INFORM

International News on Fats, Oils, and Related Materials





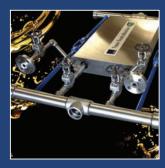
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6 Chia: Superfood or superfad?

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Recovering oil from spent bleaching earth

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- A rapid analytical method for bioactives in edible oils

 Researchers describe a new method for the rapid, simultaneous determination of tocopherols, carotenoids, and sterols in intact oils.
- Accelerated shelf life studies of extra virgin olive oils using the Oxitest method

A rapid analytical technique for examining fat oxidation in whole samples was recently used to analyze the oxidative stability of extra virgin olive oils coming from two regions of Italy.

New way to attach lipids to proteins could streamline drug development

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Chia: Superfood Laura Cassiday or superfad?

Chia, tiny seeds once known chiefly for their ability to sprout fuzzy green "hair" on terra cotta figurines, has now become one of the world's hottest superfoods. Many formulators are incorporating chia seeds into a variety of foods, such as breads, granola bars, breakfast cereals, yogurt, and smoothies. Indeed, the diminutive seeds pack a mighty nutritional punch, delivering surprisingly high levels of an omega-3 fatty acid, fiber, protein, vitamins, minerals, and antioxidants. And as a plant, chia may represent a more sustainable source of omega-3 fatty acids than seafood. Yet are the health claims made about chia supported by solid science, or is chia simply the latest in a long list of superfoods that promise benefits but fail to deliver?

- Chia is an increasingly popular food ingredient among consumers and manufacturers.
- The nutrient-rich seeds are good sources of α-linolenic acid (an omega-3 fatty acid), fiber, protein, vitamins, minerals, and antioxidants, prompting claims that chia is "superfood."
- Few clinical trials of chia supplementation have been conducted, and those that have are limited by small samples sizes, short durations, and inconsistent results.

ANCIENT GRAIN

Chia, or *Salvia hispanica*, is a flowering plant of the mint family, native to Central America. The plant produces oval-shaped seeds about 1 mm in diameter. Most chia seeds are gray with dark spots, although some are white in color (Fig. 1). *Salvia hispanica*, which grows best at tropical and subtropical latitudes, is now grown commercially in Mexico, Guatemala, Bolivia, Argentina, Ecuador, Nicaragua, and Australia.

Chia has been grown by native peoples in Central America for centuries. The plant was an important food crop for the Aztecs in pre-Columbian times (Valdivia-López, M.Á. and A. Tecante, http://dx.doi.org/10.1016/bs.afnr.2015.06.002, 2015). The Aztecs roasted and ground chia seeds for use in tortillas, tamales, and beverages. They also used the seeds for religious ceremonies and trade. During colonial times, chia consumption decreased, with the exception of a drink called "Agua de chia." When soaked in water, chia seeds absorb about 12–14 times their weight in liquid. The seed coat contains a polysaccharide that swells in contact with water, forming a gelatinous capsule around the seed that thickens chia beverages.

The US Food and Drug Administration (FDA) considers chia a food, not a food additive, making it exempt from regulation. "It appears that chia has been consumed by native cultures for long periods of time, and we are not aware of any safety concerns," the FDA wrote in a 2005 letter to chia researcher Wayne Coates, then at the University of Arizona (http://tinyurl.com/chia-FDA). In 2009, chia seed was approved by the European Union as a "Novel Food," allowing its incorporation into bread at up to 5%.

CHIA COMPOSITION

The word "chia" comes from the Nahuatl (or Aztec) word "chian," meaning "oily." Chia seed yields 25–40% extractable oil (Mohd Ali, N., et al., http://dx.doi.org/10.1155/2012/171956, 2012). The United States Department of Agriculture (USDA) has reported that chia seed con-





FIG. 1. A. Black and white chia (Salvia hispanica) seeds. Brown-colored seeds are likely immature. B. Magnified view of chia seeds.



omega-3 fatty acids docosahexaenoic acid (DHA) and eicosapentaenoic (EPA), which have been linked to various benefits, including cardiovascular and neurological health (Cassiday, L., http://tinyurl.com/fish-oil-Inform, 2016).

Chia contains a higher protein content than most grains and cereals (Valdivia-López, M. Á. and A. Tecante, http://dx. doi.org/10.1016/bs.afnr.2015.06.002, 2015). In addition, the protein is more complete in terms of amino acid content. However, chia cannot be used as a sole source of protein because the seed lacks sufficient lysine. Chia also contains more fiber than most other grains, with soluble and insoluble fiber in a ratio of about 1:5. The antioxidants in chia, mostly polyphenolic compounds such as isoflavones, inhibit lipid peroxidation in the seeds. On a per-gram basis, chia contains more ALA, fiber, protein, and calcium than either flax seed or salmon (Fig. 2).

The nutrient content of chia varies based on the region where it is grown and the growing conditions. Reported ranges of nutrient compositions include protein, 16–24%; total lipids, 26–34%; ALA, 57–65% of lipids; and fiber, 22–38% (Nieman, D.C., et al., http://dx.doi.org/10.1016/j.nutres.2009.05.011, 2009; Ayerza, R. and W. Coates, Poult. Sci., 2000; Ayerza, R., http://doi.org/10.5650/jos.58.347, 2009). "The nutrient content of chia will vary depending on the area where it was grown, the elevation, how much rain if got, when it got the rain, was it very hot or cooler, and the soil conditions," says Coates, now professor emeritus at the University of Arizona, in Tucson, Arizona, USA, and president of azCHIA in Sonoita, Arizona (www.azchia.com). "And that's true with any crop. There's nothing magical about chia."

According to Coates, he was one of the first researchers to study chia. In 1991, as an agricultural engineer with the University of Arizona, Coates was working on a project in northwestern Argentina. "We were working with a grower there trying to find alternative crops to beans and tobacco, the two main crops," says Coates. "We planted a bunch of different seeds to see what would grow there, and chia did pretty well. So we started looking into what it might be good for." Coates and his colleagues analyzed the composition of chia,

helped the Argentinian growers expand to commercial production, and examined possible health benefits of the seed.

At azCHIA, Coates sells whole and milled chia, both black and white seeds. Chia is typically a mixture of 95% black and 5% white seeds. "If you pick out the white seeds and plant them, you'll get white seeds. If you plant only black seeds, you'll get black seeds," says Coates. "But if you analyze the nutrient composition of black and white seeds, there's basically no difference." So seed color is primarily a matter of consumer preference, he says.

Chia seed maturity can also influence nutrient composition. For example, ALA increases 23% from the immature to the mature stage of the seed (Mohd Ali, N., et al., http://dx.doi.org/10.1155/2012/171956, 2012). "The mature seeds are white or black," says Coates. "You see pictures of chia on the web with a lot of brown seeds in it. Those are immature seeds, and they don't have the same composition." He also notes that many commercially available chia sources contain weed seeds and plant parts. "Everybody's jumping on the chia bandwagon," says Coates. "The problem is that there's a number of people selling very poor quality chia, and the public doesn't know good from bad."

THE QUESTION OF CULTIVARS

Through the centuries, humans have modified chia, like all crops, by selective breeding. While most commercially available chia is a mixture of different seeds, some companies offer seeds derived from a single cultivar, which they claim boosts the nutritional value and ensures consistency. Angelo Morini, founder and CEO of Anutra, LLC (www.shopanutra.com), developed the Anutra cultivar during a trip to a Mexican village called Acatic in 2002. "I found out that there are literally hundreds of different types of chia," says Morini. "I saw the opportunity to develop, through selective breeding, a new cultivar that maximized the omega-3, antioxidants, fiber, and protein. It didn't take us long because the people in Acatic had some real good seeds that they had been using through the years." Morini says that unlike other chia brands, Anutra has a standard of identity as a result of its consistent nutrient profile.

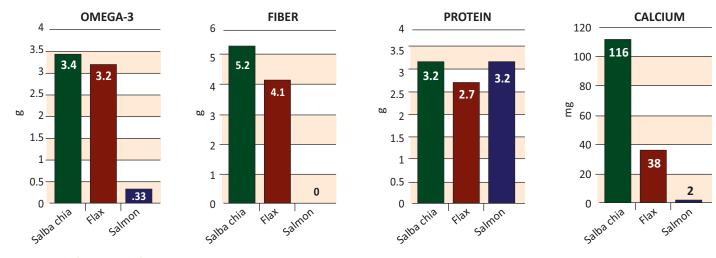


FIG. 2. Chia (Salba brand) has more omega-3 fatty acids, fiber, protein, and calcium than an equivalent amount of flax seed or Atlantic salmon.

"You'll get some people that will grow chia in the lowlands," says Morini. "They can call it chia, but the omega-3's very low, the protein's low, the nutrition, in general, is inferior. But they can still sell it as chia."

Another company, Salba Chia (Centennial, Colorado, USA), offers two white chia seed cultivars called Sahi Alba 911 and 912 (www.salbasmart.com). "Most chia you get at a store is grown as a wild crop, with anywhere from five to 40 different strains," says Hank Ralston, business development manager at Salba. "With that, the nutritionals can fluctuate about 30% left or right." According to Ralston, Salba's South American partners analyzed 80 different strains of chia to isolate two cultivars with higher amounts of ALA and protein. "Our cultivars are not only consistent from a nutritional side, but they also have consistent absorption of liquid, so that people don't have to reformulate finished products like breads each time they get a new batch of chia," says Ralston. Salba chia comes in three major forms: whole, milled, or sprouted seeds.

But not everyone is convinced that certain chia cultivars are better than others. "They're basically all the same thing," says Coates. "There's really only one chia, and that's the one we started with. The bigger effect can be the climate where it's grown."

FISH OIL SUBSTITUTE?

Epidemiological studies and some clinical trials have indicated a decreased risk of cardiovascular disease and inflammation with increased consumption of EPA and DHA in the form of oily fish or fish oil supplements (Cassiday, L., http://tinyurl.com/ fish-oil-Inform, 2016). Concerns about the sustainability and heavy metal contamination of seafood make plant-derived omega-3s an attractive possibility, especially for vegetarians and those who dislike or are allergic to fish. However, fish and fish oil provide EPA and DHA directly, whereas ALA in chia must be converted by the body into these longer-chained omega-3s.

In the United States, most adult males and females already consume sufficient amounts of ALA (2.1 g/day and 1.6 g/day, respectively) to satisfy adequate intake recommendations (1.6 g/day and 1.1 g/day ALA, respectively) (Nieman, D.C., et al., http://dx.doi.org/10.3390/nu7053666, 2015). After absorption from the gut, ALA has three major fates: 1) β-oxidation to produce energy in many cells and tissue types; 2) incorporation into cell membranes and storage pools, primarily in adipose tissue; and 3) conversion to longer-chain omega-3 fatty acids, including EPA and DHA, mainly in the liver (Baker, E.J., et al., http://dx.doi.org/10.1016/j.plipres.2016.07.002, 2016). β-oxidation accounts for approximately 15–35% of the ALA consumed. The conversion rate of ALA to EPA, and especially DHA, is low, with gender-related differences. Studies using radiolabeled ALA have provided estimates for conversion rates. In men, 0.3-8% and <1% of ingested ALA is converted to EPA and DHA, respectively. The conversion rates for women are significantly higher: up to 21% and 9% of ingested ALA is converted to EPA and DHA, respectively.

Most studies that have examined the effects of increased ALA consumption have reported higher levels of EPA in blood lipids and blood cells, whereas most report no signifi-



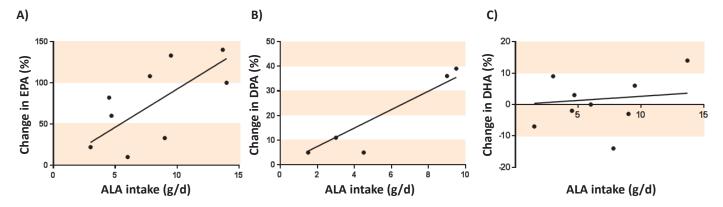


FIG. 3. Change in plasma phospholipid EPA (A) and DHA (B) as a result of increased ALA intake. Reprinted from Baker, E. J., et al., (2016) "Metabolism and functional effects of plant-derived omega-3 fatty acids in humans." *Prog. Lipid Res. 64*: 30–56, with permission from Elsevier.

cant increases in DHA content (Baker, E.J., et al., http://dx.doi. org/10.1016/j.plipres.2016.07.002, 2016). There is a positive linear relationship between ALA consumption and the amount of EPA in plasma phospholipids: For each 1 g increase in ALA, there is a 10% increase in EPA content (Fig. 3). In contrast, there is little or no change in DHA levels in plasma phospholipids with increasing ALA consumption.

"Chia could be a way of increasing the EPA content of blood and cells a little bit, but it would not be as effective as eating more oily fish or taking a fish oil supplement," says Philip Calder, professor of nutritional immunology at the University of Southampton, in the United Kingdom "Where studies have compared ALA with EPA plus DHA for functional effects, ALA is always much weaker, on a per-gram basis. As a generalization, I would say ALA is about one-tenth as potent as EPA on a per-gram basis."

Omega-3 and omega-6 fatty acids compete for the same enzymatic pathway to produce longer-chain fatty acids. For much of human evolution, the dietary ratio of omega-6 to omega-3 fatty acids was about 1:1. However, modern Western diets have omega-6:omega-3 ratios ranging from 10–25:1, driving preferential elongation of omega-6s over omega-3s. Thus, the conversion of ALA to EPA and DHA might be enhanced if LA intake is decreased at the same time that ALA consumption is increased (Baker, E.J., et al., http://dx.doi.org/10.1016/j.plipres.2016.07.002, 2016). Chia has an omega-6:omega-3 ratio of approximately 1:3.

Although ALA from chia is unlikely to provide abundant amounts of EPA and DHA, it remains possible that ALA could have its own health effects, independent from the longer-chain fatty acids. However, clinical studies of the effects of ALA-enriched diets on cardiovascular disease and inflammation have produced inconsistent results (Baker, E.J., et al., http://dx.doi.org/10.1016/j.plipres.2016.07.002, 2016).

HEALTH OR HYPE

Because chia's commercial use as a food crop is relatively recent, the seeds have not been widely studied in the clinic, and existing research on chia's health benefits is sparse and inconclusive. Animal studies have shown promise. Feeding

rats chia seed improved their serum lipid profile, lowering triglycerides and low-density lipoprotein (LDL) cholesterol while raising high-density lipoprotein (HDL) cholesterol (Ayerza, R. and W. Coates, http://dx.doi.org/10.1159/000100818, 2007).

