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Minimizing Contaminants

ALSO INSIDE:

Preserving micronutrients

Improving olive oil extraction

Dow/DuPont merger

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CONTENTS

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Erratum

Oops!

Some text on page 18 of the February 2016 issue is truncated and missing. A correct version of the text is available in the digital edition at http://tinyurl.com/hhyp7ms.

The errors in the print edition were made during production and are not a reflection on the author.

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Minimizing process contaminants in edible oils

Laura Cassiday

Refined edible oils undergo a complex transformation from the seed or fruit to the odorless, transparent, mild-flavored cooking oils preferred by consumers. After harvesting and extraction of the crude oil, a multi-step refining process removes impurities such as phospholipids, free fatty acids, pigments, metals, and waxes. However, edible oil processing can inadvertently introduce its own contaminants. Because some of these may be detrimental to human health, industry is adopting new strategies to detect and mitigate process contaminants in edible oils.

- Process contaminants are compounds introduced during the production of finished foods.
- Some process contaminants, such as mineral oil and phthalates, can be removed during edible oil refining. However, others, including 3-MCPD esters and glycidyl esters, may be created.
- Industry is adopting new strategies to detect and mitigate process contaminants in edible oils.

Process contaminants are defined as compounds introduced during the production of finished foods, for example, refined vegetable oils. Some contaminants are already present in the crude oil, like polycyclic aromatic hydrocarbons that form during heat drying of the crop by open fire. Others, including 3-monochloropropane-1,2-diol (3-MCPD) esters and glycidyl esters, are produced from compounds within the oil during refining. Some contaminants, such as petroleum-derived mineral oil and phthalates from certain plastics, can enter the oil from exogenous sources (for example, machinery or tubing) during processing. Trans fatty acids, which arise primarily during the hydrogenation of vegetable oils, are also considered process contaminants. Although partially hydrogenated oils are being phased out worldwide, low levels of trans fats can also be formed in some oils at the high temperatures used in refining. Fig. 1 summarizes the steps during edible oil refining at which process contaminants can be introduced and removed.

According to Florence Lacoste, head of analysis and expertise at the French Institute for Fats and Oils (ITERG) in Pessac, the major process contaminants of concern for edible oils are 3-MCPD esters, glycidyl esters, mineral oil, and phthalates (Table 1). At present, neither the European Commission nor the US government has passed regulations limiting the levels of these contaminants in edible oils, Lacoste says. Nonetheless, experts are actively researching new methods to quantify these contaminants and eliminate them from food oils.

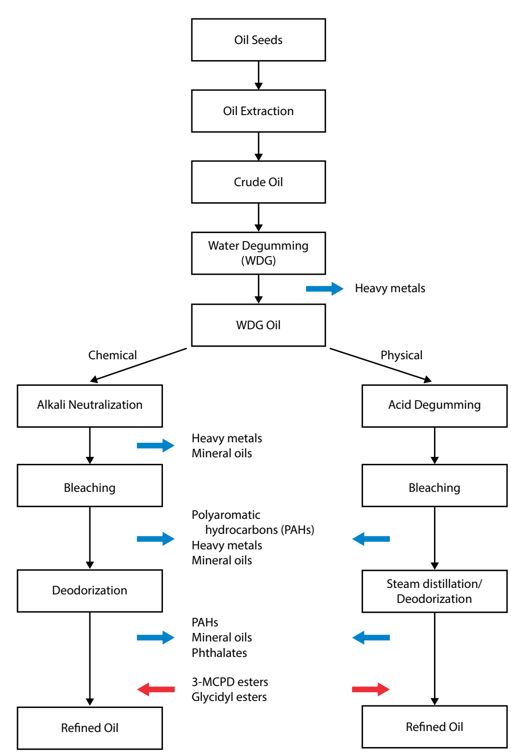


FIG. 1. Steps in edible oil refining during which process contaminants may be removed (blue arrows) or introduced (red arrows). Credit: AOCS

3-MCPD ESTERS

3-MCPD esters—members of a family of chemicals called chloropropanols—were first detected in refined vegetable oils in 2006. These contaminants can also be found in oil-containing foods such as bread, margarine, french fries, baby food, and infant formula. Little or no 3-MCPD esters are present in unrefined or virgin oils or animal fats.

3-MCPD esters are the most co mon chloropropanols in food, but related compounds such as 2-MCP; 1,3-DCP, and 2,3-DCP have also been detected (Watkins, C., *Inform,* 2009). Chloropropanols form in oils when subjected to elevated temperatures (above about 140 °C), such as during the deodorization step of edible oil refining. Deodorization is a steam distillation process carried out at low pressures (2–5 mbar) and elevated temperatures (180–270 °C) to remove volatile compounds, primarily aldehydes and ketones, that can adversely affect an oil's odor or flavor.

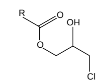
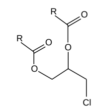
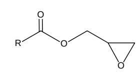
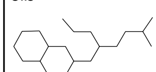
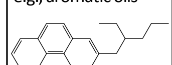
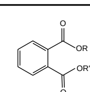
Chloropropane esters originate when triacylglycerides (TAGs) in an oil are hydrolyzed to diacylglycerides (DAGs) and monoacylglycerides (MAGs), which then undergo substitution with chloride ions. Depending on the identity of the fatty acid in the 3-MCPD ester, 14 different 3-MCPD monoesters and 49 diesters can be formed. 3-MCPD levels tend to be highest in palm, walnut, and grapeseed oil, where they can exceed concentrations of 2 mg/kg (Lacoste, F., http://dx.doi.org/10.1051/ocl/2013060, 2014).

When 3-MCPD esters are consumed, the digestive process rapidly releases free 3-MCPD from the ester. No human toxicological data are available on 3-MCPD esters, but animal studies have indicated that long-term intake of free 3-MCPD is carcinogenic and has been linked with infertility in rats, renal toxicity, and suppression of immune function. In 2007, Germany's Federal Institute for Risk Assessment (BfR) conducted an assessment of 3-MCPD esters in oil-containing foods. The BfR concluded that levels of 3-MCPD should be reduced, especially in infant formula and follow-up formula, although no acute danger was expected from current levels (Bakhiya, N. *et al.,* http://dx.doi.org/10.1002/mnfr.201000550, 2011).

