitoring by coupling to flow chemistry systems, allowing the observation of reaction intermediates and competing synthetic pathways of benzyne synthesis via diazotization (Browne *et al.*, 2012).

## Ambient MS methods

Portable MS systems that are coupled to GC and LC separation have revolutionized many analyses once confined to the laboratory, but they still require extensive sample preparation due to the constraints of chromatography, thus increasing total analysis time. To combat the need for sample preparation, several portable MS platforms have been developed to allow coupling to ambient ionization methods (ambient MS), negating the need for chromatographic separation. Ambient MS methods, as recently reviewed (Huang *et al.*, 2011), use a variety of different mechanisms to desorb and/or ionize target analytes directly from samples of interest, including complex matrices such as biological fluids, foodstuffs, cosmetic and pharmaceutical formulations, and tissue cross-sections.

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The research groups of R. Graham Cooks and Zheng Ouyang at Purdue University have pioneered the development of portable MS systems capable of ambient ionization methods (Xu *et al.*, 2010), and a ruggedized commercial product offering similar capability, the Flir Systems Atmospheric Inlet MS (AIMS), is now available. The systems from Cooks and coworkers and Flir Systems have been extensively applied to desorption electrospray ionization (DESI) for analyte ion generation, a well-established ambient method that uses a pneumatically assisted electrospray to desorb neutral analyte from samples of interest as secondary ions that are detected via MS.

Coupling DESI to high-performance portable MS systems offers intriguing capabilities, particularly in trace surface analysis. Figure 2 on page 614 shows a negative-ion DESI mass spectrum acquired from trace residues of ascorbic, linolenic, and erucic acids with the Flir Systems AIMS. Even at low deposited amounts (165 ng each), each analyte is seen with high sensitivity and selectivity due to MS/MS capability. Detection limits in the low- to sub-nanogram range are commonly obtained on these systems.

The maturation of portable MS systems capable of ambient ionization could not come at a better time, as we are arguably in the golden age of MS ionization method development. In a recent review by Weston (2010), 29 different ambient MS methods appeared in the literature between 2004 and 2009, and novel ways to analyze samples directly continue to be developed. Although not every ambient MS method can be coupled to portable instrumentation, several have been demonstrated or hold high promise for implementation. For instance, García-Reyes and co-workers (2009) used low temperature plasma probe—a plasma-based method that uses energetic particles created from a discharge gas such as helium to desorb/ionize analyte—for quality assessment of raw olive oil samples. Besides potential adulterants, free fatty acids, phenolics, and associated volatile species were all readily detected with this method. Eberlin and coworkers (Alberici *et al.*, 2009) developed easy ambient sonic-spray ionization (EASI), which is solvent-based like DESI but creates charged droplets purely through high-pressure nebulizing gases rather than voltage assistance. By using EASI-MS, direct characterization of main soybean biodiesel components was accomplished, including free fatty acids and triglycerides, depending on the quality of the fuel.

Work continues toward broadening the applicability and simplifying the design of ambient MS methods. One of the simplest ambient MS methods reported is paper spray ionization, as depicted in Figure 3a (page 615). Paper spray MS allows generation of gas-phase analyte ions of samples deposited onto chromatographic paper that has been pre-cut into triangles. After application of a small aliquot of solvent to the paper and high voltage via a spring clip electrical connection (e.g., an alligator clip), pre-loaded analytes migrate through the paper to the tip, where they are ionized via localized electrospray and field ionization mechanisms. While a paper spray ionization source can be assembled from general consumables found in most labs, it has proven to be highly useful, particularly for therapeutic drug monitoring from whole blood spots (Espy *et al.*, 2012) and contaminant analysis from foodstuffs such as meat, powdered spices, and milk (Zhang *et al.*, 2012). Interestingly, it seems that in the case of plant tissues, one can bypass the use of chromatographic paper by substituting in a triangular section of the tissue itself, a technique denoted as leaf spray MS. As seen in Figure 3b (page 615), it is operationally similar to paper spray MS. A recent report shows that leaf spray MS can be used to screen pesticide residues directly from the peel and pulp of fruits and vegetables, including independent analysis of the flavedo and albedo components of orange peel (Malaj *et al.*, 2012).