Researchers have hypothesized that the high fiber content of chia could improve satiety and thus promote weight loss. The insoluble fiber in chia has a high water-holding capacity, which could be expected to induce a sense of fullness. Nieman *et al.* assessed the effectiveness of chia seed in promoting weight loss and altering disease risk factors in 76 overweight or obese men and women (http://dx.doi.org/10.1016/j.nutres.2009.05.011, 2009). The study participants ingested 25 g whole chia seed or a placebo mixed in 250 mL water, twice a day, for 12 weeks. This amount of chia provided 19 g dietary fiber and 8.8 g ALA per day. Other than drinking the beverage, the subjects made no special attempts to lose weight.

After the 12-week study, the participants in the chia group had a 24.4% increase in plasma ALA levels from baseline, but no changes in plasma EPA or DHA. Also, no changes in body mass or composition of the participants were observed, suggesting that, in the absence of other lifestyle interventions, chia does not promote weight loss. The researchers likewise did not detect any changes in disease risk factors such as systolic blood pressure, serum lipoproteins, serum glucose, C-reactive protein (CRP), or inflammatory cytokines.

In another study, Nieman *et al.* examined the effects of chia seed oil on human running performance (http://dx.doi. org/10.3390/nu7053666, 2015). During prolonged, intensive exercise, ALA is strongly mobilized from adipose tissue, increasing almost six-fold in the plasma. The researchers hypothesized that ALA may serve as a fuel source during prolonged exercise, especially after carbohydrate stores are depleted. Chia, a rich source of ALA, could enhance exercise performance and reduce post-exercise inflammation. To test this hypothesis, 24 runners who had fasted overnight were instructed to drink 500 mL flavored water, with or without 7 kcal/kg chia seed oil. This dose of chia oil provided 0.43 g ALA per kg body weight, or about 31 g ALA (a large dose) for the average subject. Thirty minutes later, the participants ran on a treadmill to exhaustion.

Although plasma ALA levels were elevated 337% compared to baseline in the chia group, the run time to exhaustion did not differ between the chia and water groups. In addition, no differences were observed in the respiratory exchange ratio (a measure of fatty acid oxidation), plasma glucose, oxygen consumption, blood lactate, or ratings of perceived exertion between the two groups. Cortisone and inflammatory markers increased with exercise to a similar extent in both groups. Because anti-inflammatory effects of ALA may depend on its conversion to EPA, which takes at least one week, further research is needed to determine whether chronic ALA supplementation can reduce post-exercise inflammation.

"Chia seeds are a good nutrient source, but definitely overhyped by companies and distributors," says David Nieman, professor and director of the Human Performance Laboratory at Appalachian State University, North Carolina Research Campus in Kannapolis, USA. "We have conducted several randomized trials, and have found no benefits on weight loss, disease risk factors, or athletic performance."

However, some studies have indicated health benefits of chia. Vuksan *et al.* investigated the effects of supplementing conventional therapy for type 2 diabetes with chia (http://dx.doi.org/10.2337/dc07-1144, 2007). People with type 2 diabetes have an elevated risk for cardiovascular disease, and new treatments are needed to complement existing therapies. Twenty subjects with well-controlled type 2 diabetes consumed either 37 g/day Salba chia or wheat bran for 12 weeks.

Plasma levels of both ALA and EPA increased two-fold in the group consuming chia. The researchers found that the Salba chia mitigated three risk factors for cardiovascular disease: Systolic blood pressure was reduced by 6.3 mmHg, high-sensitivity CRP (a marker of inflammation) decreased by 40%, and von Willebrand factor (a prothrombotic glycoprotein) was reduced by 21% in the chia group. No changes in body weight, blood lipids (triglycerides, LDL cholesterol, HDL cholesterol), fasting plasma glucose, or hemoglobin A1c were observed.

In another study, Vuksan *et al.* assessed whether Salba chia could reduce blood sugar spikes following meals (known as postprandial glycemia) in healthy subjects (http://dx.doi. org/10.1038/ejcn.2009.159, 2010). A reduction in postprandial glycemia could help explain the improvement in the three cardiovascular risk factors that the researchers had observed in their earlier study of people with type 2 diabetes. Eleven healthy individuals consumed white bread containing 0, 7, 15, or 24 g of ground Salba chia. The researchers collected blood samples and appetite ratings 2 h after consumption.

Post-prandial glycemia was reduced in a dose-dependent manner. On average, each gram of Salba chia baked into white bread decreased post-prandial glycemia by 2% compared with white bread lacking chia. In addition, appetite ratings for all doses of chia were decreased 2 h after consumption, suggesting an effect on satiety.

Vuksan claims that the results from his studies on Salba chia cannot be extended to regular chia. "People make incredible profits on chia because they sell it based on our research," he says. "Unlike regular chia, Salba has a very, very consistent

composition. The composition of Salba that we analyzed in 2000 closely resembles that of Salba in our most recent study, more than 10 years later." According to Vuksan's experience and others' data reported in the literature, the composition of regular chia is much more variable, he says.

However, some researchers, such as Nieman, do not believe that large discrepancies in chia clinical trial results can be explained by the chia source. "All types of chia seeds are concentrated sources of ALA, soluble fiber, and minerals," says Nieman. "There is no meaningful difference between types of chia seeds that would 'magically' make one seed (within the context of a normal diet) induce weight loss or alter disease risk factors."

FORMULATING WITH CHIA

Whether or not clinical data currently support the superfood status of chia, consumer interest in the trendy seed is unlikely to wane any time soon. Therefore, many formulators are incorporating chia into foods, including breads and other baked goods, snack foods, and beverages.

Chia may be an attractive option for food manufacturers who want to make omega-3 claims. As of January 1, 2016, the FDA prohibited food labels from claiming that products are "high in," "rich in," or "excellent sources of" EPA or DHA because no reference values for the nutrients have been established (http://tinyurl.com/FDA-omega3-claims). In contrast, the FDA has taken no action against ALA claims of "high

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in," "good source of," and "more" because ALA is an essential fatty acid with a recommended intake.

From a formulator's perspective, chia has some attractive properties compared with flax seed, another plant-based source of ALA. Because of its thinner seed coat, whole chia is easily digested by the human body, whereas flax seed must be ground to enable digestion and absorption of nutrients. However, flax seed lacks the antioxidants of chia, making it prone to lipid oxidation and reduced shelf life. In contrast, chia is very stable. According to Ralston, sensory tests have indicated that whole chia seeds are stable for 5 years from the date of harvest. "When you mill or sprout chia, or press it to produce an oil, it's stable for about 18 months before you start to see a degradation in quality," he says.

Several methods exist to extract oil from chia seed, including compression, solvent, and supercritical fluid extraction. The latter method typically yields the highest purity and ALA content (Mohd Ali, N., et al., http://dx.doi.org/10.1155/2012/171956, 2012). Chia oil is not used in foods as much as whole or milled chia, primarily because oil production is an inefficient process, with over 80% waste. In addition, chia oil does not offer the fiber or protein content of whole or milled chia.

Andrew Stewart, a formulator and managing partner at Toronto-based Amazing Grains, LLC, incorporates Salba chia into all of his products. "Amazing Grains is a blend of sprouted whole grains with super seeds and fruits and vegetables," he says. "It was the Salba chia that won us the 'Startup Ingredient of the Year' award presented by Nutraingredients.com in Geneva in May." Like Vuksan, Stewart says he will only work with Salba chia because of its consistency.

"I find that the addition of Salba chia into a variety of products has the ability to lower the glycemic index," Stewart says. He has also developed a fat-replacement system by emulsifying any edible oil, water, and Salba chia. "I've been able to bring down the fat content in a wide variety of products," says Stewart.

"Some people want to include chia in their products, but they don't really add enough to make a difference," says Stewart. "I include Salba chia in my formulations at anywhere from 5 to 20 percent. Twenty percent goes into cereals." When water or milk is stirred into the cereal, the Salba gels up, creating a thick porridge-like texture, he says.

The unique texture of hydrated chia can allow the partial replacement of eggs in baked goods. "To replace eggs with our Salba seed, we say to use a 4:1 ratio of either the milled product or the whole seed to water," says Ralston. "A lot of vegans and people with other dietary restrictions are coming up with some really creative recipes using Salba."

Although the mucilaginous texture of hydrated chia seeds works well for some applications, many consumers find the mixture, which resembles tapioca pudding, unappetizing. As a result, Morini developed a milling process that mitigates the slimy texture of chia seeds. The patented process combines mixing, milling, heating, and drying to produce extremely fine chia particles (80–90 microns). When mixed with water, the chai particles revert to a submicron size.

According to Morini, the microfine Anutra chia is much easier to work with than standard whole or milled chia. Because of

the high content of lipids and insoluble fiber in regular chia, the ingredient can be difficult to solubilize in products like juices. "We developed a product that you can put in hot water, cold water, oils, dairy products, and it works beautifully. It stays in suspension," says Morini. He says that he is currently working with different food companies to fortify their products with the microfine Anutra. For example, adding 1 g of the product to chocolate milk would allow a "good source of omega-3" claim, and 2 g would allow an "excellent source" claim. "They can put our product into chocolate milk, and it stays in suspension," says Morini. "And suddenly they don't need to use all these thickeners like carrageenan, locust bean gum, or guar gum."

SUPPLY AND DEMAND

Many food manufacturers who are hesitating to take the plunge into chia are worried about potential supply problems. "Their concern is, is there going to be enough production to sustain their product line?" says Coates. "With new crops, it's always the chicken and egg thing. The producers say, "If we produce it, who's going to buy it?', and the big companies say, 'If we buy it, are we going to be guaranteed a supply?'" Like all crops new to the world market, the price of chia can fluctuate widely based on supply and demand. "You can never really find out who's producing it and how much and what's in storage right now," says Coates.

According to Stewart, because Salba chia is a specific cultivar produced by a single company, it is not subject to the price fluctuations of regular chia. "Right now there is a shortage of regular chia, and of course, regular chia prices bounce up and down guite a bit based on supply and demand," he says. "One of the great things about Salba is that its price hasn't changed in the last 10 years I've been working with it."

Stewart notes that some large food companies balk at incorporating chia into their products because it is more expensive than standard ingredients such as flour, sugar, and eggs. "A lot of big manufacturers are deathly afraid to pass along the price increase for healthier products to the consumer," he says. "But it's been proven that people will pay more for healthier choices."

And at least for know, chia is perceived by consumers as a healthier choice than most other whole or refined grains. Whether the nutrient-dense seed lives up to the superfood hype will depend on results from larger-scale and longer-term clinical trials of chia's suspected health benefits.

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



Recovering oil Albert J. Dijkstra from spent bleaching earth

- One way to reduce the cost of bleaching edible oils is to use less bleaching earth. This can be accomplished if before the spent earth is discarded, it is brought into contact with unbleached oil (high-adsorbate concentration) rather than with spent bleaching earth.
- On the other hand, spent bleaching earth contains not only extractable oil, but also non-extractable organics that result from oil-degradation reactions catalyzed by the bleaching earth. Consequently, slurrying spent earth in triglyceride oil and pumping the slurry to the extractor lengthens the amount of time in which degradation reactions can occur and non-extractable organics can form.
- Recent investigations demonstrated that in spite of increased degradation losses, sending a slurry of spent bleaching earth to the extractor saves on oil and increases profitability in other ways as well.

The variable cost of bleaching edible oils consists of three main elements: 1. the cost of the bleaching earth itself, 2. the cost of oil lost in the spent earth, and 3. the cost of disposing the spent earth. As can be expected, attempts to reduce the cost of the bleaching process have focused on all three cost elements of the process [1].

One development that allows less bleaching earth to be used makes use of adsorption isotherms (Freundlich, Langmuir) which illustrate that at adsorption equilibrium, the adsorbate loading onto the adsorbent increases when the adsorbate concentration in the oil increases. In other words, before the spent earth is discarded, it should be brought into contact with unbleached oil (high adsorbate concentration) rather than with bleached oil. This is achieved in the staggered filtration system invented by W.R. Grace [2] and in the Öhmibleach process [3].

The amount of oil lost in spent earth can be reduced by blowing the filter cake with steam before opening the filter and discarding the cake. Proper blowing reduces the residual oil content to about 25 weight % of the spent earth, but the spent earth also contains non-extractable organics. These are degradation products of unsaturated triglycerides, the formation of which is catalyzed by the acid sites on the activated earth. Their formation is exothermic and may cause spent earth to start smouldering. Another process to reduce the amount of oil lost with the spent earth involves extracting the spent earth with a solvent such as hexane, and thereby recovering the oil.

Landfill disposal of spent earth can be a cost factor. This may involve no more than transport costs if the spent earth is used in brick-making or fed to a biogas fermenter, but it is of course much more attractive to sell the spent earth. For instance, if it is mixed into the meal, it can be sold at meal prices. However, remember the bovine spongiform encephalopathy (mad cow disease) epidemic in the United Kingdom? It resulted from feeding cows with insufficiently heated meat and bone meal from sheep with scrapie that were processed at the same slaughterhouse. Since then, a regulation came

into force stipulating that by-products may only be mixed into meal of the same agricultural origin. You are allowed to mix the filter cake resulting from winterizing sunflower seed oil into sunflower seed meal but not into soybean meal. Moreover, you must mix this cake into said meal at the same production site—a practice that favors integrated oil mill/refineries.

So what is the best way to deal with spent bleaching earth for a vertically integrated oil mill/refinery? On paper, the process in which the spent earth is sent to the extractor looks best because:

- it recuperates the extractable oil from the spent earth, which oil is then sold at oil price;
- it avoids the necessity to blow the filter cake to minimum residual oil level, simplifies the filter cake treatment and thus saves money;
- it allows the extraction residue (varnished clay) to be sold at meal value, which value is higher than what was paid for the bleaching earth; and
- in comparison with mixing the spent earth into the meal, its prior extraction has the advantage that it introduces less non-proteinaceous material into the meal.
 So, if there happens to be some leeway in meeting the

minimum protein content specification, prior extraction may permit some hulls or even water to be added to the meal to be sold at meal value.

To realize these savings, some investment is required to enable the filter cake to be transferred to the extractor. This investment would comprise a slurry vessel fitted with an agitator into which the filter could be emptied, a supply of crude oil to convert the cake into a slurry that can be transferred by pump, and a buffer vessel from which the slurry is pumped into the extractor. This investment is small in comparison with the potential savings listed above, so what stops refiners from adopting this profitable process?