In their analysis, the BfR assumed a "worst-case scenario" that 100% of 3-MCPD esters are cleaved to release free 3-MCPD during digestion. However, a subsequent *in vitro* study indicated that 3-MCPD is released more slowly from 3-MCPD diesters than monoesters, and that the majority (about 85%) of 3-MCPD in foods is in the diester form (Seefelder, W., *et al.,* http://dx.doi.org/10.1080 /02652030701385241, 2008). Therefore, exposure to free 3-MCPD from oil-containing foods may not be as high as anticipated.

Although 3-MCPD esters have only recently been detected in edible oils, free 3-MCPD was known to be a problematic contaminant of hydrolyzed vegetable proteins and soy sauce since the 1980s. As a result, in 2001 the European Commission set a maximum

TABLE 1. Major process contaminants in edible oils

Contaminant	Structure	Source	Possible Mitigation Strategies
3-MCPD Esters	Monoesters  Diesters 	Formed during deodorization (beginning at 140 °C) when DAGs or MAGs undergo chloride substitution	• Reduce DAG levels in crude oil by changing harvesting practices or using enzymatic treatment. • Wash raw oils to remove chloride precursors. • If possible, use chemical rather than physical refining. • Use neutral rather than activated bleaching earth to minimize chloride ions. • Remove 3-MCPD esters from refined oil with adsorbents.
Glycidyl Esters		Formed during deodorization (beginning at 230 °C) from DAGs by radicalar mechanism	• Reduce DAG levels in crude oil by changing harvesting practices or using enzymatic treatment. • If possible, use chemical rather than physical refining. • Minimize deodorization temperatures or use two-step deodorization. • Remove glycidyl esters from refined oil with adsorbents or by acid-catalyzed degradation.
Mineral Oil	MOSH: linear, branched, and cyclic alkanes e.g., napthenic oils  MOAH: alkylated aromatic hydrocarbons e.g., aromatic oils 	Formed by heating of petroleum oil. Enters edible oils from machinery used in harvesting and processing.	• Maintain machinery to prevent leakage of fuel or lubricants. • Use "white" (food-grade) mineral oil for edible applications. • Use relatively high (>240 °C) deodorization temperature.
Phthalates		Migrate from plastic materials in contact with oils and fats.	• Use phthalate-free plastics for edible oil processing. • If possible, use physical refining rather than chemical. • Use relatively high (>240 °C) deodorization temperature.

MAG, monoacylglyceride; DAG, diacylglyceride; MOSH, mineral oil saturated hydrocarbons; MOAH, mineral oil aromatic hydrocarbons.

concentration level of 20 µg/kg for 3-MCPD in hydrolyzed vegetable proteins and soy sauce, and a maximum Tolerable Daily Intake (TDI) of 2 µg 3-MCPD per kg body weight. Currently, no regulations address levels of 3-MCPD esters in edible oils or any other foods.

In 2013, the European Food Safety Authority (EFSA) issued a preliminary report on the occurrence of 3-MCPD in foods (European Food Safety Authority, http://dx.doi.org/10.2903/j.efsa.2013.3381). Like the BfR, the EFSA assumed a worst-case scenario of 100% conversion of 3-MCPD esters to 3-MCPD during digestion. Over the period 2009–2011, the EFSA reviewed data from 14 EU member states on the occurrence of 3-MCPD in various foods. They found that most population groups consumed less than half the TDI, but toddlers and the elderly are at risk of exceeding this limit. However, groups such as FEDIOL—the federation representing the EU vegetable oil and protein meal industry—and the American Oil Chemists' Society (AOCS) have pointed out that the EFSA risk assessment was conducted during a time when there were no validated methods for analyzing 3-MCPD esters in oils and fats (Watkins, C., *Inform,* 2014).

"I don't think the EFSA preliminary risk assessment is particularly reliable because they were using a lot of different methods to analyze 3-MCPD at that time," says Richard Cantrill, chief science officer at AOCS. He notes that a final EFSA evaluation on 3-MCPD in foods has been expected, but had not yet been issued as of December 2015. "Our issue with the latest EFSA evaluation that we're awaiting is whether they took into account newer data, or still relied on the previous material, because that might be a problem," says Cantrill.

Until recently, analyses of 3-MCPD esters in oils and fats were plagued by reproducibility problems. Several methods were in use, but researchers were not certain that they gave accurate or comparable results. Also, 3-MCPD could be formed or lost during sample preparation if strict protocols were not followed. To help rectify this situation, in 2013 AOCS adopted three validated methods for the determination of 3-MCPD esters, 2-MCPD esters, and a related compound, glycidyl esters (described in detail in the next section), in edible oils and fats. AOCS sponsored a collaborative study of three popular analytical methods using reference samples of known composition; 20 different labs from eight countries generated 31 data sets. The result: All three methods gave the same results with reasonable confidence. "Before, there was really a battle of methods," says Lacoste. "Now we know for sure that using one of these three methods will bring you the same results."

The methods are all indirect, meaning that they measure total 3-MCPD without differentiating among the various isomers. All three indirect methods chemically cleave the esters from 3-MCPD, and then measure free 3-MCPD by gas chromatography/mass spectrometry (GC/MS). "From a lab perspective, the methods are all very similar and require the same amount of dexterity," says Cantrill. They differ in details of the sample preparation steps, he says. For example, one of the methods requires the reactions to be carried out in a freezer, whereas the others do not.

An advantage of the indirect methods is that, because all of the 3-MCPD ester isomers are converted to 3-MCPD, the GC/MS signal is amplified greatly compared with what the signal would be for individual isomers. The validated methods are reliable for 3-MCPD, 2-MCPD, and glycidyl ester concentrations above 1 mg/kg. AOCS has also explored direct methods for 3-MCPD ester analysis, but "it has been a bit of an analytical nightmare," says Cantrill. "You would need a standard for each of the 63 potential isomers, and a lot of them are going to be present at low levels that are below the limits of detection." He adds that a direct method might be important if one of the isomers was found to be more toxic than others, but he thinks this is unlikely.

Now that researchers have reliable methods to quantitate 3-MCPD esters, they can focus their efforts on finding ways to mitigate the process contaminant. The challenge is to maintain product quality, including an oil's appearance, odor, flavor, and stability, while minimizing the formation of 3-MCPD esters. Although 3-MCPD esters arise as temperatures increase during deodorization, the critical step for reducing their formation is bleaching, said Roland Verhé, professor emeritus at Ghent University in Belgium, in a presentation at the 2015 AOCS Oils and Fats World Market Update in Dublin, Ireland. According to Verhé, the formation of 3-MCPD esters is so rapid beginning at 140 °C that controlling their formation during deodorization is not possible.