**List of commercially available portable MS systems**

Company	Instrument	Website
1 <sup>st</sup> Detect	MMS-1000	<a href="http://www.1stdetect.com">www.1stdetect.com</a>
Advion, Inc.	expression	<a href="http://www.advion.com">www.advion.com</a>
Flir Systems, Inc.	Griffin 60	<a href="http://gs.flir.com/technology/mobile-labs">gs.flir.com/technology/mobile-labs</a>
	AIMS	
Microsaic Systems, Plc.	3500 MiD	<a href="http://www.microsaic.com">www.microsaic.com</a>
Torion Technologies, Inc.	Tridion-9	<a href="http://www.torion.com">www.torion.com</a>
Smiths Detection	Guardion	<a href="http://www.smithsdetection.com">www.smithsdetection.com</a>
Syft Technologies, Ltd.	Voice 200	<a href="http://www.syft.com">www.syft.com</a>

The combination of ambient ionization methods with portable MS technologies offers great potential in chemical analysis, and researchers from various fields of science are already taking advantage of the enhanced throughput and negligible preparation requirements. Portable MS systems continue to be produced with smaller form factor and higher performance, but due to the restrictive nature of critical instrumental components, size reduction may never reach the point where we each have our own personal mass spectrometer. Then again, that's what "they" said about the computer. . .

*Christopher C. Mulligan is an assistant professor of analytical chemistry at Illinois State University (Normal, Illinois, USA), where his research*

*involves the design and application of portable MS instrumentation with specific interest in method development for homeland and travel security, food safety, forensic science, and environmental monitoring. He is currently developing portable MS systems for use in crime scene investigation with the help of funding from the US National Institute of Justice. Mulligan can be contacted at [mulligan@ilstu.edu](mailto:mulligan@ilstu.edu).*

*Kyle E. Vircks is an M.S. candidate in the Department of Chemistry at Illinois State University (Normal, IL). His current research in the Mulligan lab involves the utilization of portable instrumentation and ambient mass spectrometry in forensic science. His specific areas of interest in forensic science include drug chemistry and fire debris analysis.*

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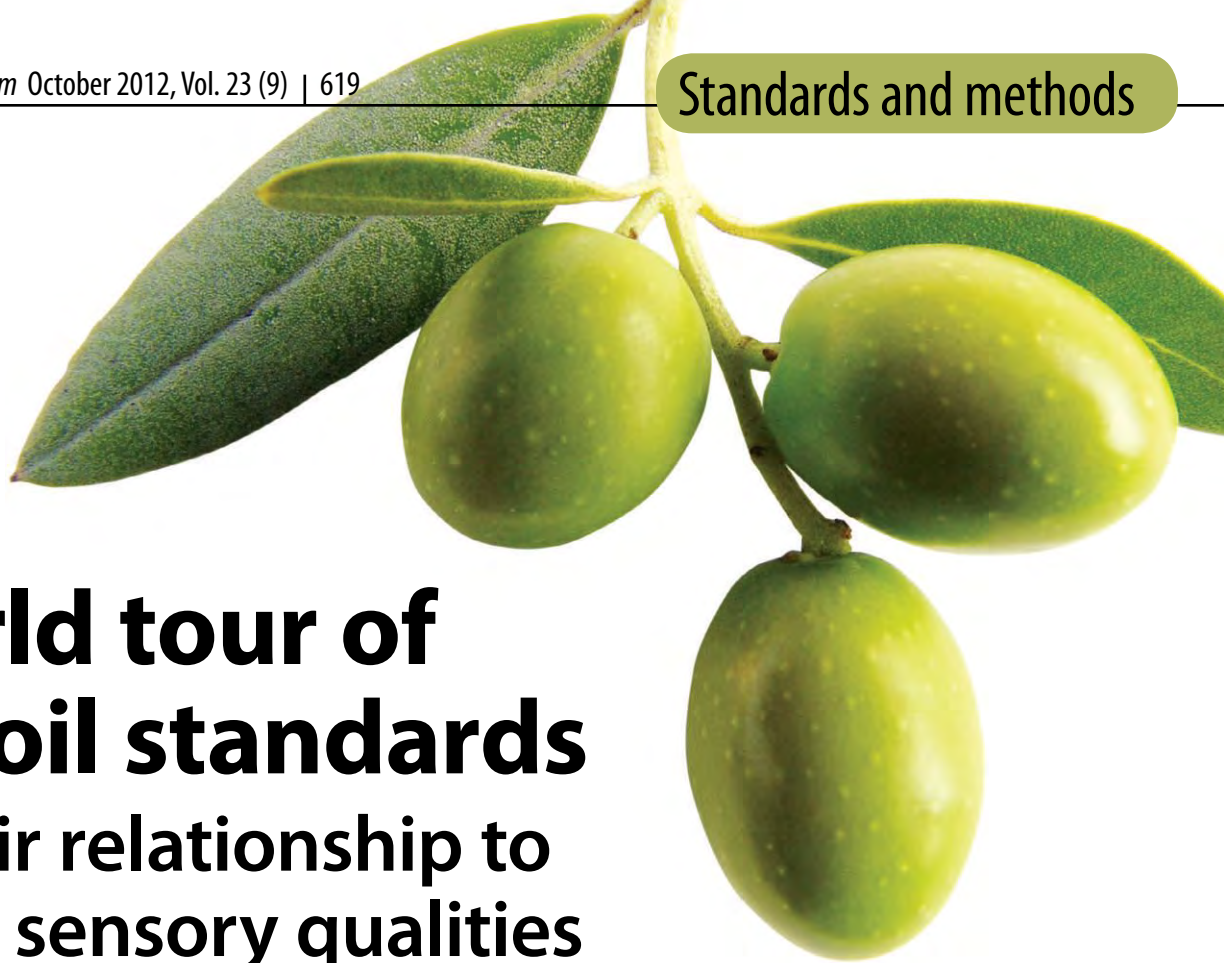
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# A world tour of olive oil standards and their relationship to olive oil sensory qualities