Well, there is an unanswered question. Just imagine that the acid-activated bleaching earth particles went on catalyzing the conversion of extractable oil into non-extractable organics while kept in the slurry vessel and the intermediate storage vessel. Slurrying the cake with oil provides the reagent, and storage provides a reaction time that is much longer than the contact time between earth and oil during the actual bleaching process. To answer this question, a simple experiment was conducted in a vertically integrated soybean oil mill *cum* refinery operating this process (Fig. 1). A sample of spent bleaching

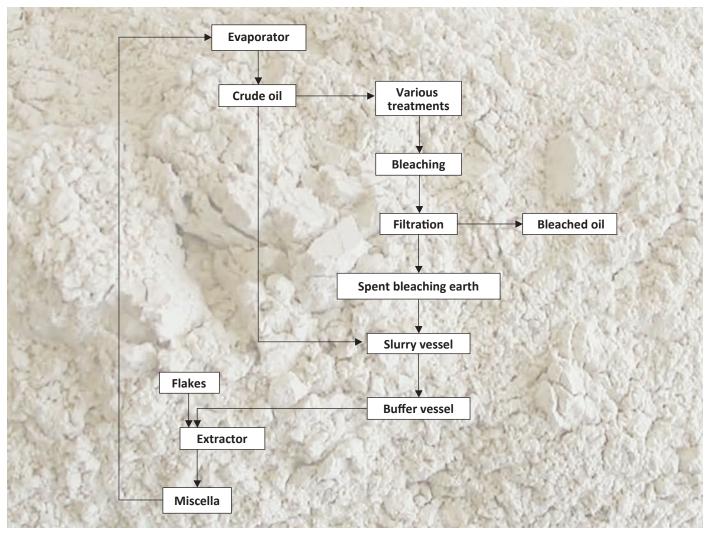


FIG. 1. Schematic for a vertically integrated oil mill/refinery in which the spent earth is sent to the extractor.

earth was taken when the filter was discharged into the slurry vessel. One aliquot of this sample was immediately extracted with hexane in a Soxtec Avanti automatic Sohxlet apparatus, which also provides a drying cycle. The weight loss on hexane extraction was 38.7%; this illustrates that the cake had not been blown exhaustively. The dried sample was then placed in a muffle furnace and heated at 600°C for four hours. This treatment caused all water present to be evaporated and all organics that had not been extracted by hexane to be burnt off, resulting in a weight loss of the dried sample of 18.6%; when the measurements were repeated with another sample, a value of 18.4% was found.

To mimic what happens in the plant, another aliquot of the sample bleaching earth was slurried with crude soybean oil in a ratio of 2:3 and stored at 52°C for a period of 12 hours before it was analyzed. It turned out to be difficult to extract this slurry in the Soxtec Avanti apparatus, so the slurry was filtered first, and the filter cake was then extracted in the Soxhlet apparatus. The dried sample obtained was treated in the muffle furnace and, again, its weight loss was determined. If there had been no additional degradation catalyzed by bleaching earth, this loss should have been 18.4—18.6%, as it was in the evaporated samples. Oil degradation increases the weight loss on ashing, and if this weight loss had reached some 40% (calculated on the hexane-extracted sample) this would have indicated that more oil had been degraded than would have been present in an exhaustively blown filter cake. That would, of course, have defeated the purpose of the whole exercise.

Fortunately, the weight losses for the duplicate determinations were 20.2% and 21.6%, respectively, leading to additional degradation product formation of 1.6% and 3.4%, respectively. So, while a significant (p < 0.004) increase in the amount of degradation products was established, it was so

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small that it would hardly affect the profitability of the process in which non-exhaustively blown filter cake is slurried in crude oil and the slurry is fed to an extractor, where the oil in the spent earth is recovered.

Refiners in vertically integrated oil mills who recover oil from spent bleaching earth by slurrying the earth in oil and pumping the slurry to the extractor can therefore heave a sigh of relief. Refiners who dispose of their spent earth in another way should compare this way with the above spent-clay extraction. If they conclude that their method is more profitable, I would like to hear from them.

Albert Dijkstra started his career by working as chief chemist in the chemical industry. In 1978 he switched to edible oils and fat and became R&D director of the Vandemoortele Group in Belgium. Since 1997 he enjoys working from his home in S-W France as consultant, author, inventor, reviewer, and editor. He has won several awards and is a Fellow of the AOCS. He can be contacted at albert@dijkstra-tucker.be.

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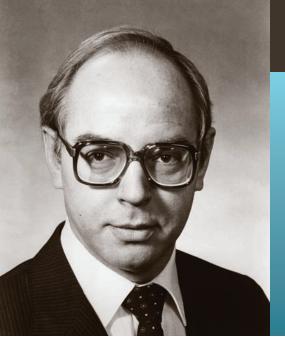
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Gut-brain endocannabinoid Nicholas V. DiPatrizio signaling as a fatty acid sensor

- Our bodies generate cannabis-like lipid signaling molecules called endocannabinoids, which promote the seeking, sensing, and storage of highenergy nutrients for future metabolic use.
- Tasting specific dietary fatty acids generates endocannabinoids in the gut, which are thought to promote positive feedback to the brain and the consumption of fatty foods.
- Overactive gut-brain endocannabinoid signaling may promote overeating of energy-rich foods and contribute to obesity, type-2 diabetes, and cardiovascular disease.

Consumption of dietary fats is critical for survival. Essential fatty acids found in our diet provide the building blocks of a variety of molecules that regulate cellular function and communication, including the eicosanoid signaling molecules, the endocannabinoids. Indeed, the endocannabinoid system is an important regulator of feeding, energy balance, and reward. This endogenous lipid signaling system is located throughout all organs in mammals (and possibly other animals), and is comprised of the endocannabinoids, their receptors (cannabinoid CB₁ and CB₂ receptors), and enzymes that synthesize and break them down (see Fig. 1). In general, activation of the endocannabinoid system increases food intake, while inhibiting its activity reduces food intake and body weight. Recent evidence from our group suggests that the endocannabinoid system may serve as a fatty acid sensor, which drives us to consume foods containing high levels of dietary fats [1].

DIETARY FATS

Considerable debate has been focused on the question whether fat taste should be considered a sixth primary taste quality along with sweet, sour, bitter, salty and umami. In 2015, fat made it to the ranks as a taste primary, and was given the name "oleogustus" (Latin for *fat taste*) by the taste researcher Richard D. Mattes, Distinguished Professor at Purdue University in Lafayette, Indiana, USA [2].

Dietary fats are detected in the oral cavity by receptors (e.g., CD36) that participate in transducing the presence of fat in the mouth into electrical signals carried to the brain by several cranial nerves [3].

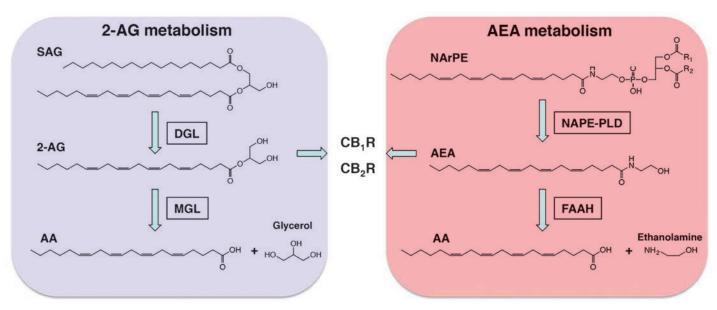


FIG. 1. The two primary endocannabinoids have distinct biosynthetic and degradative pathways. Biosynthesis of the monoacylglycerol, 2-arachidonoyl-sn-glycerol (2-AG), is mediated by diacylglycerol lipase (DGL) dependent hydrolysis of 1,stearoyl,2-arachidonoyl-snglycerol (SAG). 2-AG is degraded by monoacylglycerol lipase (MGL), resulting in arachidonic acid (AA) and glycerol. Biosynthesis of the fatty acid ethanolamide, anandamide (AEA), is mediated by N-acylphosphatidylethanolamide phospholipase D (NAPE-PLD) dependent hydrolysis of N-arachidonoylphosphatidylethanolamide (NArPE). AEA is degraded by fatty acid amide hydrolase (FAAH), resulting in AA and ethanolamine. Both, 2-AG and AEA activate cannabinoid type 1 (CB1R) and cannabinoid type 2 (CB2R) receptors on the surface of cells throughout the body. Alternative endocannabinoid metabolic pathways have been suggested; however, the most well-established are described.

Mounting scientific evidence suggests that fat receptors are essential for our attraction to fatty foods. Indeed, preference for fatty foods is missing in mutant mice that lack receptors thought to be responsible for sensing fats.

Fatty foods contain several distinct fats in the form of triacylglycerols but very low levels of free fatty acids. An increasing number of studies indicate, however, that free fatty acids are required for fat taste, and triacylglycerols are not. For example, lipases generated by glands in the mouth hydrolyze triacylglycerols and release free fatty acids at concentrations that activate fat receptors. Furthermore, pharmacological inhibition of oral lipase activity in humans and rodents diminishes their ability to reliably detect dietary fats [3].

ENDOCANNABINOIDS IN THE GUT CONTROL THE INTAKE OF FATTY FOODS

Biochemical, molecular, and behavioral evidence published by our research group suggests that gut-brain endocannabinoid signaling controls dietary fat intake. To study the role of fat taste in these processes, we used a rodent sham feeding model that isolated oral exposure of foods from any post-ingestive influence [3]. Tasting dietary fats (corn oil emulsion)—but not carbohydrate (sucrose) or protein—initiated production of the endocannabinoids 2-arachidonoyl-sn-glycerol and anandamide, in the rat small intestine [4]. This result was specific to the small intestine, because tasting corn oil failed to affect endocannabinoid levels in other organs. Importantly, inhibiting endocannabinoid signaling at cannabinoid CB₁ receptors in the small intestine with peripherally restricted CB₁R antagonists that do not enter the brain blocked sham feeding of corn oil.

Moreover, surgical disruption of the vagus nerve—which communicates neurotransmission between the brain and gut blocked increases in intestinal endocannabinoid levels after fat exposure, indicating that brain-gut signaling is required for endocannabinoid activity in the gut. Together, the results suggest that tasting dietary fats leads to production of the endocannabinoids in the gut, which, in turn, sends signals back to the brain and promotes intake of foods rich in fats (see Fig. 2, page 20).

SPECIFIC COMPONENTS OF DIETARY FAT CONTROL ENDOCANNABINOID SIGNALING IN THE GUT

Our diet contains a wide variety of lipids that include saturated, monounsaturated, and polyunsaturated fats. We next asked if texture (e.g., creaminess sensation) or specific dietary fatty acids contained in corn oil induces gut-brain endocannabinoid signaling and preference for foods containing fats [5]. To address this question, separate groups of rats were sham fed emulsions containing corn oil, mineral oil (which shares similar textural properties to corn oil), or mineral oil with the addition of saturated (stearic acid), monounsaturated (oleic acid), diunsaturated (linoleic acid), or polyunsaturated (linolenic acid) free fatty acids.

In contrast to corn oil, tasting mineral oil alone failed to increase endocannabinoid levels in the epithelium of the small intestine. Similar to corn oil, however, tasting mineral oil that contained oleic and linoleic acid drove robust increases in endocannabinoid levels, but stearic or linolenic acid failed to elicit a similar response. Furthermore, when given a choice

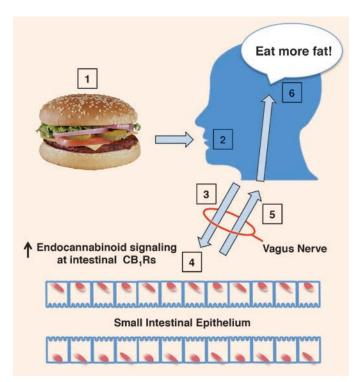


FIG. 2. Tasting dietary fats drives gut-brain endocannabinoid signaling and further feeding. Fatty foods (1) enter the mouth where their triglyceride content is hydrolyzed by lipases, and free fatty acids are released. Free fatty acids are detected by receptors located in the oral cavity (2), which in turn, activate nerves that carry electrical signals to the brain for processing. The brain communicates bi-directionally with peripheral organs via the vagus nerve. Recent evidence suggests that the presence of fats in the oral cavity increases neurotransmission carried by the vagus nerve to the gut (3) and stimulates an upregulation of endocannabinoid levels in small intestinal epithelium of rodents (4). Endocannabinoid signaling at local cannabinoid CB1Rs is thought to modify vagal signaling back to the brain (5) and promote the further consumption of fatty foods (6).

between tasting emulsions contained mineral oil alone or those also containing linoleic acid, rats exhibited strong preferences for linoleic acid. Notably, pharmacological blockade of peripheral CB $_1$ Rs inhibited preference for linoleic acid. Collectively, these studies reveal that tasting specific unsaturated dietary fats drives endocannabinoid signaling in the small intestine of rats, and this signaling at local CB $_1$ Rs controls preferences for foods containing linoleic acid.

TARGETING PERIPHERAL ENDOCANNABINOID SIGNALING PATHWAYS FOR THERAPEUTIC GAIN

In summary, we propose that gut-brain endocannabinoid signaling serves a critical role in promoting the intake of energy-rich foods for survival. Overactive gut-brain endocannabinoid activity, however, may promote overeating of energy-rich foods and contribute to obesity, type-2 diabetes, and cardiovascular disease.

Pharmacological inhibitors of CB_1Rs show clinical promise for the treatment of obesity and related metabolic disorders.

Unfortunately, CB₁R antagonists that cross into the brain lead to serious psychiatric side effects (e.g., depression).

Recent studies discussed above from our group and others suggest that targeting CB_1Rs in peripheral organs with specially designed CB_1R inhibitors that do not reach the brain also reduces feeding and body weight similarly to inhibitors that do reach the brain. Thus, targeting peripheral CB_1Rs for therapeutic gain is attractive due to reduced risks of side effects when compared to brain-penetrant cannabinoid receptor ligands. Further development and design of peripherally-restricted CB_1R ligands will be instrumental for the safe treatment of metabolic syndrome and possibly eating disorders (e.g., binge eating disorder).