During the bleaching step of edible oil refining, natural or acid-activated clay is added to the oil to absorb colors and remove residual phosphatides, metals, and soaps. Hydrochloric acid can leach from activated bleaching earth, introducing chloride ions that are necessary for the formation of 3-MCPD esters. Therefore, using natural instead of acid-activated bleaching earth can greatly reduce the formation of these process contaminants. According to data presented by Verhé, using natural bleaching earth in a standard physical refining of palm oil produced only 1.25 mg/kg of 3-MCPD esters, compared with 4.21 mg/kg of 3-MCPD esters for activated bleaching earth. Chemical refining, which removes free fatty acids by reaction with sodium hydroxide and separation by centrifuge or gravity, rather than by high-temperature deodorization as in physical refining (Fig. 1, page 6), further reduced levels of 3-MCPD esters to 0.48 mg/kg, possibly because the sodium hydroxide treatment lowers free chloride ions. However, chemical refining is not a preferred option for palm oil, said Verhé, due to excessive oil loss.

Other possible strategies for reducing 3-MCPD formation include removal of precursors from the crude oil. Oils such as palm, coconut, and corn are especially susceptible to 3-MCPD ester formation because of their high content of DAGs. However, palm oils from different regions of the world and different processors can vary widely in 3-MCPD ester content. "The first important step to control 3-MCPD ester formation in palm oil is to achieve harvest at the most appropriate date, when the fruits are ripe but not overripe, and then to bring them as fast as possible to the processing plant," says Bertrand Matthäus of the Max Rubner-Institute in Detmold, Germany. "Normally after the ripe palm fruit bunches are cut from the tree, they lay for some hours before collection, and during this time enzymes degrade the triacylglycerides to diacylglycerides and monoacylglycerides." Bruising of the palm fruit upon falling to the ground increases contact between lipolytic enzymes and the palm oil.

In addition to changing harvesting practices, oil producers might be able to lower DAG content by breeding new oilseed species with lower activities of lipases—the enzymes that convert TAGs to DAGs and MAGs. Or they could add enzymes that transform DAGs back into TAGs. Washing crude oils to reduce chloride ions is another option, although this has been shown to have only a minor effect on 3-MCPD ester formation in palm oil, says Matthäus. Finally, researchers are exploring ways to remove 3-MCPD esters from refined oils, for example, by adding adsorbents.

GLYCIDYL ESTERS

Although related to 3-MCPD esters, glycidyl esters have distinct structures, formation mechanisms, physiological effects, and mitigation strategies. Like 3-MCPD esters, glycidyl esters originate from DAGs in the oil; however, glycidyl esters require much higher temperatures (above 230 °C) for formation. Glycidyl esters, which contain an expoxide ring (Table 1, page 7), form by the elimination of a fatty acid from a DAG through a radical intermediate. Depending on the identity of the remaining fatty acid, seven distinct glycidyl esters can be formed.

Glycidyl esters tend to be most abundant in refined palm oil because of its high DAG content (4–12%; mean, 6.5%) and the elevated temperatures (up to 270 °C) used in the deodorization step of physical refining. Oils with a high free fatty acid content, like palm oil, typically must undergo physical as opposed to chemical refining, though the latter uses a lower deodorization temperature. Very little glycidyl esters has been detected in other refined oils, which usually contain less than 2.5% DAGs and undergo chemical refining.

Similar to 3-MCPD esters, glycidyl esters are presumed to be digested within the human body to release free glycidol—a compound listed as "probably carcinogenic to humans" by the International Agency for Research on Cancer (IARC) based on its ability to induce tumors in numerous organs in rodents. In addition, glycidol is genotoxic, or capable of damaging DNA. In 2009, the BfR conducted an initial safety evaluation of glycidyl esters in foods, concluding that the risk to adults is low (Bakhiya, N. *et al.,* http://dx.doi.org/10.1002/mnfr.201000550, 2011). The BfR recommended that action be taken to lower the level of glycidyl esters in infant formula, but they did not find evidence of any immediate health risks for infants. Currently, there are no EU or US regulations for levels of glycidyl esters in edible oils.

The three AOCS-validated indirect methods for 2-MCPD and 3-MCPD ester analysis can also be used to quantify glycidyl esters. Two of the methods (AOCS Official Methods Cd 29a-13 and 29b-13) convert glycidyl esters to 3-monobromopropanediol (3-MBPD) prior to GC/MS analysis. The third method (Cd 29c-13) consists of two assays. In assay A, both 3-MCPD esters and glycidyl esters are converted to 3-MCPD in the presence of chloride. Assay B measures 3-MCPD in the absence of chloride, so that glycidyl esters cannot be converted to 3-MCPD. Glycidyl esters are then calculated from the difference of assay A (the sum of 3-MCPD and glycidol) and assay B (3-MCPD). In addition, in 2012 AOCS validated a direct method for glycidyl ester analysis that uses solid phase extraction and LC/MS (Method Cd 28-10). Using this method, researchers can quantitate glycidyl esters directly, without needing to convert them to other compounds.

Because high temperatures are required for glycidyl ester formation, optimizing the deodorization step of oil refining is critical. In palm oil, the production of glycidyl esters is minimal below 200 °C but increases rapidly above 230 °C (Craft, B. D. *et al.,* http://dx.doi.org/10.1016/j.foodchem.2011.10.034, 2012) (Fig. 2). Typically, physically refined palm oils are deodorized at temperatures of up to 270 °C for 3–6 hours. "For very low glycidyl ester levels, the deodorization temperature should be less than 230 degrees Celsius," says Gerrit van Duijn, a consultant in Vlaardingen, The Netherlands, and retired global oil processing technology director at Unilever. "This can only be achieved by chemical refining. However, especially in palm oil refining, this would lead to high oil losses and a high effluent cost."