**Rod Mailer**

Incidents of adulteration, substitution of poor quality oil, and mislabeling of refined oil products have underscored the importance of international standards for olive oil. Having clear standards that everyone in the world agrees on helps customers know what they are buying—particularly when the oil comes from a distant location. Clear standards ensure safety and give producers a set of measurable metrics to strive toward. They discourage fraud by verifying whether products are genuine and, most importantly, they provide a gauge for freshness and quality.

Standards refer to the methods by which oils are tested and the limits that are applied to each of those tests. There are many organizations that produce standard methods:

- AOCS—American Oil Chemists' Society
- IOC—International Olive Council
- ISO—International Organization for Standardization
- AOAC International—Association of Official Agricultural Chemists
- CEN—European Committee for Standardization or Comité Européen de Normalisation

There are also several organizations that set limits for those standard methods, including:

- IOC—International Olive Council
- EC—European Commission
- CAC—Codex Alimentarius Commission

**Table 1.** List of International Olive Council (IOC) members and date of membership (last update: December 2010)

European Union		European Union	
Tunisia:	1956	Syria:	1997
Libya:	1956; rejoined 2003	Croatia:	1999
		Jordan:	2002
		Iran:	2004
Morocco:	1958	Montenegro:	2007
Israel:	1958	Iraq:	2008
Algeria:	1963	Albania:	2009
Egypt:	1964	Argentina:	2009
Lebanon:	1973	Turkey:	2010

- National governments that set their limits based on these bodies or other standards.

Other bodies, such as the Federation of Oils, Seeds and Fats Associations (FOSFA), are concerned with world trade in oilseeds, oils, and fats. They do not set standards but work with buyers and sellers to set limits on which they agree to make trades.

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**Table 2.** IOC-recognized laboratories 2012

Chemical testing laboratories recognized by IOC	Sensory laboratories recognized by IOC
Argentina	Australia
Australia	Argentina
Canada	Cyprus
France	Czech Republic
Greece	France
Italy	Germany
Morocco	Greece
Portugal	Israel
Slovenia	Italy
Spain	Jordan
Syria	Morocco
Tunisia	New Zealand
Turkey	Portugal
USA	Slovenia
	Spain
	Syria
	Tunisia
	Turkey

Unfortunately, having so many organizations setting limits and methods to analyze olive oil has created confusion for producers, traders, and consumers. Undoubtedly, a single international standard is essential to simplify trade, to combat fraud, and to provide a common goal for producers.

## International Olive Council

The International Olive Council (IOC) is an international intergovernmental organization set up in Madrid, Spain, in 1959, under the auspices of the United Nations. The Council's mission is to contribute to the development of olive growing and serve as a world forum for discussing policymaking issues and for meeting present and future challenges.