Nicholas DiPatrizio's passion for scientific discovery was solidified during his undergraduate research studies at Temple University in Philadelphia, USA, where he studied the role for cannabinoid signaling pathways in feeding behaviors. He subsequently earned a Biomedical Ph.D. in the Neurosciences at Drexel University College of Medicine, and completed his post-



doctoral fellowship in the laboratory of the lipid biochemist, Daniele Piomelli, at the University of California Irvine School of Medicine. DiPatrizio is a recipient of the National Institutes of Health Pathway to Independence K99/R00 grant award from the National Institute on Drug Abuse for his work examining the role for endocannabinoids in mediating dietary fat intake. In 2015, he launched his independent research program as an assistant professor of biomedical sciences at the University of California's newest medical school located in Riverside, California, USA. His research program investigates the neural and molecular mechanisms that regulate sensory processing, food reward, and energy balance in health and disease.

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A rapid analytical method for bioactives in edible oils

Clare L. Flakelar and Paul D. Prenzler

- The growing popularity of cold-pressed and minimally refined oils and the adoption of softer processing technologies that preserve healthful bioactive compounds have increased the need to develop efficient analytical methods for measuring these compounds in edible oils.
- A new method using high-performance liquid chromatography—diode array/mass spectrometric detection (HPLC-DAD/MS) was recently developed to enable the rapid, simultaneous determination of tocopherols, carotenoids, and sterols in intact oils.
- The method eliminates the need for a lengthy hydrolysis step when determining sterol compounds and achieves, in a single 30-minute analysis, results comparable to those obtained from three separate analyses lasting several hours.

For more than 50 years, the edible canola oil market has been driven by yield and shelflife. To achieve yield and shelf-life targets and satisfy consumer preferences for transparency and neutral flavor, the majority of the oil is heavily processed to remove impurities. During this processing, many important bioactive compounds in the oil are diminished, or lost entirely. More recently, shifting consumer preference toward products that are more natural and less-refined has prompted investigation into processing modifications that improve the retention of bioactives, and ways to obtain seed stocks with enhanced levels of these healthful compounds.

Relative to other fat sources, canola oil has a desirable fatty acid composition: It is both high in unsaturated fats and low in saturated fats, with an omega-3:omega-6 ratio of 2:1. In its crude form, canola oil possesses several minor components that offer considerable health benefits. Sterols, tocopherols, and carotenoids are all classes of fat-soluble minor compounds found in unrefined canola oil. Sterols, otherwise known as phytosterols in plants, have been shown to actively reduce low-density-lipoprotein (LDL) cholesterol levels and to possess disease-fighting properties. Tocopherols, which are collectively grouped with tocotrienols to form the Vitamin E compounds,



FIG. 1. HPLC-DAD-MS system as described

are most commonly known for their antioxidant behavior. Carotenoids, which primarily exist in unrefined canola oil as lutein (90%) and β-carotene (up to 10%), exhibit powerful protective properties for skin and eyes. Once consumed, β-carotene is converted to retinol (Vitamin A), whereas lutein accumulates in the eye, where it protects the macula from light damage. Consequently, there is increased interest in monitoring these compounds in all food sources, including edible oils.

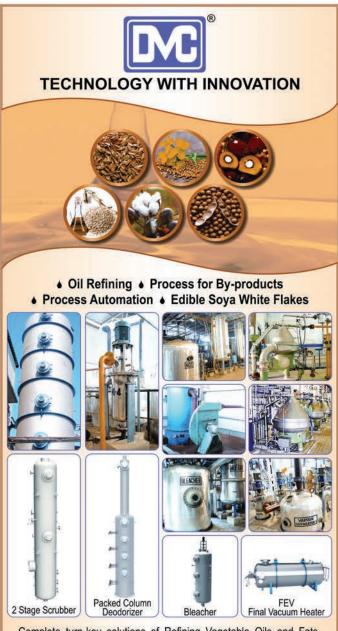
The emergence of minimally refined oils and adoption of "softer" processing techniques make it possible to retain healthful compounds in the oil and to market edible oils based on their nutrient content. Meanwhile, the bioactives in edible oil could potentially be increased by breeding new canola lines with enhanced levels of specific compounds in the seed, as was shown to be feasible in a previous study [1]. Such efforts would obviously increase the need to measure bioactives in edible oils, as well as to develop rapid and efficient analytical methods for determining such compounds.

Standard techniques exist for the quantification of sterols and tocopherols in vegetable oils via gas chromatography (GC) (AOCS Official Method Ch 6-91) and normal-phase liquid chromatography (LC) (AOCS Ce 8-89), respectively. However, there is no AOCS method for the analysis of carotenoids in canola oil, because AOCS does not develop methods for pigments. Carotenoids are typically determined using LC with detection at 450 nm. The requirements for determining these compounds using current methods involve laborious sample preparation (e.g., hydrolysis of sterol esters), multiple analytical instruments, and the preparation of multiple samples for each oil due to incompatibilities among compound classes. For example, because carotenoids are susceptible to light and heat degradation, they cannot be determined with sterols, as the samples used to analyze sterols must be heated for hydrolysis. Given the interest in bioactive compounds and difficulties

associated with current methods, our research group set out to develop a technique for analyzing intact oils that involves minimal sample preparation, a single instrumental technique, and the simultaneous and selective detection of compounds in all three classes.

We recently reported the development of such a method [2] based on normal phase HPLC with both mass spectrometric (MS) and diode array detection (DAD). Since this method uses normal phase HPLC, the oil sample is simply diluted in hexane and then injected directly into the chromatograph, minimizing sample preparation (Fig. 1). DAD detection is used to quantify tocopherols (294 nm) and carotenoid compounds (453 nm), consistent with previously established methods. Sterols are quantified by MS only, since they are not readily detected by UV absorbance when using *n*-hexane as a solvent.

A key feature of this new method is the use of multiple reaction monitoring (MRM) on the MS, which results in superior selectivity due to the monitoring of specific target ions based on the known fragmentation behavior of each compound. All analytes of interest can be detected using the MS, although carotenoids were not abundant enough in the canola oil for MS quantification. In contrast, when the method was



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TABLE 1. Bioactive compounds analyzed using the HPLC-DAD/ MS method

Compound	Compound class	Detector used for quantification
lutein	carotenoid	DAD/MS*
β-carotene	carotenoid	DAD/MS*
α-tocopherol	tocopherol	DAD/MS
γ-tocopherol	tocopherol	DAD/MS
δ-tocopherol	tocopherol	DAD/MS
β-sitosterol	sterol	MS
campesterol	sterol	MS
brassicasterol	sterol	MS
cholesterol	internal standard	MS

*MS detection only possible for oils with high concentration of carotenoids (12 and 80 $\mu g/mL$ for β -carotene and lutein, respectively).

applied to other types of vegetable oil, the carotenoids in palm fruit oil were found to be well above the limit of quantification (LOQ) for MS detection, thus illustrating the potential of MS quantification for analyzing samples with higher concentrations of carotenoids. Table 1 defines the compounds and detection technique used for quantification.

When monitoring individual ion transitions on the MS, we detected two peaks for each sterol compound, one eluting early and one later. These peaks correspond to the sterol esters which co-eluted with the lipid structures, and the "free" sterols. This retention order is expected, since free sterols are more polar (due to the absence of a fatty acid group), and consequently retained on the column longer. This separation provides the analyst with the ability to distinguish between free and esterified sterols, providing even more detailed information on the oil composition. Further testing was conducted to see if co-elution was having an effect on quantification, but no significant effects were found. We also resolved a technical issue involving the low viscosity of the hexane causing the system to operate at low pressure. Placing a restrictor in the system increased overall system pressure and prevented back-pressure fluctuations. In spite of occasional negative press about inconsistent NP results, our retention times, peak shapes, and peak areas were all very consistent. We believe this is likely due to allowing sufficient time for the system to equilibrate between runs.

Although the method described above was developed for canola oil, it has been applied successfully to other vegetable oils, including olive oil, sunflower oil, and carotino oil (a blend of red palm fruit and canola oil). It can also be modified to examine a wider range of oils with different bioactive compositions, by monitoring a different suite of ion transitions characteristic to the compounds of interest. The new method, which was published in *Food Chemistry* [2], has since has been extended to investigate carotenoids in grape skin extracts, indicating that the new method presents further opportunities

to evaluate bioactives in other food sources. Its method development was part of an Australian research project currently underway at Charles Sturt University. The project aims to investigate bioactives in canola seed and oil, and ways in which they can be enhanced to further improve canola oil quality.

Clare Flakelar is a doctoral student at Charles Sturt University (CSU), Wagga Wagga, Australia, where she investigates several classes of bioactive compounds in canola seed and crude canola oil, and the agronomic and oil processing impacts on these compounds, under the supervision of Associate Professor Paul Prenzler. Her PhD project is funded by the Grains Research Development Corporation (GRDC) and the Graham Centre for Agricultural Innovation (a joint collaboration between Charles Sturt University and Department of Primary Industries). So far, the research has been presented at seven national and two international conferences, and has resulted in two publications. Clare is currently preparing her thesis, "Australian canola seed and oil quality-the influence of varietal traits and processing parameters on valuable minor components," for submission in 2017, and can be contacted at cflakelar@csu.edu.au.

Paul Prenzler is an associate professor at CSU, where his research typically involves the application of different analytical techniques to solve chemical problems of significance to the rural region in which the university is located, such as surveying Australian canola varieties for bioactive compounds with

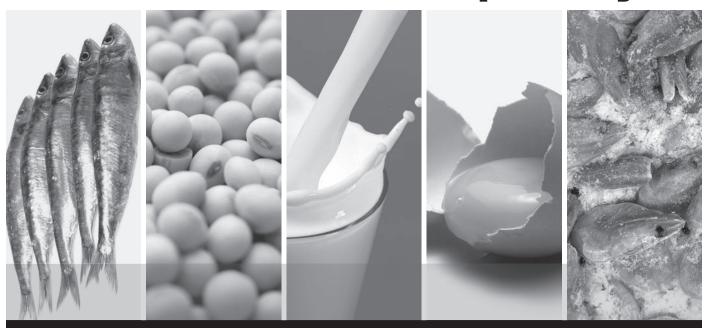
Suggested reading

[1] Flakelar, C.L., D.J. Luckett, J.A. Howitt, G. Doran, and P.D. Prenzler, Canola (Brassica napus) oil from Australian cultivars shows promising levels of tocopherols and carotenoids, along with good oxidative stability, J. Food Compos. Anal. 42: 179-186, 2015.

[2] Flakelar, C.L., P.D. Prenzler, D.J. Luckett, J.A. Howitt, G. Doran, A rapid method for the simultaneous quantification of the major tocopherols, carotenoids, free and esterified sterols in canola (Brassica napus) oil using normal phase liquid chromatography, Food Chem. *214*: 147–155, 2017.

roles in oil quality and health benefits, applications of gas chromatography-combustion-isotope ratio mass spectrometry in food authentication and traceability, development of a robust antioxidant test for lipid systems, analytical methods for diagnosis and prognosis of coeliac disease, and consumer preference studies on Australian chickpeas. A co-author of four book chapters and 99 refereed publications, Prenzler is the recipient of the Vice-Chancellor's Award for Research Excellence, chair of the Riverina-Murray Section of the Royal Australian Chemical Institute, and actively involved in promoting chemistry in High Schools. He can be contacted at PPrenzler@csu.edu.au.

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Accelerated shelf life studies of extra virgin olive oils using the Oxitest method

Marisa C. Caruso, Fernanda Galgano, Teresa Scarpa, Paola Ornaghi, and Fabio Favati

- The VELP Scientifica Oxitest is an analytical instrument that can be used to examine fat oxidation in whole samples.
- The Oxitest method was used to analyze the oxidative stability of extra virgin olive oils coming from two regions of Italy.
- There was a strong correlation between the total quantity of polyphenols and oxidation stability, notwithstanding differences in cultivation and geographic origin.

In the food industry, it is very important to determine the shelf life of raw materials and processed foods as quickly as possible. The Oxitest method is a rapid analytical technique used to examine fat oxidation. The method's key advantage is that results are obtained by analyzing whole samples, whereas other techniques require the fat content to be extracted before oxidation tests are performed. The Oxitest method has been recognized as an AOCS International Standard Procedure (Cd 12c-16) thanks to VELP Scientifica's efforts to develop the instrument and method in collaboration with universities and research centers. The new method is in the *Official Methods and Recommended Practices of the AOCS*, 7th Edition, and can be preordered at https://www.aocs.org/store/shop-aocs/shop-aocs?productId=72560532.

Lipid oxidation is influenced by a variety of factors, including light, heat, fatty acid composition and degree of saturation, the form and concentration of oxygen, and the presence of minor compounds such as antioxidants, metals, and pigments. Systematic studies on these factors have been conducted to improve the oxidative stability of foods (Comandini *et al.* 2009). Lipid oxidation is a slow process that could last days or months, depending on the sample. The Oxitest reactor subjects samples to a high-oxidative-stress environment, so that their resistance to oxidation can be evaluated in a short period of time (Comandini *et al.* 2009).



The instrument features two thermostated and hermetically sealed titanium chambers in which oxygen is purged until the pressure within both chambers is between 0-8 bar (Fig. 1). The temperature is then set to the desired level (room temp. to 110 °C). The Oxitest measures the absolute pressure change inside the two chambers monitoring the oxygen uptake of the active components of the samples, and automatically generates a value expressed in time, called the Induction Period (IP). The longer the IP, the more resistant a sample is to oxidation over the life of the sample.

During the past several years, shelf life studies of vegetable oils have raised considerable attention, and several scientists have started investigating how vegetable oils respond to oxidation tests. Extra virgin olive oil has become an increasingly popular subject of such studies, as this particular oil is an important component of the Mediterranean diet and is broadly recognized for its positive health benefits.

Lipid oxidation is the main degradation process affecting the shelf life and compromising the organoleptic and nutritional characteristics of oils, fats, and foods that contain them. (Tura et al. 2007). Olive oil has more oxidative stability than other vegetable oils due to its low unsaturated fat content (Martinez et al., 2014). Extra virgin olive oils are particularly resistant to oxidation due to the high quality of raw material and softer method of extraction. In fact, extra virgin olive oils have a higher percentage of natural antioxidants, particularly of polyphenols and α -tocopherol (Favati et al. 2013).