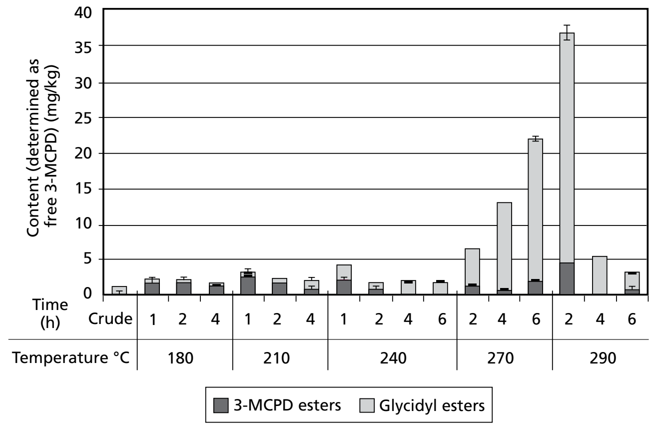


FIG. 2. Formation of 3-MCPD esters and glycidyl esters during deodorization of crude palm oil at different temperatures and times. Credit: *Processing Contaminants in Edible Oils,* AOCS Press

According to Matthäus, the deodorization step during physical refining should be conducted at as low a temperature or for as short a time as possible, while still removing volatile compounds and contaminants. "What you can also do is to introduce a two-step deodorization, where you start with a lower temperature for a longer time, and then you finalize the deodorization with a higher temperature for a very short time," he says.

A 2012 study by Craft *et al.* found large fluctuations in glycidyl ester levels according to the origin of commercial palm oil samples (range, 0.24–17.34 mg/kg; average 8.52 mg/kg) (http://dx.doi.org/10.1016/j.foodchem.2011.10.034). Surprisingly, there was no apparent correlation between total glycidyl ester content and DAG levels (Fig. 3a, page 10). However, when focusing on samples that originated from a single refinery, the team did find a strong positive correlation between glycidyl esters and DAG content (Fig. 3b, page 10). The researchers hypothesize that the large differences in glycidyl ester content among different suppliers reflects a range of temperatures used for deodorization.

Craft *et al.* also discovered a positive linear correlation between levels of free fatty acids and DAG in crude palm oil. This finding is important from a practical standpoint because the free fatty acid level is routinely measured at palm oil refineries as a means of quality control. To minimize glycidyl ester production, the researchers recommend an upper limit of 1.9–2.5% free fatty acids in crude palm oil, which corresponds to 3–4% DAG. As with 3-MCPD esters, any strategies that reduce the DAG content of crude oils should also reduce glycidyl ester formation. Also, it is possible that glycidyl esters could be removed from the refined oil with adsorbents or by degrading the esters into MAGs.

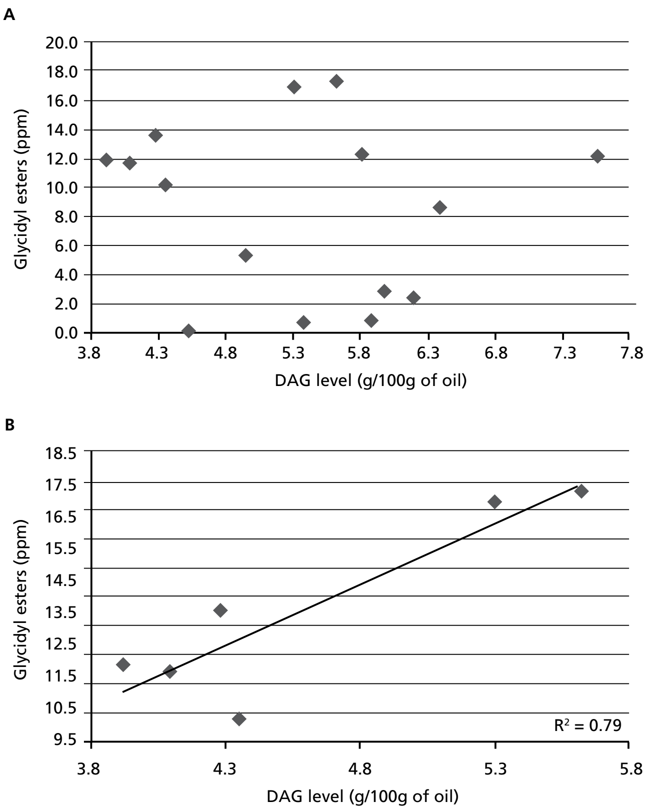


FIG. 3. (a) Relationship between glycidyl ester and DAG levels in palm oil and palm olein samples (n = 15). (b) Analysis of a subgroup of samples obtained from the same refinery (n = 6) shows a strong positive correlation (R2 ~0.8). Credit: *Processing Contaminants in Edible Oils,* AOCS Press

MINERAL OIL

"Mineral oil" is the common name for a complex mixture of hydrocarbons derived from petroleum. Considering only those compounds with fewer than 20 carbon atoms, there are more than 100,000 possible hydrocarbons in mineral oil (Lacoste, F., http://dx.doi.org/10.1051/ocl/2013060, 2014). Mineral oil compounds can be divided into two structural groups: mineral oil saturated hydrocarbons (MOSH), which include linear and branched alkanes and alkyl-substituted cycloalkanes; and mineral oil aromatic hydrocarbons (MOAH), which are alkyl-substituted polyaromatic hydrocarbons.

Sources of mineral oil contamination in edible oils include machinery used for harvesting and food processing. Harvesters have been shown to contaminate sunflower seeds with diesel oil at up to 3 mg/kg and with lubricating oil at up to 5 mg/kg (EFSA, http://dx.doi.org/10.2903/j.efsa.2012.2704, 2012). Oil mills use mineral oils for cleaning and maintenance of machinery, and the mineral oil can leak from stirring units or other machinery into the edible oil. Levels of mineral oil in edible oils produced in large quantities were found to be 10–30 mg/kg; in specialty oils produced in smaller amounts, 100¬–1,000 mg/kg (EFSA, http://dx.doi.org/10.2903/j.efsa.2012.2704, 2012). Usually, the source of mineral oil contamination in edible oils cannot be identified. In 2008, 100,000 metric tons of sunflower oil from the Ukraine was found to be contaminated with mineral oil at concentrations above 1,000 mg/kg. The composition of the mineral oil corresponded to that of base oil for manufacturing lubricating or hydraulic oils. The mineral oil may have been added fraudulently because mineral oil is cheaper than sunflower oil (Grob, K., http://dx.doi.org/10.1002/ejlt.200800234, 2008).