The IOC has had a very strong influence on olive oil standards, particularly in the traditional growing areas of the Mediterranean zone. This has helped standardize testing and identify instances of fraud. The IOC introduced the term extra virgin olive oil, which has become internationally recognized as the best grade of genuine and fresh olive oil. The members of the IOC are listed in Table 1 (page 619).

Many other countries (Table 2) also use the expertise of the IOC, particularly in developing a roster of internationally recognized laboratories for chemical testing and sensory analysis.

## National standards

The standards used in most countries are based on those of the IOC. Many organizations, including CAC and the European Commission, also base their methodologies and their limits on IOC. In the United States, Australia, and South Africa, modifications of IOC standards are used to cover natural variation within products. Canada uses IOC methods but has a unique approach. If oil is tested and fails to meet one of the parameters, other tests are used to confirm its authenticity. Although many countries use IOC limits, they often acknowledge that their products, while of high quality, are often outside those limits.

**Table 3.** Comparison of grades of olive oil for organizations and countries (\*same as for IOC)

	IOC	Codex	eC	Australia	USA	China
1	<b>extra virgin</b>	*	*	*	USA *	*
2	<b>Virgin</b>	*	*	*	USA *	<b>Medium grade virgin</b>
3	<b>Ordinary virgin</b>	*	NO	NO	NO	NO
4	<b>Lampante virgin</b>	NO	*	*	USA *	*
5	<b>Refined</b>	*	*	*	USA *	*
6	<b>Olive oil (blend of refined and virgin olive oils)</b>	*	Olive oil composed of refined and virgin olive oil	Olive oil composed of refined and virgin olive oil	USA *	<b>blended olive oil</b>
7	<b>Crude olive-pomace oil</b>	NO	Crude olive-residue oil	*	USA *	*
8	<b>Refined olive-pomace oil</b>	*	Refined olive-residue oil	*	USA *	*
9	<b>Olive-pomace oil (blend of refined pomace oil and virgin olive oils)</b>	*	Olive-residue oil	Olive-pomace oil—composed of refined olive—pomace oil and virgin olive oils	USA *	Blended olive-pomace oil

Israel has adopted IOC regulation but, according to an industry representative, in reality only free fatty acid and peroxide levels are used in local trade. According to this industry representative, oils from Israel often do not meet IOC standards—particularly with respect to linolenic acid levels and  $\Delta^7$ -stigmastanol. It has been shown that campesterol levels of some Israeli cultivars such as Barnea are also often above the IOC limits (Mailer, R., J. Ayton, and K. Graham, The influence of growing region, cultivar and harvest timing on the diversity of Australian olive oil, *J. Am. Oil Chem. Soc.* 87:877–884, 2010), and this is true of these cultivars in many countries.

In spite of the benefits IOC limits provide, there are deficiencies in that the regulations do not allow for natural variation in a product that is grown over a wide environmental range. As a result, there has been a divergence from the IOC standard, even within the international organizations. For example, IOC, EC, and CAC have different “classifications” for olive oil. CAC has no limit for linolenic acid, whereas IOC has a limit of 1.0%. The limit for 2-glycerol monopalmitate is  $\leq 0.9\%$  for the EC and IOC standard whereas the CAC standard is 1.5%. Individual countries also vary in their domestic standards. Some of the deviations for national standards from IOC classification of olive oil are shown in Table 3.

## evidence of oils that fail to meet IOC standards

Much has been written about the limitations of existing standards in recent years. Research scientists in Australia have identified numerous samples of particular cultivars, or cultivars grown in various environmental conditions, that are outside the recognized limits. Since 2001, Australia has illustrated to CAC that the limit for linolenic acid is too low to cover olive oil grown over a range of sites. It has also been shown in many reports that campesterol limits for cultivars regularly exceed IOC limits, regardless of where they are grown. Evidence of samples exceeding 4.0% campesterol has been published in:

- Spain—cv. Cornicabra. 75% of samples over four seasons (Salvador *et al.*, *Food Chem.* 74:267–274, 2001)
- Australia—cv. Barnea. 94% average 4.5% (Mailer and Ayton, RIRDC Report 2008, [www.rirdc.gov.au](http://www.rirdc.gov.au))
- Argentina—cv. Arbequina. 70% maximum 5.5% (Ceci and Corelli, *J. Am. Oil Chem. Soc.* 84:1125–1136, 2007)