FIG. 1. Sample loading in the Oxitest chambers

Further reading

Comandini, P., V. Verardo, P. Maiocchi, and M.F. Caboni, Accelerated oxidation: Comparative study of a new reactor with oxidation stability instrument, *Eur. J. Lipid Sci. Technol.* 111: 933–940, 2009

Favati, F., N. Condelli, F. Galgano, and M.C. Caruso, Extra virgin olive oil bitterness evaluation by sensory and chemical analyses, *Food Chem.139*: 949–954, 2013

Martínez, M., M. Fuentes, N. Franco, J. Sánchez, and C. De Miguel, Fatty acid profiles of virgin olive oils from the five olive-growing zones of Extreemadura (Spain), *J. Am. Oil Chem. 91*: 1921–1929, 2014

Tura, D., C. Gigliotti, S. Pedò, O. Failla, D. Bassi, and A. Serraiocco, Influence and cultivar and site cultivation on levels of lipophilic and hydrophilic antioxidants in virgin olive oils (*Olea europea* L.) and correlations with oxidative stability, *Sci. Hort.* 112:108–119, 2007

Scientists from the University of Basilicata (Potenza, Italy), the University of Verona, and VELP Scientifica (Usmate, Italy) have conducted stability tests on extra virgin olive oil as a part of the "Eufolia Mediterranea" project, aiming to assess the behavior under oxidative stress of extra virgin oils from conventional and experimental olive cultivation. Three samples from the cultivar *Ogliarola del Bradano* were obtained from the same field in the Basilicata region in southern Italy. All three samples were extracted in the same facility under the

same operative conditions. The oxidation stability tests were performed at a constant temperature of 90°C and an oxygen pressure of 600 kPa, with three repetitions for each sample.

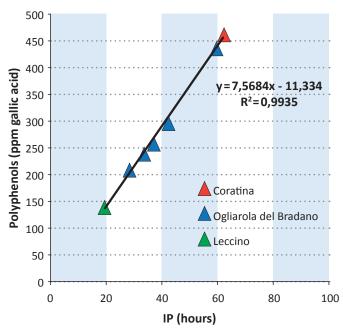
The three samples differed with respect to the year and the method in which the olives were cultivated. The first sample (PFI 2013 C) was obtained in 2013, from a conventional cultivation. The second (PFI 2014 C) was also obtained from a conventional cultivation, but in the following year (2014). The third sample (PFI 2014 E), also obtained in 2014, was obtained from an experimental cultivation in which low environmental impact and high-efficiency technologies were used.

When the IP values of the three investigated oils were compared, the oil from conventional cultivation in 2013 had a higher oxidative stability than the oil that was conventionally cultivated in 2014, which was likely due to climatic parameters. However, the sample obtained from the experimental cultivation in 2014 was considerably more stable, with an IP value 10 h longer than the sample obtained from conventional cultivation during the same year, indicating that the experimental cultivation techniques had a positive effect on the oxidative stability of extra virgin olive oil.

The oxidative stability of the three oils from the Eufolia Mediterranea project was compared with the oxidative stability of other oils from the Basilicata region, including two oils from the same variety (*Ogliarola del Bradano*), one oil from the variety *Leccino*, and another one from the *Coratina* variety. Among the samples tested, those from the *Leccino* variety had the lowest IP value (19.5 h), and those from *Coratina* the highest (62 h).

Moreover, the oxidative stability of five PDO (Protected Designation of Origin) extra virgin olive oils from the Garda lake area in northern Italy, was analyzed. These oils were characterized by IP values ranging from 38 to 78 hours

A) Basilicata Oils



B) Garda DOP Oils

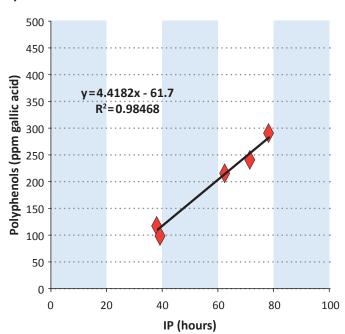


FIG. 2. Correlation between IP values and total polyphenol content for Basilicata(a)and Garda(b)extra virgin olive oils

The IP assessed values for all the investigated oils span over a wide range, namely from 20 to 78 hours, with an observed overlapping of the specific ranges of the Basilicata and the Garda oils, showing anyhow the latter the highest IP values. However, the available analytical data did not allow to highlight a possible direct correlation between the geographical origin and IPs. In the study the extra virgin oils were also characterized taking into account other parameters: acidity, peroxides, K232, K270, p-Anisidine, chlorophyll, carotenoids, tocopherols, and total polyphenols. The results showed that when considering oils having the same geographical origin it was possible to highlight a significant positive correlation between the total polyphenols content and the assessed IPs, with R²> 0.993 and R²> 0.984 for Basilicata and Garda oils, respectively (Fig. 2). Conversely, such a strong correlation could not be observed for the other investigated parameters.

Fernanda Galgano is associate professor in the School of Agricultural, Forestry, Food and Environmental Science (SAFE) at the University of Basilicata (Potenza, Italy), where Marisa C. Caruso is a researcher and Teresa Scarpa is a research fellow. Fabio Favati is associate professor in the Department of Biotechnology at the University of Verona (Verona, Italy). Paola Ornaghi is a scientist of the Analytical Department of VELP Scientifica (Usmate, Italy).

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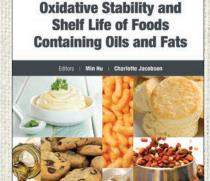




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Oxidative Stability and Shelf Life of Foods Containing Oils and Fats

Edited by Min Hu and Charlotte Jacobsen February 2016 | ISBN 9781630670566

Oxidative Stability and Shelf Life of Foods Containing Oils and Fats is the first book to address the critical role of oils and fats in the successful development, and improvement of, food and pet food products. From ensuring products are consumer acceptable in nutritional and sensory aspects to extending the lifespan between production and consumption, oxidative stability provides important options for foods containing oils and fats. This book explores both analytical and evaluative methods for bulk oils and fats, frying oils and fried foods, and food emulsions as well as for specific food product categories. Featuring 13 chapters written by renowned experts, this reference offers valuable insights into the latest research and applications.

- Presents the challenges of assessing oxidative stability and strategies for minimizing lipid oxidation and extending the shelf life of various food products
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- Includes new information on packaging technologies to control lipid oxidation
- Directed toward scientists working in the food and pet food industries

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A conversation with **Dr. Milton J. Rosen**

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

Laura Cassiday

During the 2017 AOCS Annual Meeting & Industry Showcases in Orlando, Florida, USA, April 30—May 3, a special session celebrating the 20th volume of the *Journal of Surfactants and Detergents (JSD)* will be presented in honor of AOCS Fellow Milton J. Rosen, one of the great pioneers of surfactant research and longtime advisory board member of the journal. I recently talked with Rosen about his impressive career. This issue's Olio column is devoted to him.

Few scientists have enjoyed such a long and illustrious career as Milton J. Rosen. Born in 1920 in Brooklyn, New York, Rosen received his master's degree from the University of Maryland in 1941. With the outbreak of World War II, Rosen served his country in the Pacific Theater as a member of the US Army. After discharge, Rosen accepted a teaching position at Brooklyn College, and in 1949, he earned a PhD in organic chemistry from the Polytechnic Institute of Brooklyn.

In his lab at Brooklyn College, Rosen became a pioneer in the field of surfactant research. Among his greatest scientific contributions were a series of studies investigating the structure-property relationships of surfactants; derivations of equations for surfactant synergism; and some of the earliest studies of gemini surfactants. In 1979, Rosen published the first edition of his groundbreaking and authoritative work, *Surfactants and Interfacial Phenomena*. "Everybody in the industry's got a copy of his book, *Surfactants and Interfacial Phenomena*," says Dennis Murphy, research fellow at Stepan Company, and a PhD student in Rosen's lab from 1985 to 1989. "It's in its fourth edition now, and it's kind of like the go-to book for surfactants." Recalling his time in Rosen's lab, Murphy describes Rosen as a "caring, very enthusiastic scientist with a great sense of humor."

In his 70 years as an active researcher, Rosen obtained more than 150 publications or patents. In 1985, he founded the Surfactant Research Institute at Brooklyn College, and in 1991, he established the AOCS Samuel J. Rosen Award, in honor of his father. Rosen was a longstanding member of the advisory board for the *Journal of Surfactants and Detergents*

(*JSD*), and in 1999, he was elected an AOCS Fellow. Rosen retired from the Surfactant Research Institute in 2011, at the age of 91. Now 96, he and wife of 68 years, Ellen, remain active: They take cruise vacations, and they are giving a talk at their local Temple on what is was like to live in Jerusalem during a research sabbatical in 1958. Rosen also stays abreast of developments in surfactant research, frequently reading *Inform* and *JSD*. Here are some highlights from our recent conversation.

How did you become interested in surfactants and detergents?

In 1942, I took a job with a firm in Brooklyn, then known as Glyco Products. They made emulsifiers and food additives, which were surfactants, but they knew nothing at all about how they worked or the basis for their actions. They knew that they could sell them and make money, and that's all they were interested in. That's how I got involved with surfactants. I was aware of the fact that we didn't know why they worked the way they did, or what other structures would be similar to them and their activities. We just knew that these were things that we could sell and make money for the firm.

How did you go from there to running your own surfactant research lab at Brooklyn College?

I came to Brooklyn College in 1946. As I said, I was working in the chemical industry from 1942 to 1944, and then I was drafted. When I came out of the Army, I had a job waiting for me at another firm. I went to the firm, and I said, "I'm



AOCS Fellow Milton J. Rosen in his lab at Brooklyn College.

here," and they said, "Well, can you wait until January, because we're opening up a new lab and we'd like you to start there." So I said, "Well, how do I eat between the end of August and January?" One of the people there, who was a Brooklyn College graduate, said to me, "Why don't you go to Brooklyn College?" It was just after World War II, and the soldiers were coming back, and they were anxious to have chemists to teach chemistry there. He said, "Why don't you go there for one semester, until the end of January, and then you can come to work?" So I called up Brooklyn College, told them I had a master's degree in chemistry, and they hired me immediately over the phone. They were so hungry for chemistry teachers. So I went there for 6 months and stayed for 65 years.

I had a lab there, and I started doing research on surfactants. First I did work on analyzing them because I was teaching a course in the analysis of organic compounds, so I used the methods that we were using there to analyze surfactants. Then I started to work on the physical chemistry of surfactants and, essentially, why and how they work. And that's where I got started on the major work that I did, which was structure-property relationships in surfactants. So it all came about because I didn't get a job at the other company.

What would you say were your biggest scientific contri-• butions to the field of surfactants and detergents?

Well, it was generally the knowledge of the relationship of the chemical structure of surfactants to how they work. So we now know how surfactants work and why they work, and what structures do what, and what other structures do other things, but basically that's the work that was my research for all those years.

As you know, in 2017 we're celebrating the 20th anniversary of the Journal of Surfactants and Detergents. Do you have a favorite or most memorable paper that you published in JSD?

I just learned that when JSD was first formed, I had the first paper that was published by them, on page 1 of the first edition [Zhu, Y.-P., Rosen, M. J., and Morrall, S. W. (1998) "Chemical structure/property relationships in surfactants. 17 N-substituted-N-acyl glycinates in pure and synthetic hard river water," JSD 1: 1-9].

Among my more recent papers over the last 20 years, I'd say that the paper on gemini surfactants [Rosen, M. J., and Tracy, D. J. (1998) "Gemini surfactants," JSD 1: 547-554] is one of my favorites because it introduced the world to a new type of surfactant, geminis, which are doing quite well, I understand.

When did you retire from Brooklyn College?
I retired in 2011, just five years ago. The story behind that is, at Brooklyn College, like many institutions, you have to retire when you are 65. For me, that would have been in 1985. So a few years before that, I established a surfactant research institute. I was getting enough grants that I was self-supporting in my research, and I was able to then establish an official research institute of the City University of New York at Brooklyn College. As director of that, I didn't have to retire. So I retired in 2011 instead of 1985. I really loved what I was doing. I enjoyed every minute of my time doing research.

What do you see as the future of the surfactants field?
I think what's important is, first of all, that surfactants must be based on renewable resources, not petroleum or other things like that. They have to be based on renewable resources, which would be essentially fats and proteins, which I used and which can be used in surfactants. They must have practically no, or no significant, effect on the environment—environmentally favorable. And they must be mild or have no effect on skin surfaces. That's where the areas of research are and should be because people are really quite environmentally intelligent and concerned.

There's going to be a session at the 2017 AOCS Annual Meeting that is in honor of you and your research. How do you feel about that?

I'm excited. I won't come because I hate to travel by plane. I'm 96 now, almost 97. My wife is very excited, also. She said, "Are you going?", and I said, "No, unfortunately, I'm not going to go." But really I'm very honored. It's a real surprise, and I'm appreciative.

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

The Samuel Rosen Award

At the 1991 AOCS Annual Meeting, Milton J. Rosen announced the establishment of the Samuel Rosen Memorial Award, in honor of his father. Born in Poland in 1889, Samuel Rosen came to the United States at the age of two. His parents divorced, and his father, who had taken the two boys of the family, remarried. Unfortunately, he neglected to tell the woman he married that he had two sons until after the wedding, and she had little interest in raising two boys. Soon Samuel and his older brother found themselves out on the streets of New York City, forced to fend for themselves, when Samuel was only 7 years old. Samuel sold newspapers for a profit of 15–20 cents per day, and slept in a Newsboys' Lodging House that charged 5 cents per night (with a free breakfast, as long as he made his bed). Whenever he could save enough money to take a few days off from work, Samuel would attend school.

When he was older, Samuel obtained a job at a printing press, where he learned about the various inks used to print books and newspapers. Largely self-educated, Samuel Rosen then acquired a job as an industrial chemist, formulating printing inks for the J.M. Huber Co. After 40 years with this company, he joined the printing inks technical service division of Interchemical Corp. Retiring in his 70s, Samuel Rosen lived until the age of 97, passing away shortly after the death of his wife of 72 years, Fannie.

The Samuel Rosen Memorial Award was established in memory of his dedication to and enthusiasm for applied chemistry. The award recognizes significant accomplishments in applications of the principles of surfactant chemistry, by scientists who have spent the majority of their careers in industry.



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Chemical notification process "very different" under new TSCA

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

Kelly Franklin

Bringing a new chemical to the market under the new Toxic Substances Control Act (TSCA) will be a very different process to that which manufacturers have grown accustomed to. As amended by the Lautenberg Chemical Safety Act, TSCA now requires an "affirmative finding" of safety for pre-manufacture notices (PMNs). If the agency decides a substance poses an unreasonable risk, it must issue an order to prohibit or restrict it—and these are already on the rise in the program's early days.

According to Jeffrey Morris, deputy director for programs at Office of Pollution Prevention and Toxics (OPPT), this requirement is a very big change to the new chemicals process.

And Lynn Bergeson, managing partner at Bergeson & Campbell, said she's not sure industry appreciates "just how significantly the ballgame has changed."