Mineral oil is arguably the largest contaminant in the human body, with mineral paraffins detected in human body fat on the order of 1 g (Grob, K., http://dx.doi.org/10.1002/ejlt.200800234, 2008). In animal and human studies, MOSH with carbon numbers C_{16}–C_{35} accumulate in tissues such as lymph nodes, spleen, and liver, where they may cause microgranulomas. There is little absorption of MOSH with carbon numbers above C_{35}. MOAH are more toxic than MOSH; in particular, MOAH with three or more aromatic rings may be mutagenic and carcinogenic. Edible oil producers typically use food-grade, or "white," mineral oil, which has a carbon number of C_{24}–C_{30} and is virtually free of MOAH, for approved applications such as dust suppression in oilseeds. "The toxicity of these food-grade oils is not the same as mineral oils used as lubricants," says Lacoste. "Lubricants are generally not food-grade, so they have some aromatic components, and the toxicity is much higher."

Mineral oil contamination of edible oils can be detected in a GC chromatogram as a large hump of an unresolved complex mixture, with a few spikes from naturally occurring plant alkanes. In 2015, Lacoste and her colleagues published an International Organization for Standardization (ISO) method to determine the levels of different MOSH (C_{10}–C_{56}) in vegetable oils (ISO 17780). The rapid method used a two-step procedure: fractionation on silica gel impregnated with silver nitrate, followed by GC analysis with internal standards. Lacoste is also leading a project sponsored by the European Committee for Standardization (CEN) to quantify both MOSH and MOAH in vegetable oils. "This work, a collaborative study involving 12 laboratories, is now at the stage of a draft method," says Lacoste. "The method, which uses online HPLC-GC-FID, would be applicable at 10 mg/kg for saturated hydrocarbons and 10 mg/kg for aromatic hydrocarbons." The draft method must still be approved by a vote at the level of CEN, she says.

According to van Duijn, contamination of edible oils with mineral oil can largely be avoided by "good housekeeping"—maintaining plant machinery and guarding against leakage of lubricants or hydraulic fluids. For mineral oil in direct contact with food, white oils should be used. Fortunately, much of the mineral oil that does find its way into edible oils is removed during the refining process. Gasoline and diesel can be removed during neutralization and bleaching, and mid-fraction mineral oil (C_{20}–C_{35}) is volatilized in the deodorization step. However, heavy-fraction mineral oil, such as grease and hydraulic oils, is not removed during refining and remains in the oil.

PHTHALATES

Phthalates—esters of phthalic acids with different alcohols—are used to soften and increase the flexibility of plastics and vinyl. The most common phthalate is bis(2-ethylhexyl) phthalate (DEHP), which accounts for 50% of world production. Because phthalates are not chemically bound to the plastic or vinyl polymer, they can migrate from plastic materials into fatty foods. "Phthalates are very lipophilic, and plastic tubing may contain more than 20% of phthalates," says Lacoste. "So the migration of phthalates into edible oils could be huge." Phthalates are suspected endocrine disruptors that cause reproductive problems in animal studies.

Regulations in the European Union and the United States prohibit the use of phthalates in plastics that contact fatty foods. "The big oil refiners know about this," says Cantrill. "It may be a problem for small refiners or producers who do not understand some of the risks involved in not using food-grade plastics." The highest levels of phthalates (more than 1 mg/kg) have been detected in walnut, virgin olive, and grapeseed oils.

To analyze phthalates in vegetable oils, ITERG uses a direct method based on solid-phase microextraction followed by GC/MS, Lacoste says (http://dx.doi.org/10.1051/ocl/2013060, 2014). The method rapidly and sensitively detects levels of several phthalates, including DEHP, at limits lower than 0.1 mg/kg. Deodorization is the most important refining step for the removal of phthalates from edible oils. In a 2008 ITERG study, chemical refining removed 19–87% of phthalates, depending on the molecular weight, whereas the higher-temperature deodorization used in physical refining totally eliminated the three studied phthalates from the edible oil (http://dx.doi.org/10.1051/ocl/2013060, 2014).

A BALANCING ACT

In general, the deodorization step of edible oil refining appears to be the most critical to optimize in order to mitigate process contaminants. "What every refiner has to do is find the balance between the removal of harmful contaminants and the formation of new contaminants," says Matthäus. "And that is the reason why the deodorization temperature should not be too low, say below 240 degrees Celsius."

"There's really a difficult choice among all these contaminants," agrees Lacoste. She says that researchers are investigating approaches such as two-step deodorizations to solve this problem. However, for some contaminants such as long-chain mineral oils (C_{35}–C_{40}), "at the moment, we don't how to eliminate them from the vegetable oil," she says.

Van Duijn says that process contaminants could be reduced if more variation in product specifications was allowed, depending on the function of the oil or fat in the food. "Why should the free fatty acid content of seed oils used for cold applications always be below 0.1%?" he asks. Van Duijn notes that for extra virgin olive oil, the limit for free fatty acids is 0.8%. "A higher free fatty acid limit would allow a lower deodorization temperature," he says. Van Duijn also questions why palm oil should be white if it is destined for spreads in which carotenoids are used for coloring. Less stringent bleaching conditions (in other words, using natural instead of acid-activated bleaching earth) would limit the production of 3-MCPD esters. "The technical challenge is to introduce this flexibility to refineries without increasing costs," van Duijn says.

Matthäus agrees that financial considerations play a large role in any proposed changes to edible oil refining. "In the last eight or nine years, a lot of research has been done, and there are a lot of approaches usable to reduce the amount of process contaminants in the final product," he says. "The main problem is that no one is willing to pay for higher-quality oils, so the levels of process contaminants have not really been reduced in recent years."

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A deeper look at MCPD and glycidyl esters

Detailed information about the formation of monochloropropanediol (MCPD) and glycidyl esters, their detection and quantitation, toxicological properties, and successful mitigation strategies for decreasing their concentrations in edible oils can be found in *Processing Contaminants in Edible Oils: MCPD and Glycidyl Esters,* 1st edition, available at http://bit.ly/3MCPD.