**Table 4.** Comparison of trade standards (%) for fatty acid and sterol profiles (\*same as for IOC)

Fatty acid	IOC	Codex	Australia	USA	South Africa
Myristic	$\leq 0.05$	*	*	*	*
Palmitic	7.5–20.0	*	7.0–20.0	*	7.0–20.0
Palmitoleic	0.3–3.5	*	*	*	*
Heptadecanoic	$\leq 0.3$	*	*	*	*
Heptadecenoic	$\leq 0.3$	*	<0.4	*	<0.4
Stearic	0.5–5.0	*	*	*	*
Oleic	55.0–83.0	*	53.0–85.0	*	53.0–85.0
Linoleic	3.5–21.0	*	2.5–22.0	*	2.5–22.0
Linolenic	$\leq 1.0$	No std.	<1.5	<1.5	<1.5
Arachidic	$\leq 0.6$	*	*	*	*
Gadoleic	$\leq 0.4$	*	$\leq 0.5$	*	$\leq 0.5$
Behenic	$\leq 0.2^*$	*	*	*	*
Lignoceric	$\leq 0.2$	*	*	*	*
Cholesterol	$\leq 0.5$	*	*	*	
Brassicasterol	$\leq 0.1$		*	*	
Campesterol	$\leq 4.0$		$\leq 4.8$	$\leq 4.5$	
Stigmasterol	< Campesterol		$\leq 1.9$	*	
Delta-7-stigmastanol	$\leq 0.5$		*	*	
Apparent $\beta$ -sitosterol	$\geq 93.0$		$\geq 92.5$	*	

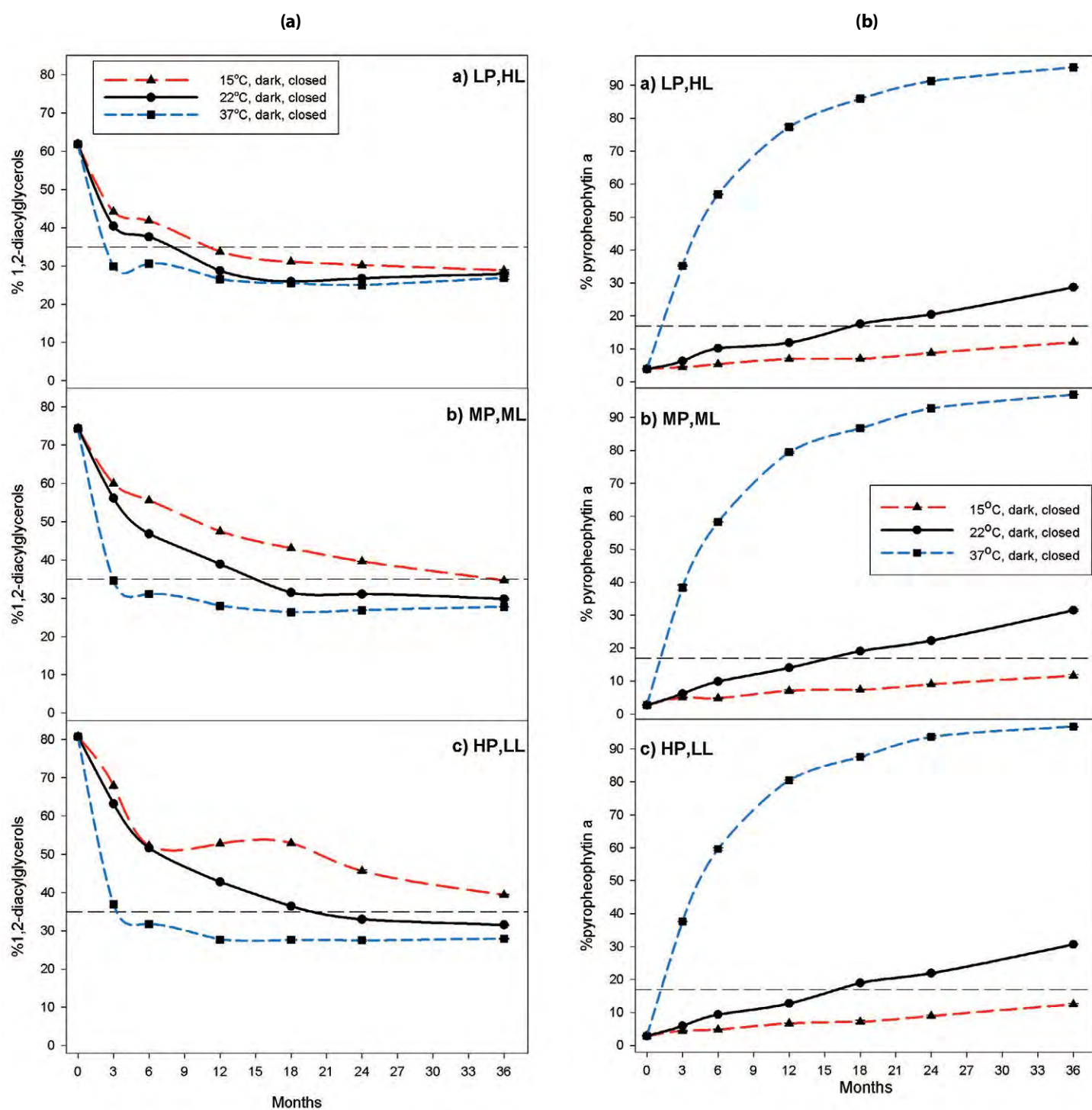
- Greece—cv. Koroneiki. Average of 72 samples = 4.2% (Koutsakis *et al.*, *J. Am. Oil Chem. Soc.* 76:1477–1481, 1999)