EVOLVE AND DEVELOP

Morris said the new TSCA will evolve and develop through time.

"It's going to be a lot of working through particular cases and learning from them. We're going to have to look at what we've done and realize—maybe we should have done this a different way, and make adjustments. That's just going to be the nature of implementing a significant change to the law."

But Bergeson pointed out that it's not incumbent on just the US Environmental Protection Agency (EPA) to manage the change. PMN submissions, she said, will now require more strategic review, and companies should more closely consider available information that can be submitted "to rebut certain inferences that otherwise might follow."



OBSERVATIONS ON THE GROUND

Sharing her perspectives as a product stewardship manager, Shannon Gainey, from global specialty chemicals company Evonik Corporation, said companies will need a couple of years to "weather the storm" of the new chemicals process.

Regarding the affirmative determination, Gainey said in her experience, unless a new substance being notified has a low human health hazard and low environmental hazard, there will be a hazard consideration. And, unless the exposure to this substance is essentially zero, the EPA will likely identify a risk.

Bearing in mind how the EPA evaluates new chemistries, she said, the agency's models "will predict a very conservative number in terms of how potentially toxic something is, and the models generally will overestimate the quantity potentially released to the environment," based on manufacturing, processing and uses described in the PMN.

So in situations where a chemical has predicted toxicity especially ecotoxicity—a PMN submitter, she said, may benefit from producing data. This is particularly the case for chronic toxicity, as this is an area where data gaps may force the EPA to rely on the outcome of models instead.

Further considerations for businesses, she said, include:

 allowing for, and communicating to customers, a longer lead time to bring new substances to the market;

- planning for increased costs;
- communicating early and often with the EPA, particularly around what assumptions it is using in its evaluations, or on the data gaps it faces;
- increased testing, including possibly expanding capacity to run phys-chem properties;
- preparing for increased controls and restrictions following the review process, including educating customers that significant new use rules (Snurs) and orders are the way of the future; and
- collaborating within industry, including sharing case studies.

Gainey also said that companies may consider alternative uses for existing substance already on the inventory, while the new chemicals process continues to mature.

REASONABLY FORESEEN

Under the new law, the EPA must now make an affirmative finding of safety on both the known and "reasonably foreseen" uses of a substance.

Morris said the agency's interpretation of the law is that "known uses" are those included in a PMN. The EPA is currently interpreting "reasonably foreseen" to mean the known uses of similar substances.

"If there are uses associated with a close analog to this chemical—a chemical that seems that it's closely related and could likely be used in the same way as the PMN substance—and [these uses] are not on the PMN form, then we have to make our determination not only on the uses that are in the PMN form, but also on those reasonably foreseen uses."

In the past, he said, a minority of submitters received consent orders but "that's going to change now." The agency will need to issue an order and a significant new use rule (Snur) to articulate what protections would be needed to address a finding for not only the known use, but also for those reasonably foreseen ones.

In many cases, the order might just bind the submitter to the conditions outlined in its PMN. But, said Morris, "it's still an order, and it's still something new."

INSUFFICIENT INFORMATION

Regarding the law's new insufficient information finding, Morris said this is also a new area for industry and the EPA alike. As "most PMN submissions do not come with a full battery of information that would allow us to make a determination," in many cases, the EPA will rely on analogous substances, he said.

But "it's not clear that in a lot of those cases that just going from an analogy—without a full body of information from a PMN submission—is going to allow us to move past that designation of 'insufficient information.'"

This raises several questions, said Morris, such as:

- what are the testing implications for insufficient information:
- what information does the law require the agency to ask for beyond what it has previously requested for new chemicals; and
- should the EPA require tests that the regulated community is familiar with, or should the agency be exploring more 'creative', or alternative ways that it can use to gather the information it needs to make its determination.

Evonik's Gainey said that for some early PMN determinations, the agency's upfront testing requests have apparently been "pretty significant." Some have included 90–day inhalation studies, for example.

"It will be clear very soon," said Morris, that the discussion around the testing implications of the changes to Section 5 of TSCA will be "very important" to how the EPA operates.

Kelly Franklin is editor, North America, for Chemical Watch.

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Pentadesma butyracea for cocoa butter equivalents and other applications Leslie Kleiner



Pentadesma butyracea, also known as African butter tree, is an evergreen that grows in West tropical Africa and produces in its kernels an edible fat similar to shea butter. A study by Tchobo, et al. (2007), of seeds grown in the country of Benin revealed that the fatty acid composition of the oilseed is mostly comprised of stearic acid and oleic acid. Furthermore, Tchobo, et al. (2009), described the production of a cocoa butter equivalent (CBE) by enzymatic transesterification of P. butyracea butter with ethyl palmitate. CBEs are of interest because these non-lauric fats have physical and chemical properties similar to cocoa butter, and can therefore be used to partially or fully replace it. To learn more about *P. butyracea* and its possible growth in Latin America, I interviewed Hemal de Silva and Manfred Harrer. De Silva is an agriculturalist in Sri Lanka who has experience growing *P. butyracea*, and provided seeds to Harrer, a food engineer and agriculturalist in Colombia, for testing in the Amazon region.

Could you describe planting to harvesting production of P. butyracea? What kind of requirements are needed for a successful crop?

(ds): P. butyracea remains an overlooked oilseed source in many forests of the West Tropical African region. However, this tree was introduced to the Fujian and Yunnan Provinces of China in the 1960s, and it has adapted well to its sub-tropical climate. In Benin—where the annual rainfall can be as low as 1,000 mm—it grows well, given proximity to streams that provide additional moisture. In Sri Lanka, the same regions where tea (Camellia sinensis) is traditionally grown have a climate suitable for the growth of P. butyracea. Seeds from a single Peradeniya tree planted in 1897 in the National Botanical Garden were used to establish the first small-scale plantation in Sri Lanka in 2009. The hypogeal fresh seeds germinate in seven to ten days, and seedlings for transplanting can be raised within four to six months. This species tolerates water logging and eventually grows into

a deep-rooted, strong, and sturdy tree yielding good quality timber. As a mono crop, it can be planted at 400 plants per hectare (ha), and can then be thinned out if necessary after about 15 to 20 years. The tree reaches maturity in approximately seven to eight years (Adomako, 1977) and, depending on climatic conditions, it can produce fruit throughout the year, as reported in the Seychelles. The fruit also yields edible pulp, 40 to 50% of the fresh fruit by weight.

What potential applications for P. butyracea do you see • in Sri Lanka, and other world regions?

(dS): Despite the importance of oilseed from Pentadesma butyracea, its production remains negligible in the Tropical West African Region. However, Badoussi, et al. (2014), reported many regional processing variations used to obtain *P. butyracea* butter; most of these processes are carried by local women. P. butyracea is not widely grown in Sri Lanka, but if this tree crop were to be established on a commercial scale for butter production and use of the fresh fruit, it would stimulate the local economy and aid in the development of small- and large-scale growers. Regarding uses in Sri Lanka and other areas of the world, the butter could be used as a source for CBE production, a trans-fat- free alternative for confectionery and other food applications, and for cosmetic applications.



What motivated you to try growing P. butyracea in Colombia?

(H): As a food engineer (University of Hohenheim, Germany), I worked for almost a decade in quality assurance and process engineering, specializing in baking technology. After meeting a Columbian woman who later became my wife, I decided to move to Columbia and start a bakery business. During this process, I realized that most of the nuts required for bakery applications were being imported. Therefore, I decided to explore the possibility of growing nuts locally.

What has your experience growing nuts—particularly • P. butyracea—in Colombia been like?

(H): First, it is important to mention that in many rural areas in Colombia much of the agriculture is by trial and error, since some of these areas have high incidences of poverty and a lack of agricultural investments. I first started planting Macadamia nuts, but with the large diversity of climate and soil conditions in Colombia, this crop proved to be challenging. Some common challenges were too much rain (5,000 to 6,000 mm rain per year), and too high of a requirement for fertilizers. Since Macadamia nut, I have screened many seeds, including Asian and African oilseed species, which generally start production 7 to 15 years after planting. Acquiring these seeds was also challenging, since guerrilla conflicts made it difficult to acquire seeds and farming land in the Amazons. The seeds of P. butyracea germinate easily, and the plant develops and grows well in soils of poor farming quality. P. butyracea requires little fertilization and does not seem to be affected by high levels of rain. Although the plants are growing well, I have not yet harvested the seeds from my farm, because it takes 7–8 years to reach that stage.

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



Further reading

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Tchobo, et al., Enzymatic synthesis of cocoa butter equivalent through transesterification of Pentadesma butyracea butter, J. Food Lipids 16: 605-617, 2009.

PATENTS

Ultrasound contrast agents and process for the preparation thereof

Schneider, M., et al., US9364569, June 6, 2016

Method for preparing a lyophilized matrix and, upon reconstitution of the same, a respective injectable contrast agent comprising a liquid aqueous suspension of gas-filled microbubbles stabilized predominantly by a phospholipid. The method comprises preparing an emulsion from an aqueous medium, a phospholipid, and a water-immiscible organic solvent. The emulsion is then freeze-dried and subsequently reconstituted in an aqueous suspension of gas-filled microbubbles. The method allows obtaining suspensions comprising microbubbles having a relatively small diameter and a narrow size distribution.

Compositions and methods for multimodal imaging

Jaffray, D., et al, Univ. Health Network, US9393326, July 7, 2016

There is provided signal-modifying compositions for medical imaging comprising a carrier and signal-modifying agents specific for two or more imaging modalities. The compositions are characterized by retention efficiency, with respect of the signal modifying agents that enables prolonged contrast imaging without significant depletion of the signal modifying agent from the carrier. The carriers of the present invention are lipid-based or polymer-based, the physico-chemical properties of which can be modified to entrap or chelate different signal modifying agents and mixtures thereof, and to target specific organs, tumors, or tissues within a mammal.

Liposome-peptide conjugate and method of using same to induce production of anti-HIV antibodies

 $Haynes, B.F., {\it et\,al.}, Univ.\, of\, Duke, US9402893, August\, 2, 2016$

The present invention relates to a method of inducing the production of anti-HIV antibodies. The method comprises administering at least one liposome-peptide conjugate, wherein the peptide comprises a membrane proximal external region (MPER) epitope.

Lipid-based compositions of antiinfectives for treating pulmonary infections and methods of use thereof

Weers, J., Insmed Inc., US9402845, August 2, 2016

A system for treating or providing prophylaxis against a pulmonary infection is disclosed comprising: a) a pharmaceutical formulation comprising a mixture of free anti-infective and anti-infective encapsulated in a lipid-based composition, and b) an inhalation delivery device. A method for providing prophylaxis against a pulmonary infection in a patient and a method of reducing the loss of anti-infective encapsulated in a lipid-based composition upon nebulization comprising administering an aerosolized pharmaceutical formulation comprising a mixture of free anti-infective and anti-infective encapsulated in a lipid-based composition is also disclosed.

Gas-filled microvesicles with polymermodified lipids

Gorgerat, G., et al., Bracco Research SA, US9446156, September 20, 2016

Image-enhancing contrast agents for use in diagnostic and/or therapeutic methods, particularly in the form of gas-filled microvesicles, with enhanced stability. The gas-filled microvesicles are stabilized by a layer of amphiphilic material and comprise from 0.15% to 1.0% by moles of a lipid bearing a hydrophilic polymer. The lipid bearing a hydrophilic polymer is preferably a phospholipid linked to polyethyleneglycol.

Botanical extracts from oil palm vegetation liquor for cosmeceutical applications

Sambanthamurthi. R., et al., Malaysian Palm Oil Board, US9381145, July 5, 2016

A cosmeceutical composition comprising phenolic compounds, fruit acids and sugars, extracted from the vegetation liquor of the palm oil milling process has been suggested. This composition is rich in antioxidants and significantly improves skin health, including preventing aging of skin.

Primary PVC plasticizers derived from vegetable oils, process for obtaining primary PVC plasticizers derived from vegetable oils, and plasticized PVC composition

De Quadros, J. F., Jr. and J.A. De Carvalho, Nexoleum Bioderivados Ltda, US9388292, July 12, 2016

PVC plasticizers are composed of epoxidized bioesters of vegetable oil fatty acids obtained by partial transesterification with an alcohol and glycerine; further acetylation and epoxidation; and PVC compounds plasticized with bioesters resulting from partial transesterification, acetylation, and epoxidation, belonging to the technical field of polymer additives that were developed to improve the properties of PVC polymers in addition to providing a lower cost for renewable compounds, such as those obtained with the use of vegetable oils. The epoxidized bioesters are composed by mixtures of epoxidized ethyl esters and acetylated and epoxidized mono-, di-, and triglyceril esters, presenting oxirane index between 4 and 8.

Method for enhancing catalytic activity of a lipase

Thakker, D.R., Univ. of North Carolina at Chapel Hill, US9416383, August 15, 2016

Methods for enhancing a biological activity, for example, catalytic activity, of a lipase, are provided. In some embodiments, the methods include the step of alkylating one or more cysteine residues present within the lipase. Also provided are modified polypeptides for which a biological activity is enhanced by the disclosed methods, methods for using the disclosed polypeptides, including for the transesterification of renewable oils to produce a biofuel, and cell free systems that include a lipase, to which one or more moieties, such as steroidal moieties, are conjugated.

Use of oils with high concentrations of polyunsaturated fatty acids in plastics and surface coatings

Morgenstern, D. A., et al., Monsanto Technology LLC, US9376580, June 28, 2016

Oil compositions having a high concentration of polyunsaturated fatty acids are described for use in various applications, including use as drying oils, in ink compositions, and coating compositions. Oil compositions wherein the double bonds of the fatty acids are substantially epoxidized are described and used as plasticizers and thermal stabilizers for various halogenated polymer compositions.

Sunflower oil with high heat stability

Velasco Varo, L., et al., Consejo Superior Investigacion, US9375023, June 6, 2016

The invention relates to a sunflower oil with high heat stability, which is characterized in that between 15% and 45% of the fatty acid total are saturated fatty acids (palmitic acid and stearic acid), between 45% and 75% of the fatty acid total is oleic acid, and more than 85% of the tocopherol total corresponds to the sum of γ -tocopherol and δ -tocopherol. The invention also relates to sunflower seeds that contain an oil with the aforementioned characteristics and sunflower plants which as a result of self-pollination produce seeds with the aforementioned characteristics. In addition, the invention relates to the use of said oil in food and animal feed and for the formulation of biolubricants and biofuels.