Pulsed electric field: groundbreaking technology for improving **olive oil extraction**

Eduardo Puértolas, Saioa Alvarez-Sabatel, Ziortza Cruz

Olive oil is a key component of the Mediterranean diet, and is recognized worldwide for its organoleptic and healthful properties. Extra virgin olive oil (EVOO) and virgin olive oil (VOO), the top categories according to world olive oil council standards, are only obtained by "cold" mechanical methods that preserve their characteristics. Industrial extraction of these premium olive oils basically involves three phases: (1) the crushing of the fruit to break plant tissues and release the oil, (2) the malaxation (slowly churning) of the milled olives to assist coalescence of the oil drops (typically at <27 °C, < 1h), and (3) the mechanical recovery of the oil by centrifugation (continuous mode; industrial process) or pressing (discontinuous mode; traditional-artisanal process). Afterwards, the olive oil is usually filtered or decanted to remove any possible solid residues, and finally bottled and marketed. While small mills process between 300 and 3,000 kg of olives per hour, the largest ones process up to about 12,000 kg of olives per hour and per each production line.

- **Pulsed electric field (PEF) has been proposed to increase olive oil extraction yields, while increasing the concentration of health-related molecules and consistently maintaining the highest oil quality.**
- **In some conditions, this technology would also allow the malaxation temperature to be reduced, preserving yields and potentially even improving sensory quality.**
- **Scaling up for industrial purposes is already possible, and the first pilot study at 520 kg/h has been conducted. The potential benefits of PEF are highlighted and quantified in this article.**

BIG OPPORTUNITIES TO IMPROVE YIELDS IN HIGH-QUALITY OLIVE OIL PRODUCTION

One of the most important industrial handicaps of VOO and EVOO production is the low efficiency of current mechanical extraction techniques. Although efficiency depends on the ripeness and variety of the olives and milling parameters (among other factors), typically only 80% of the oil present in the fruit is recovered by physical methods. The rest (20%) of the oil remains in the olive waste generated at the end of the process (Puértolas, *et al.,* http://dx.doi.org/10.1016/j.foodres.2015.12.009, 2016). Additionally, significant amounts of bioactive compounds like polyphenols are also lost with the olive waste. To recover as much oil as possible, the most widespread solution in oil mills is to increase malaxation time and/or temperature, or to run a second extraction cycle with the olive waste at high malaxation temperature. Such practices have a negative impact on chemical and sensory parameters, so the resulting olive oils are of low quality and category (neither EVOO nor VOO).

Pulsed electric field (PEF) is an emerging physical technology that has been largely studied for improving mass transfer processes in the food industry (Puértolas, *et al.,* http://dx.doi.org/10.1146/annurev-food-022811-101208, 2012). PEF pretreatment of crushed olives (olive paste) prior to centrifugation constitutes a great opportunity for improving extraction of VOO and EVOO, and increasing oil yield and bioactive compounds recovery—polyphenols recovery, for example (Puértolas, *et al.,* http://dx.doi.org/10.1016/j.foodchem.2014.07.029, 2015).

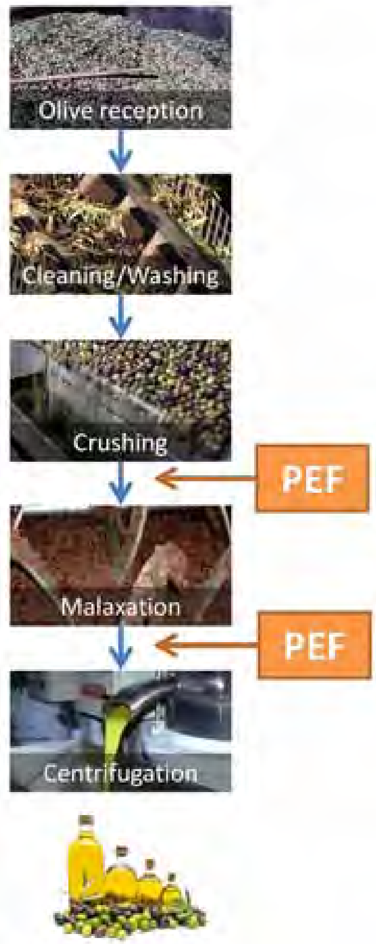


FIG. 1. Application of pulsed electric field (PEF) in the conventional production outline of olive oil

PULSED ELECTRIC FIELDS: FUNDAMENTALS AND KEY ASPECTS

PEF-assisted extraction essentially involves the discharge of direct current electric pulses of short duration (µs-ms) and high voltage (up to 50 kV) into a soft plant material located between at least one high-voltage electrode and one ground electrode (Puértolas, *et al.,* 2016). These electric pulses generate a pulsed electric field (typically up to 10 kV/cm), which produces the formation of pores in the cell membranes of plant tissues (Puértolas, *et al.,* 2012). This process, called electroporation, enhances the diffusion of solutes through the cell membranes, favoring the recovery of intracellular substances, including oil and molecules of interest. Furthermore, the application of electric fields has also been described *"per se"* as an effective demulsification technique, since electric fields facilitate coalescence processes and consequent separation of oil from water. Therefore, PEF could improve olive oil yields by a double mechanism: the improvement of oil extraction from olive tissue, and the releasing of olive oil trapped in vegetable oil-water emulsions. It is important to note that PEF is considered a "cold technology," as the conventionally used intensities do not cause a notable increase in the temperature (normally less than 5 °C). This is absolutely essential to preserve the valued organoleptic properties of the best olive oils (EVOO and VOO).

Fig. 1 summarizes the different stages of olive oil industrial production and the points where PEF has been proposed to be applied: (1) after malaxation, to facilitate the demulsification of the oil and also to improve mass transfer during the subsequent centrifugation, and (2) just after crushing. In this last case, PEF is mainly applied to enhance the subsequent release of oil during malaxation based on the electroporation of the cell membranes (Abenoza, *et al.,* http://dx.doi.org/10.1007/s11947-012-0817-6, 2013). Applying 30 minutes of malaxation at low temperature (15 °C), these authors achieved a 14.1% higher oil yield using PEF pretreatment (2 kV/cm), compared to the control process. Furthermore, the extraction yield was similar to that obtained for the control process at high temperature (26 °C). Therefore, PEF technology could help reduce maceration temperature from 26 to 15 °C, without affecting the extraction yield. Processing at this low temperature prevents the loss of volatile compounds which are essential for sensory quality, so the olive oil recovered by this innovative method could potentially present better organoleptic properties than those obtained using higher malaxation temperatures.