From the combined database of the Australia Oils Research Laboratory and Modern Olives Laboratory for samples from Australian producers, analyzed in 2010, 39% (254 of 651) exceeded 4.0% campesterol, including samples from multiple seasons, regions, and varieties.

Individual countries have made amendments to their own national standards, in particular to fatty acid profiles, although this parameter has limited usefulness in identifying fraud and has virtually no value in determining quality. The Australian standard for fatty acid composition, which was originally based on IOC limits, has been expanded to recognize the actual values that represent high-quality Australian olive oil. CAC removed the limit for linolenic acid because various countries were

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**FIG. 1.** Effects of temperature on the ratio of 1,2-diacylglycerol (a) and pyropheophytin A (b) on three different types of oil. Horizontal line shows reject point.

unable to agree on an acceptable level. The United States has adopted the original CAC level of 1.5% for linolenic acid to suit the oil processed there. (Linolenic acid limits vary according to where olives are grown and harvested.) An example of some of the country-specific changes for fatty acids and phytosterol levels is shown in Table 4 (page 621).

Despite this, the differences among countries are not as great as these data may suggest. In fact, there is universal agreement to the limits

recognized for most tests including unsaponifiable matter, *trans*-fatty acid content, wax content, difference between actual and theoretical ECN 42 (equivalent carbon number), stigmastadiene content, trace metals (mg/kg), free fatty acids, peroxide value, and absorbency in the ultraviolet. There is also no disagreement about the measurement of sensory characteristics and the levels for attributes and defects as described by the IOC.

Perhaps the most important deficiency of international standards is the lack of an agreed method to determine the freshness of oil. The Australian industry has tried to overcome this by adopting two methods that have been modified and validated by the German Society of Fat Research (DGF; see <http://tinyurl.com/DGF-Methods>). These methods are the 1,2-diacylglycerol method and measurement of pyropheophytin A (both of which have been adopted by ISO). Australia has set a limit for 1,2-diacylglycerol of  $\geq 35\%$  and for pyropheophytin A of  $\leq 17\%$  to avoid rejecting good-quality oil.