Apparatus for determining the texture of food material

Loeser, U., et al., Kraft Foods R & D Inc., US9417214, August 16,

The present application relates to an apparatus for determining the texture of food material, wherein the apparatus comprises a device for generating a vibrational impact, such as a piezo actuator; a device for measuring vibrations, such as a vibrometer; a holding mechanism, comprising a container body for containing the food

material therein or a string to attach the food material thereto; and an analyzer for comparing measured vibrations with at least one reference value, determined prior to the measurement.

Method for inhibiting the growth of bacteria

Petschow, B.W., Mead Johnson Nutrition Co., US9439885, September 13, 2016

The present invention is directed to a method for inhibiting the growth of pathogenic bacteria in an infant formula comprising supplementing the infant formula with at least one diglyceride antimicrobial agent.

Composition and method to improve the fuel economy of hydrocarbon-fueled internal combustion engines

Jung, A.K., et al., BASF Corp., US9447351, September 20, 2016 A composition and method of improving the fuel economy of hydrocarbon fuel-powdered internal combustion engines. The composition contains a propoxylated and/or butoxylated reaction product of (a) at least one fatty acid, fatty acid ester, or mixture thereof and (b) a dialkanolamime. The composition is added to a hydrocarbon fuel in an amount of about 5 to about 2,000 ppm, based on the weight of the hydrocarbon fuel, to reduce friction within the engine and achieve an enhanced fuel economy.

Inhibition of pancreatic lipase

Richardson, J.C., et al., Rd. Biomed. Ltd., Univ. of Newcastle Upon Tyne, US9402858, August 2, 2016

The present invention provides the use of an alginate IO inhibit pancreatic lipase. The use may be directed to the control of weight in animals including human beings. Preferably the fraction of guluronate dimers in the alginate is at least 0.5.

Enzymatic generation of functional lipids from cereals or cereal bi-streams

Sorensen, J.F., Dupont Nutrition Biosci. ApS, US9370193, June 21,

The present invention relates to the modification of lipids in lipid-containing plant material, such as cereal bran, for the generation of functional lipids. The present invention further relates to the preparation of compositions comprising such functional lipids as well as the use of these compositions comprising functional lipids and other functional compounds derived from the action of lipid modifying enzymes for the preparation of composition suitable for the preparation of bio-ethanol as well as food products, such as bread.

Microencapsulation of bioactive substances and methods of making the same

Harel, M., Advanced Bionutrition Corp., US9445613, September 20, 2016

The present invention relates to microparticles and methods of making such microparticles that protect a bioactive substance from heat, humidity, and oxidation. A microparticle comprising a bioactive substance, an agglomerating agent, an emulsifier, and solid fats is disclosed. A method to produce a microparticle comprising an agglomerated bioactive substance enrobed in a double layer of solid fats and emulsifier is also disclosed.

Method and apparatus for the making of a fuel

Collings, A., Ausbiodiesel Pty Ltd., US9428703, August 30, 2016
A method of making a fuel is disclosed. The method has a first step of exposing a liquid having biologically derived particles therein to an ultrasonic wave producing cavitation in the liquid and release of a precursor of the fuel from at least some of the particles into the liquid. The method also comprises the step of exposing the liquid with another ultrasonic wave insufficient to produce substantial cavitation in the liquid, another ultrasonic wave providing a reaction between the liquid and the precursor to form the fuel.

Composition for cosmetic, pharmaceutical and dietary applications

Rebmann, H., Lipoid GMBH, US9433570, September 6, 2016 A composition including 0.5–40 weight percent vegetable extract, 30–90 weight percent sugar, and 0.5–30 weight percent phospholipid for cosmetic, pharmaceutical, or dietary uses.

Gasohol fuel composition for internal combustion engines

Rawat, J., Bharat Petroleum Corp Ltd., US9447343, September 20, 2016

The present invention relates to a gasohol fuel composition that prevents corrosion of the metallurgies involved in fuel storage tanks, vehicle fuel tanks, fuel distribution systems, and transportation systems. The novel gasohol fuel composition comprises of a major portion of an alcohol blended gasoline fuel and a minor portion of a corrosion inhibitor formulation, wherein the corrosion inhibitor formulation comprises of (i) a reaction product of (a) a monosaturated fatty acid, and (b) an azomethine compound derived from a condensation reaction between a carbonyl compound and an amine compound; (ii) a fatty acid oil or ester selected from a group comprising of castor oil, palm oil, soyabean oil, and methyl soya ester; (iii) a dispersing agent, the dispersing agent

being a sulfonate compound; and (iv) a viscosity reducing agent selected from a group comprising of ethanol, isopropanol, and propargyl alcohol.

Use of sophorolipids and derivatives thereof in combination with pesticides as adjuvant/additive for plant protection and the industrial non-crop field

Giessler-Blank, S., et al., Evonik Degussa GMBH, US9351485, May 31, 2016

Use of sophorolipids as adjuvants in combination with pesticides as tank mix additive and/or as formulation additive for crop protection and for the industrial non-crop sector.

Acetylated monoglyceride of 12-hydroxystearic acid and blends with epoxidized fatty acid esters

Chaudhary, B.I., et al., Dow Global Tech LLC, US9422418, August 23, 2016

The present disclosure is directed to plasticizer compositions. A composition is provided which includes a castor-free acetylated glyceride of 12-hydroxystearic acid (AGHA) having a hydroxyl number from 0 to less than 15. The castor-free AGHA finds application in coatings for wire and cable.

Plasticized polymeric compositions

Kaytan, H., ISP Investments Inc., US9359486, June 7, 2016

The present invention provides plasticized polymer compositions comprising an 7V-allcyl-2-pyrrolidone and a fatty acid ester. These compositions exhibit enhanced hardness, tensile strength, and/or elongation at break without the use of phthalate-based plasticizers. The plasticized polymeric compositions can be employed in a wide variety of applications, such as in sheeting, tubing, and coatings.

Polymers derived from plant oil

Chisholm B.J. and A. Samim, NDSU Res Foundation, US9382352, June 5, 2016

Polymers and copolymers are formed from vinylether monomers having fatty acid ester pendent groups derived from plant oils, such as soybean oil.

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.



AOCS Journals: 2016 Editor-in Chief picks



Lipids

Lipids

- Balogun, K.A. and S.K. Cheema, Dietary omega-3 fatty acids prevented adipocyte hypertrophy by downregulating DGAT-2 and FABP-4 in a sexdependent fashion
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Journal of the American Oil Chemist' Society

- Jin, J., et al., Characteristics of specialty natural micronutrients in certain oilseeds and oils: plastochromanol-8, resveratrol, 5-hydroxytryptamine phenylpropanoid amides, lanosterol, ergosterol, and cyclolinopeptides
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EXTRACTS & DISTILLATES

Dietary cerebroside from sea cucumber (*Stichopus japonicus*): absorption and effects on skin barrier and cecal short-chain fatty acids

Duan, J., et al., J. Agric. Food Chem. 64: 7014–7021, 2016, http://dx.doi.org/10.1021/acs.jafc.6b02564.

Sphingolipids from marine sources have attracted more attention recently because of their distinctive structures and expected functions. In this study, the content and components of cerebroside from sea cucumber Stichopus japonicus were analyzed. The absorption of cerebroside from S. japonicus was investigated with an in vivo lipid absorption assay. The result revealed that *S. japonicus* is a rich source of cerebroside that contained considerable amounts of odd carbon chain sphingoid bases. The cumulative recoveries of d17:1- and d19:2-containing cerebrosides were 0.31 ± 0.16 and 0.32 \pm 0.10%, respectively, for 24 h after administration. To the best of the authors' knowledge, this is the first work that shows sphingolipids from a marine source could be absorbed in vivo and incorporated into ceramides. In addition, dietary supplementation with sea cucumber cerebroside to hairless mouse improved the skin barrier function and increased short-chain fatty acids in cecal contents, which have shown beneficial effects on the host.

Novel technologies for monitoring the in-line quality of virgin olive oil during manufacturing and storage

Ortega, J.B., et al., J. Sci. Food Agric. 96: 4644–4662, 2016, http://dx.doi.org/10.1002/jsfa.7733.

The quality of virgin olive oil is related to the agronomic conditions of the olive fruits and the process variables of the production process. Nowadays, food markets demand better products in terms of safety, health and organoleptic properties with competitive prices. Innovative techniques for process control, inspection and classification have been developed in order to to achieve these requirements. This paper presents a review of the most significant sensing technologies which are increasingly used in the olive oil industry to supervise and control the virgin olive oil production process. Throughout the present work, the main research studies in the literature that employ non-invasive technologies such as infrared spectroscopy, computer vision, machine olfaction technology, electronic tongues and dielectric spectroscopy are analysed and their main results and conclusions are presented. These technolo-

gies are used on olive fruit, olive slurry and olive oil to determine parameters such as acidity, peroxide indexes, ripening indexes, organoleptic properties and minor components, among others.

Bioactivity of phytosterols and their production in plant in vitro cultures

Miras-Moreno, B., *et al.*, *J. Agric. Food Chem.* 64: 7049–7058, 2016, http://dx.doi.org/10.1021/acs.jafc.6b02345.

Phytosterols are a kind of plant metabolite belonging to the triterpene family. These compounds are essential biomolecules for human health, and so they must be taken from foods. β-Sitosterol, campesterol, and stigmasterol are the main phytosterols found in plants. Phytosterols have beneficial effects on human health since they are able to reduce plasma cholesterol levels and have antiinflammatory, anti-diabetic, and ant-icancer activities. However, there are many difficulties in obtaining them, since the levels of these compounds produced from plant raw materials are low and their chemical synthesis is not economically profitable for commercial exploitation. A biotechnological alternative for their production is the use of plant cell and hairy root cultures. This review is focused on the biosynthesis of phytosterols and their function in both plants and humans as well as the different biotechnological strategies to increase phytosterol biosynthesis. Special attention is given to describing new methodologies based on the use of recombinant DNA technology to increase the levels of phytosterols.

Saturated branched chain, normal odd-carbon-numbered, and n-3 (omega-3) polyunsaturated fatty acids in freshwater fish in the Northeastern United States

Wang, D.H., et al., J. Agric. Food Chem. 64: 7512–7519, 2016, http://dx.doi.org/10.1021/acs.jafc.6b03491.

The fatty acid profiles of wild freshwater fish are poorly characterized as a human food source for several classes of fatty acids, particularly for branched chain fatty acids (BCFA), a major bioactive dietary component known to enter the US food supply primarily via dairy and beef fat. We evaluated the fatty acid content of 27 freshwater fish species captured in the northeastern US with emphasis on the BCFA and bioactive polyunsaturated fatty acids (PUFA) most associated with fish, specifically n-3 (omega-3) eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). Mean BCFA content across all species was $1.0 \pm 0.5\%$ (mean \pm SD) of total fatty acids in edible muscle, with rainbow smelt (Osmerus mordax) and pumpkinseed (Lepomis gibbosus) the highest at >2% BCFA. In comparison, EPA + DHA constituted 28% \pm 7% of total fatty acids. Across all fish species, the major BCFA were iso-15:0, anteiso-15:0, iso-16:0, iso-17:0 and anteiso-17:0. Fish skin had significantly higher BCFA content than muscle tissues, at $1.8\% \pm 0.7\%$, but lower EPA and DHA. Total BCFA in fish skins was positively related with that in muscle ($r^2 = 0.6$). The straight chain saturates n-15:0 and n-17:0 which have been identified previously as markers for dairy consumption were relatively high

with means of 0.4% and 0.6%, respectively, and may be an underappreciated marker for seafood intake. Consuming a standardized portion, 70 g (2.5 oz), of wild freshwater fish contributes only small amounts of BCFA, 2.5-24.2 mg, to the American diet, while it adds surprisingly high amounts of EPA + DHA (107 mg to 558 mg).

A Vernonia diacylglycerol acyltransferase can increase renewable oil production

Hatanaka, T., et al., J. Agric. Food Chem. 64: 7188-7194, 2016, http://dx.doi.org/10.1021/acs.jafc.6b02498.

Increasing the production of plant oils such as soybean oil as a renewable resource for food and fuel is valuable. Successful breeding for higher oil levels in soybean, however, usually results in reduced protein, a second valuable seed component. This study shows that by manipulating a highly active acyl-CoA:diacylglycerol acyltransferase (DGAT) the hydrocarbon flux to oil in oilseeds can be increased without reducing the protein component. Compared to other plant DGATs, a DGAT from Vernonia galamensis (VgDGAT1A) produces much higher oil synthesis and accumulation activity in yeast, insect cells, and soybean. Soybean lines expressing VgDGAT1A show a 4% increase in oil content without reductions in seed protein contents or yield per unit land area. Incorporation of this trait into 50% of soybeans worldwide could

result in an increase of 850 million kg oil/year without new land use or inputs and be worth ~US \$1 billion/year at 2012 production and market prices.

Enzymatic synthesis of steryl ferulates

Schär, A. and L. Nyström, Eur. J. Lipid Sci. Technol. 118: 1557-1565, 2016, http://dx.doi.org/10.1002/ejlt.201500586.

Steryl ferulates are plant sterols esterified to ferulic acid, a common phenolic acid. This esterification leads to sterol esters with improved biological properties, such as antioxidant activity. Commercially available and extracted steryl ferulates from rice bran are often limited in their sterol profiles. For further research and later food applications, a simple enzymatic esterification could address the lack of availability of single steryl ferulates. Whereas several enzymatic procedures for the esterification of steryl fatty acid esters have been published, no fully enzymatic procedure for steryl ferulates has been reported so far. We optimized both direct esterification of β -sitosterol with ferulic acid as well as transesterification with ethyl ferulate yielding steryl ferulates. The reaction was catalyzed by a lipase from Candida rugosa, which lead to yields of 35 and 55% for the direct esterification and transesterification, respectively. Moreover, both reactions followed a similar time course over incubation. The enzyme activity was rather low, which is probably due to the specificity of the different isoenzymes of C. rugosa lipase. However, successful conditions for a fully enzymatic synthesis of steryl ferulates are reported for the first time.

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Karen Letourneau has worked at POS Bio-Sciences for 26 years and POS Analytical Services has had an AOCS Approved Chemist on staff since 2011. "Being part of the **AOCS Lab Proficiency Program** really improves the quality of our testing." Letourneau explains that although POS Bio-Sciences had been performing marine oil lab testing for years, the laboratory was striving for greater accuracy and consistency. "Comparing our data to other labs' data helped us improve, and we used the check samples obtained through AOCS to train our technicians," she says. "Our efforts paid off and we placed first in 2014 in the AOCS Marine Oil series."