FIRST PILOT TRIAL: A SUCCESSFUL STORY

Recently, a pilot study in a small olive oil mill was conducted in Spain, demonstrating the potential of the technology at close to industrial scale (Puértolas *et al.,* 2015). A PEF unit designed and constructed by KEA-TEC (Waghäusel, Germany) was used to conduct the study. This PEF-unit included an in-line treatment chamber (tube configuration) specifically constructed to be easily incorporated into an olive oil production line. To conduct the trials, this treatment chamber was integrated into a continuous production plant (K30, Oleomio, Granada, Spain), just after the malaxation. This pilot system allowed 520 kg of olives to be processed per hour, applying electric fields of 2 kV/cm and 65 J (total specific energy applied: 11.25 kJ/kg).

In the control batch, the extraction yield was 20.0 kg of oil per 100 kg of processed olives; in the PEF-assisted extraction batch it was 22.7 kg/100kg. Based on these measurements, the PEF technology allowed us to improve the oil extraction yield by 13.3% compared to the control. Correspondingly, the PEF olive oil had higher concentrations of polyphenols, phytosterols, and total tocopherols than the control olive oil did (11.5%, 9.9%, and 15% higher, respectively). Chemical and sensory analysis showed that both control and PEF oils presented the highest quality according to the legal standards of the European Union, reaching the extra virgin olive oil (EVOO) category.

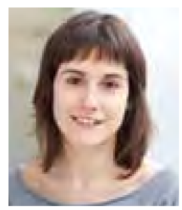
INDUSTRIAL FEASIBILITY, COST, AND POTENTIAL ECONOMIC BENEFITS

Although industry is not yet using PEF to improve vegetable oil recovery, the technology is being used commercially for other purposes. Full-scale PEF units are now operative in several food companies for other applications, such as replacing thermal blanching during potato snacks production or inactivating pathogen and spoilage microorganisms in juices and related products. Based on these applications, and taking into account the costs of electricity needed to improve oil recovery, the cost of an industrial PEF unit for this application could be estimated to be between $50,000 and $200,000, depending on the production rate. In addition, the electricity costs of the treatments would be about $0.33 per metric ton (MT) of processed olives (Puértolas, *et al.,* 2016).

In VOO and EVOO production without PEF, approximately 4 kg of oil per each 100kg of processed olives remain in the olive waste, resulting in a great monetary loss for the olive oil producers. According to the results obtained in the pilot trials, a PEF treatment could potentially help recover 50% of this residual oil. In an industrial oil mill operating at 10,000 kg/h (16h/day), a PEF treatment could potentially increase the VOO/EVOO daily production to 4,326 kg (from 32,000 to 36,320 kg). Assuming a VOO/EVOO price of $3–$6 per kg (according to pricing data from the International Olive Council), these figures mean a potential extra turnover of between $12,978 and $25,956 per production day. This would easily offset the electricity cost of the treatments ($0.33/MT) and pay back the capital cost of the PEF unit in a short time. Moreover, the potential increase in the concentration or bioactive compounds, such as polyphenols or phytosterols, could have also have a positive impact on the final oil price. In any case, more pilot and pre-industrial trials and precise economic studies should be conducted to obtain more data at near to industrial levels.

Eduardo Puértolas, who earned a Ph.D. in Food Science and Technology, is co-author of more than 40 publications (including 28 peer-reviewed papers) and has participated in more than 30 public and private projects related to emerging food technologies. He can be contacted at epuertolas@azti.es.

Saioa Alvarez-Sabatel, who earned a Ph.D. and an MsC in Food Quality and Safety, devotes her research to the impact of emerging food processing technologies on food microstructure, with special focus on food emulsions and ingredients technological functionality. She can be contacted at salvarez@azti.es



Ziortza Cruz, who earned a BSc in Food Science and Technology and an MSc in Pharmacology, has been involved in more than 50 R&D and transfer projects for the food industry. Her research field is mainly focused on emerging processing technologies for preservation and the development of new food products. She can be contacted at zcruz@azti.es

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Health effects of **individual saturated** fatty acids

During the 2015 AOCS Annual Meeting and Industry Showcases, leading researchers discussed the health effects of saturated fatty acids, particularly with respect to the updated *2015–2020 Dietary Guidelines for Americans*; recent findings regarding health effects of saturated fatty acids in dairy foods; and possible health benefits of substituting stearic acid for cholesterol-raising saturated fatty acids, such as lauric, myristic, and palmitic acids. This article summarizes their discussion.

J. Edward Hunter, Fabiola Dionisi, and Mathilde Fleith

Leading researchers discuss:

- the health effects of saturated fatty acids and the draft *2015–2020 Dietary Guidelines for Americans* (1);
- recent findings regarding health effects of saturated fatty acids in dairy foods; and
- possible health effects of substituting stearic acid for cholesterol-raising saturated fatty acids (lauric, myristic, and palmitic acids).

2015–2020 DIETARY GUIDELINES FOR AMERICANS

Two members of the Dietary Guidelines Advisory Committee (DGAC)—Frank Hu, professor of nutrition and epidemiology and professor of medicine at Harvard T.H. Chan School of Public Health (Boston, Massachusetts, USA) and Tom Brenna, professor of human nutrition and chemistry at Cornell University (Ithaca, New York, USA)—noted that the draft of the updated version of the *Guidelines* focuses its recommendations on the whole diet with emphasis on healthy food-based dietary patterns. In contrast, previous versions of the *US Dietary Guidelines for Americans* placed more emphasis on individual nutrients rather than on specific foods. This new focus on foods reverses nearly four decades of nutrition policy that prioritized single nutrient approaches rather than the whole diet.

Specifically, the DGAC draft recommended increased intakes by the US population of vegetables, fruits, whole grains, and low- or non-fat dairy foods. On the other hand, the DGAC draft recommended reduced intake of red and processed meats and lower intake of foods high in refined grains and added sugars. Regarding dietary fats, the Committee concluded that optimizing fat quality is more important than reducing total fat intake.

The recommendations to eat less "red and processed meats" and "foods high in refined grains" were not retained as such in the final version of the *2015–2020 Dietary Guidelines for Americans* (1), released in January 2016. The mentioning of these foods was replaced by nutrient guidelines. The "Key Recommendations" in Chapter 1 of the *2015–2020 Dietary Guidelines* state: "A healthy eating pattern limits: saturated fats and trans fats, added sugars, and sodium" (1).

Consistent with the American Heart Association, the DGAC recommended that the general population consume less than 10% of total calories from saturated fat per day and that sources of saturated fat be replaced with unsaturated fat, particularly polyunsaturated fatty acids. The Committee encouraged the food industry to continue reformulating and making changes to certain foods to improve their nutritional profiles. One example would be to achieve a better saturated fat to polyunsaturated fat ratio.