These two methods, as used in the Australian standard, have been adapted from DGF and the amended methods published by ISO. They show excellent agreement with sensory analysis and agreement with other oxidative stability methods. Research studies have confirmed their usefulness in determining freshness in olive oil by numerous shelf life studies. Some research being carried out at the Australian Oils Research Laboratory to justify these methods is shown in Figure 1. Three types of oil were tested:

- A “fragile” oil: low phenolic antioxidants and high polyunsaturated fatty acid
- A medium oil: medium phenolic antioxidants and medium polyunsaturated fatty acid
- A stable oil: high phenolic antioxidants and low polyunsaturated fatty acid

The oils were stored at a range of temperatures (15°C, 22°C, and 37°C shown here) over three years. Temperature was shown to strongly influence the ratio of 1,2-diacylglycerol and pyropheophytin A levels (Fig. 1). These results correlated with sensory analysis, which was carried out by the Australian Sensory Panel, and the expected degradation of the three types of oil. These results give good credibility for the use of these methods to distinguish oils that have been badly stored or even have been through a refining process. The full study report can be downloaded from [www.rirdc.gov.au](http://www.rirdc.gov.au).

## UC Davis Report

The most common tests carried out on extra virgin olive oil are free fatty acids (FFA), peroxide value (PV), and fatty acid profile (FAP). In a series of studies on olive oils on supermarket shelves in California, the University of California, Davis ([www.olivecenter.ucdavis.edu](http://www.olivecenter.ucdavis.edu)) illustrated that a large portion of the oils failed to meet extra virgin olive oil standards, based on IOC tests (Table 5). However, none of those samples failed the FFA, PV, or FAP test. The one test that did show good correlation with the sensory analysis was the UV test. Additionally, the new tests, diacylglycerol and pyropheophytin A, also showed good relationship with sensory analysis.

**Table 5.** Comparison of sensory and chemical quality of olive oils selected from supermarkets in California

		Percentage of samples failing sensory analysis that were confirmed by chemical tests					
		Sensory		IOC chemical test		DGF/Australian tests	
BRAND	# of samples failing sensory analysis	FFA	FAP	PV	UV K <sub>268</sub>	DAGs	PPP
California Olive Ranch	0	0	0	0	0	0	0
Cobram Estate	0	0	0	0	0	0	0
Lucini	2	0	0	0	100	0	0
Filippo Berio	15	0	0	0	33	33	20
Star	11	0	0	0	0	64	27
Bertolli	13	0	0	0	8	62	46
Colavita	10	0	0	0	50	70	50
Pompeian	17	0	0	0	65	100	94
Average, all brands	68 total	0	0	0	34	65	49

**Table 6.** Example of an adulterated olive oil purchased from a US supermarket in 2012 (%)

Parameter	Sample	IOC
Palmitic acid	6.8	7.5–20.0
Linolenic acid	5.0	$\leq 1.0$
Brassicasterol	6.3	$\leq 0.1$
Campesterol	23.2	$\leq 4.0$
$\Delta$ -7-Stigmastenol	0.3	$\leq 0.5$
Apparent $\beta$ -sitosterol	68.3	$\geq 93.0$
Diols	6.5	$\leq 4.5$
Total sterols (mg/kg)	4860.7	$\geq 1000$

Clearly, international standards do not allow for regional differences nor do they adequately identify cases of adulteration. Concerns by the IOC to maintain linolenic acid limits to  $<1.0\%$  are doing little to stop fraudulent practices in olive oil marketing. A variation of 0.5 to 1.5% linolenic acid has little significance in reducing fraud. An example (Table 6) of an adulterated oil purchased from a US supermarket had a linolenic acid content of 5.0% and campesterol of 23.2%. Examples such as this that exceed the standard are not rare. They show that ongoing discussions with the IOC and Codex about a standard of  $<1.0\%$  instead of the previous Codex standard of  $<1.5\%$  linolenic acid do little to prevent such

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### How standards evolve over time

The original US olive oil standards are a prime example of how standards evolve over time. One of the earliest standards, the United States Standards for Grades of Olive Oil, was established on March 22, 1948. The simplicity of this method shows the level of screening at that time. The grades included:

- US Grade A or US Fancy
- US Grade B or US Choice
- US Grade C or US Standard
- US Grade D or Substandard

Oil was graded on the basis of points for each of the following factors including:

- Free fatty acid content
- Absence of defects, such as cloudiness due to stearin and sediment in the oil.
- Odor
- Flavor

Oils had to exhibit "typical olive oil odor" that was free of the strong, green olive odor that today is considered to be an attribute of high-quality extra virgin olive oil. The oil also had to be free of any other off-odors. The descriptors for odor or flavor were as simple as:

- A—Good typical odor (flavor)
- B—Reasonably good typical odor (flavor)
- C—Fairly good typical odor (flavor)
- D—Substandard

The grade of oil was based on the total out of 100 points. Although these standards were an early attempt to ensure quality, they could not possibly address the issues of fraud and misconception the industry faces today.

fraud. The reduced allowable level of linolenic acid has simply made many genuine olive oils fall outside of the allowable limits.