KAREN LETOURNEAU



Transcriptional analysis of stearoyl-acyl carrier protein desaturase genes from olive (*Olea europaea*) in relation to the oleic acid content of the virgin olive oil

Parvini, F., et al., J. Agric. Food Chem. 64: 7770–7781, 2016, http://dx.doi.org/10.1021/acs.jafc.6b02963.

The specific contribution of different stearoyl-ACP desaturase (SAD) genes to the oleic acid content in olive (Olea europaea) fruit has been studied. Toward that end, we isolated three distinct cDNA clones encoding three SAD isoforms from olive (cv. Picual), as revealed by sequence analysis. The expression levels of olive SAD genes were determined in different tissues from Picual and Arbequina cultivars, including developing mesocarp and seed, together with the unsaturated fatty acid content. Lipid and gene expression analyses indicate that OeSAD2 seems to be the main gene contributing to the oleic acid content of the olive fruit and, therefore, of the virgin olive oil. This conclusion was confirmed when the study was extended to Hojiblanca, Picudo, and Manzanilla cultivars. Furthermore, our data indicate that the olive microsomal oleate desaturase gene OeFAD2-2, but not OeSAD2, is responsible for the linoleic acid content in the virgin olive oil.

Eicosapentaenoic acid promotes mitochondrial biogenesis and beige-like features in subcutaneous adipocytes from overweight subjects

Laiglesia, L.M., et al., J. Nutr. Biochem. 37: 76–82, 2016, http://dx.doi.org/10.1016/j.jnutbio.2016.07.019

Eicosapentaenoic acid (EPA), a n-3 long-chain polyunsaturated fatty acid, has been reported to have beneficial effects in obesity-associated metabolic disorders. The objective of the present study was to determine the effects of EPA on the regulation of genes involved in lipid metabolism, and the ability of EPA to induce mitochondrial biogenesis and beiging in subcutaneous adipocytes from overweight subjects. Fully differentiated human subcutaneous adipocytes from overweight females (BMI: 28.1–29.8 kg/m2) were treated with EPA (100–200 μM) for 24 h. Changes in mRNA expression levels of genes involved in lipogenesis, fatty acid oxidation and mitochondrial biogenesis were determined by qRT-PCR. Mitochondrial content was evaluated using MitoTracker® Green stain. The effects on peroxisome proliferator-activated receptor gamma, co-activator 1 alpha (PGC-1a) and AMP-activated protein kinase (AMPK) were also characterized. EPA down-regulated lipogenic genes expression while up-regulated genes involved in fatty acid oxidation. Moreover, EPA-treated adipocytes showed increased mitochondrial content, accompanied by an up-regulation of nuclear respiratory factor-1, mitochondrial transcription factor A and cytochrome c oxidase IV mRNA expression. EPA also promoted the activation of master regulators of mitochondrial biogenesis such as sirtuin 1, PGC1-α and AMPK. In parallel, EPA induced the expression of genes that typify beige adipocytes such as fat determination factor PR domain containing 16, uncoupling protein 1 and cell death-inducing DFFA-like effector A, T-Box protein 1 and CD137. Our results suggest that EPA induces a remodeling of adipocyte metabolism preventing fat storage and promoting fatty acid oxidation, mitochondrial biogenesis and beige-like markers in human subcutaneous adipocytes from overweight subjects.

Rapid development of a castor cultivar with increased oil content

Chen, G.Q., et al., Ind. Crop. Prod. 94: 586–588, 2016, http://dx.doi.org/10.1016/j.indcrop.2016.09.020.

Castor seed oil contains 90% ricinoleic acid which has a wide range of industrial applications. Improvement in oil content would be of great benefit to castor growers and oil processers. Two cycles of phenotypic recurrent selection were conducted through screening for high oil content castor seeds using magnetic resonance spectroscopy (NMR). Selection increased mean oil content of a base population (Cycle 0) of cultivar Impala from 50.33% to 53.87% in Cycle 1 and 54.47% in Cycle 2. Gains from Cycle 1 and Cycle 2 were 3.54% and 0.6%, respectively. The small gain in the second cycle indicated a genetic ceiling of oil content or the genetic variability of the base population was extensively explored. Nevertheless, the 54.47% mean oil content of Cycle 2 ranks the selected material in the top 1% of the entire 1103 castor accessions maintained at United States of Department of Agriculture. As a result of the recurrent selection, we found average seed weight was also increased from 0.44 g in Cycle 0 to 0.50 g in Cycle 1 and 0.54 g in Cycle 2. Correlation between oil content and weight was moderate (r = 0.43, p < 0.0001) in Cycle 0, and strong in Cycle 1 (r = 0.63, p < 0.0001) and Cycle 2 (r = 0.77, p < 0.0001). To our knowledge, this is the first report of utilizing NMR and recurrent selection for improving oil content in castor.

Camelina uses, genetics, genomics, production, and management

Bertis, M., et al., Ind. Crop. Prod. 94: 690–710, 2016, http://dx.doi.org/10.1016/j.indcrop.2016.09.034

Camelina [Camelina sativa L. Crantz] is an annual oilseed crop in the Brassicaceae family that has been cultivated since 4000 BCE. Recently, interest in its oil, meal and the developed products has increased research in this crop. This renewed interest is evidenced by the tremendous increase in peer-reviewed publications containing the word 'camelina'. Databases report 335 publications between 2013 and 2016, with 149 of those published since 2015. The objective of this review was to compile and summarize new and existing information in order to identify gaps in knowledge and areas for future research. This review includes the most recent publications in camelina description and origin, uses, genetics, genomics, breeding, molecular genetics, physiology, agronomic management, and ecosystem services. Although the breadth of research in camelina over the last few years is impressive, several areas that would benefit from further research were identified. The development of new uses and the refinement of existing uses from camelina oil and meal will continue to add value to this crop. Advances in genetics, breeding, and genomics will speed up the development of high yielding camelina cultivars, with improved seed quality as well as disease and insect resistance. Understanding and improving freezing tolerance in camelina will advance the use of winter camelina



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as a cover crop or cash cover crop in double and relay cropping systems. Better management practices and weed control alternatives will be needed to increase camelina production worldwide. Lastly, commercial development of camelina will add one more crop to the already low agricultural diversity in many parts of the world.

Composition by LC-MS/MS of new carotenoid esters in mango and citrus

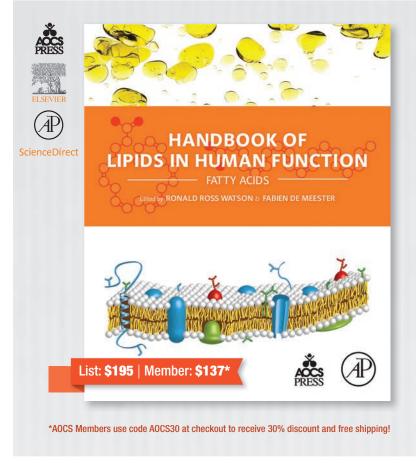
Petry, F.C. and A.Z. Mercadante, *J. Agric. Food Chem.* 64: 8207–8224, 2016, http://dx.doi.org/10.1021/acs.jafc.6b03226.

Interest in the composition of carotenoid esters of fruits is growing because esterification may affect their bioavailability. Thus, the aim was to provide a detailed identification of carotenoid esters in citrus and mango. Orange cv. "Valencia" and cv. "Pera" presented 9 free carotenoids, 38 monoesters, and 60 diesters. Violaxanthin and luteoxanthin derivatives were the major ones, followed by antheraxanthin, lutein, zeaxanthin, β -cryptoxanthin, and zeinoxanthin esters, many of them reported for the first time in orange pulp. The carotenoid ester composition of tangor cv. "Murcott," reported for the first time, showed 8 free carotenoids, 34 monoesters, and 33 diesters, with β -cryptoxanthin esters as major compounds, followed by violaxanthin and zeaxanthin esters. In citrus, carotenoids were acylated mainly with capric, lauric, myristic, myristoleic, palmitic, palmitoleic, and oleic acids. In mango, 5 free carotenoids, 2 monoesters, and 19 diesters were identified, from which many violaxanthin and neoxanthin esters were reported for the first time.

Rapid and innovative instrumental approaches for quality and authenticity of olive oils

Valli, E., et al., Eur. J. Lipid Sci. Technol. 118: 1601–1619, 2016, http://dx.doi.org/10.1002/ejlt.201600065.

The quality of virgin olive oils is assessed through the determination of several analytical parameters, whose values must be within the ranges established by the different institutions involved. In addition to official methods, there is a strong need for simple, rapid, and environmentally friendly techniques for the quality control of virgin olive oils and for addressing the challenging task of determining geographical origin and detecting adulterants. Toward this purpose, some of the most interesting applications based on optical spectroscopic techniques, on the measurement of electrical characteristics and on the use of instruments equipped with electronic chemical sensors, including also other promising techniques are herein discussed. These techniques, adequately coupled with an appropriate statistical approach, appear to be promising for assessment of several quality-related parameters. The prediction of sensory attributes and of the oxidative status of virgin olive oils have also been reviewed by adopting these selected techniques, which are also considered to be potentially appropriate solutions for identification of the geographical origin of virgin olive oils and to assess their adulteration with cheaper oils. Overall, the techniques discussed are promising and cutting-edge approaches for the establishment of useful portable instruments for in situ monitoring of the quality of virgin olive oils.



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Biodiesel program in Brazil: learning curve over ten years (2005–2015)

Nogueira, L.A.H., *et al.*, *Biofuels, Bioprod. Biorefin.*10: 728–737, 2016, http://dx.doi.org/10.1002/bbb.1718.

The biodiesel blending mandate was launched at 2005 in Brazil, by a governmental program based mainly on sustainability concerns, especially toward social and environmental aspects. Since 2007, B7, i.e., 7% biodiesel in diesel, in volume terms, has been mandatory, and B10 is the next target. In this context, this paper aims to evaluate the biodiesel program along the ten years from 2005 to 2015 in Brazil, considering the evolution of the estimated costs and the auction prices intending to verify some tendency in a learning curve perspective. A decreasing tendency of the biodiesel production costs and prices was observed, with a reduction of the profit margin in recent years, suggesting a learning trend. The Progress Ratio (PR) of the whole period was 101.7%, but in the 2011–2015, it was estimated at 59.3%. Relevant technology changes in biodiesel production process were not verified, but the increased competitiveness in the market, the gains of scale of plant profiles, and mainly the decrease of soybean oil prices explain this evolution.

AOCS MEETING WATCH

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LIPID CHEMISTRY

New way to attach lipids to proteins could streamline drug development

Protein-based drugs are used in the treatment of every kind of malady, from cancer to heart disease to rheumatoid arthritis. But the proteins are almost always modified with chemical appendages that help them navigate through the body or target specific tissues. A new study by Hao, Y., et al., "Molecular basis for the broad substrate selectivity of a peptide prenyltransferase," *Proc. Natl. Acad. Sci. USA*, online first, http://dx.doi.org/10.1073/pnas.1609869113, reveals an efficient means of attaching lipids to peptides (the building blocks of proteins). This can improve the molecules' drug-delivery capabilities.

"Medicinal chemistry has focused on using peptides as scaffolds for drugs because of the ease of their production and the chemical diversity of their amino-acid building blocks," said Satish K. Nair, a biochemistry professor at the University of Illinois, Urbana-Champaign, Illinois, USA, who led the new research with Thomas Cheatham and Eric Schmidt of the University of Utah. "However, peptides are generally ineffective drugs because they are poorly absorbed, cannot penetrate the blood-brain barrier, and are easily broken down," Nair said.

Attaching lipids "improves all of these properties, enabling peptides to be more drug-like," he said. Current methods for attaching lipids to peptides require the use of either harsh chemical solvents or expensive biological catalysts.

Nair and his colleagues focused on a little-known group of enzymes isolated from water-dwelling bacteria that have the remarkable ability to add lipids to a wide variety of proteins. The team performed a series of experiments on one family of these enzymes to discover how they recognize and interact with the peptides they modify.

The researchers discovered that one type of enzyme recognizes a simple, two-amino-acid sequence within its target proteins. They added this motif to two peptides selected at random and exposed the peptides to the enzyme. This caused the enzyme to add a lipid appendage to the proteins. The transformation was fast and efficient.

"Now that we have a very efficient way of attaching lipids to peptides, this opens up the possibility of using this approach to make large libraries of molecules that are more drug-like than peptides," Nair said.

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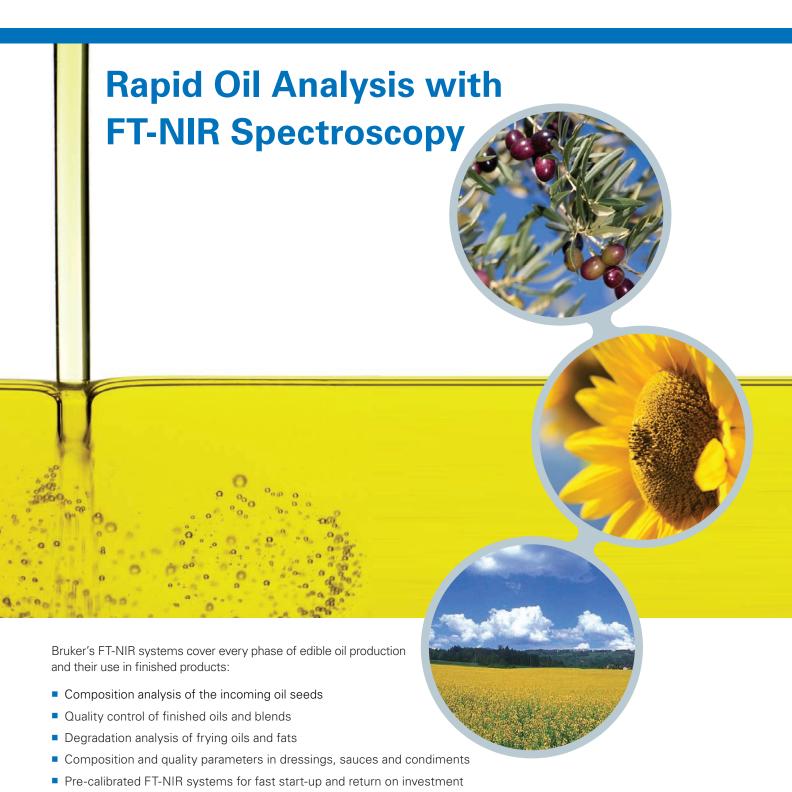
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