### Future harmonization

To help dispel confusion caused by the availability of many existing methods, international organizations have worked to harmonize methodology. For the past 20 years, AOCS has republished IOC methods in the *Official Methods and Recommended Practices of the AOCS*. Also, ISO is gradually converting IOC methods to ISO method format that meets international standards for method validation. Despite these welcome changes, some problems remain with the way limits are set for olive oil. In particular, CAC has adopted the IOC/EC standards without consideration of other international requirements.

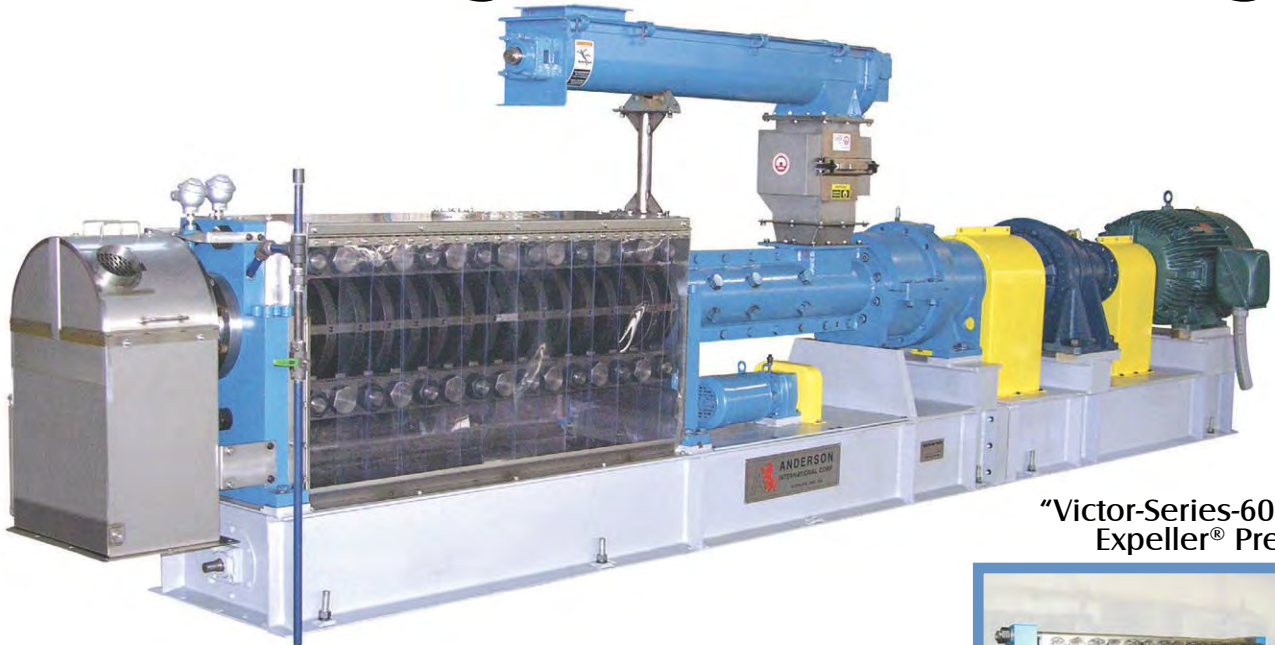
Codex Alimentarius is the logical international body to set standard limits for olive oil. Despite this, CAC's mirroring of IOC standards and refusal to recognize global differences has been disappointing, and its lack of leadership will likely lead to a continued divergence of national standards. If national standards continue to diverge, it points to deficiencies in existing international standards, in which case, the onus is on Codex Alimentarius to act.

*Rodney Mailer retired in October 2010 from his position as research fellow at the Australian Oils Research Laboratory in Wagga Wagga, New South Wales, Australia. He received the AOCS Timothy L. Mounts Award in 2008 and was named an International Fellow of the Royal Swedish Agricultural Society in 2012. He can be reached at [rod.mailer@australian-oils-research.com](mailto:rod.mailer@australian-oils-research.com).*



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