Hu discussed epidemiological evidence relating individual saturated fatty acids (SFAs) and risk of coronary heart disease (CHD). For example, in the Nurses' Health Study (a large epidemiological study) cohort, the dietary intake of short- to medium-chain SFAs (4:0–10:0) was not associated with CHD risk, whereas longer chain SFAs (12:0–18:0) were each associated with a non-significantly increased risk of CHD. Substitution of longer-chain SFAs with polyunsaturated fatty acids (PUFAs) has been associated with significantly lower risk of CHD. Additionally, very long chain SFA (20:0, 22:0, and 24:0) levels in plasma and erythrocytes have been associated with favorable

profiles of blood lipids and other CHD risk markers, such as fasting insulin and C-peptide levels.

According to Brenna, too little attention has been focused on atherogenic properties that might be induced during fat processing. He noted that partial hydrogenation of vegetable oils produces more changes in the oil than simply forming trans isomers, such as elaidic acid. Positional isomers (both cis and trans) of unsaturated fatty acids and stearic acid also are produced. He indicated that in a recent hamster study, effects of a partially hydrogenated vegetable oil differed from those of elaidic acid, the major trans monoene in most partially hydrogenated oils, or vaccenic acid, the major trans monoene of dairy fat. Brenna believes there is limited evidence for benefits of the monounsaturated fatty acid oleic acid and that it is best considered a neutral fatty acid that is among the most prominent in most human diets. He discussed branched chain fatty acids, a neglected bioactive series of mostly saturated fatty acids that are prominent in dairy and beef. His take-home message for the food industry was to increase the use of healthful ingredients such as nuts, fruits, vegetables, and legumes, and to study how processing affects levels of trace fatty acids and generates other potentially bioactive compounds.

SATURATED FATTY ACIDS IN DAIRY FOODS

Dairy foods are a major source of dietary SFAs, and consumption of low-fat instead of high-fat dairy products is often recommended by health professionals. This is because of the high SFA content of dairy fat and the association of SFAs with adverse effects on cardiovascular health. Additionally, whole-fat products contribute higher calories than low-fat products.

In discussing the topic of dairy fat and cardiometabolic health, Dariush Mozaffarian, Dean of the Friedman School of Nutrition Science and Policy at Tufts University (Boston, Massachusetts, USA), noted that the role of dairy products, particularly dairy fat, in cardiovascular health is controversial. On one hand, low-fat dairy consumption has been associated with reduced risk of hypertension and stroke. On the other hand, accumulating evidence suggests that dairy fat (such as the fat in cheese and yogurt) may be beneficial for type 2 diabetes by reducing insulin resistance. Further research is needed to establish whether specific fatty acids, such as odd-chain and medium-chain SFAs present in dairy fats, may have physiological benefits.

Additionally, Mozaffarian noted that in a US study in adults, low-fat or whole-fat milk intake was not significantly associated with long-term body weight change. In children, however, most observational studies have found that low-fat milk intake was associated with greater weight gain than was whole-fat milk intake. Mozaffarian concluded that low-fat dairy foods were no better or worse than full-fat dairy foods with respect to their effects on risk of cardiovascular disease (CVD).

Dairy fat contains trans fatty acids (TFAs) at a level of about 3% of total fatty acids. The impact of dairy TFAs compared to industrially produced TFAs is not well understood at present. TFAs are not a principal target of the 2015 DGCA recommendations because total TFA intake in the US population (aged 2 years or more) currently is believed to be low (about 0.5% of energy) (2). There is general agreement among health professionals that intake of industrially-produced TFAs from partially hydrogenated oils should continue to be reduced.

POSSIBLE HEALTH BENEFITS OF SUBSTITUTING STEARIC ACID FOR OTHER SATURATED FATTY ACIDS

It was pointed out that in the typical US diet, stearic acid is the second most common SFA after palmitic acid, with an average consumption of about 3% of daily energy. The main sources of stearic acid are the same as those of other commonly consumed SFAs (lauric, myristic, and palmitic acids), namely, meat, fish, eggs, and dairy. Fats rich in stearic acid include butterfat (12% of total fatty acids), cocoa butter (34%), and mutton and beef tallow (19%).

Ed Hunter, adjunct professor of chemistry at Xavier University (Cincinnati, Ohio, USA) noted that effects of stearic acid on CVD risk factors may be different from those of other SFAs as well as from those of unsaturated fatty acids and TFAs. He discussed studies in which stearic acid was substituted for other fatty acids (Table 1). Compared with cholesterol-raising SFAs, stearic acid lowered LDL cholesterol but was neutral with respect to HDL-C and to the ratio of total to HDL-C. Compared with unsaturated fatty acids, stearic acid tended to raise LDL-C, lower HDL-C, and increase the ratio of total to HDL-C, however these directional changes were not statistically significant. In four studies, one-to-one substitution of stearic acid for TFAs showed a decrease or no effect on LDL-C, an increase or no effect on HDL-C, and a directional decrease in the ratio of total to HDL-C. Three studies showed increased plasma fibrinogen when dietary stearic acid exceeded 9% of energy.

Considering the results, the experts agreed that stearic acid would be a good substitute for cholesterol-raising SFAs (lauric, myristic, and palmitic) and TFAs for solid fat applications. Research is needed on effects of stearic acid on emerging cardiovascular disease risk factors, such as fibrinogen, and to understand responses in different populations.

TABLE 1. Effects of substituting stearic acid for other dietary fatty acids

Parameter	Stearic Replacing Other SFAs	Stearic Replacing Unsaturated FAs	Stearic Replacing TFAs
LDL-cholesterol	↓	≅	↓ or ≅
HDL-cholesterol	≅	≅	↑ or ≅
Total/HDL-C ratio	≅	≅	↓ or ≅
Expected impact on CV health	Favorable	Unfavorable	Favorable

OTHER TOPICS

Philip Calder, a professor of nutritional immunology within medicine at the University of Southampton (UK), discussed SFAs in relation to inflammation. He noted that in contrast to eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), which are known to modulate the inflammatory process and actively initiate resolution of inflammation, the effects of SFAs in inflammation are less well established. *In vitro* studies have indicated that lauric, myristic, and palmitic acids can directly activate inflammatory cells. Macrophages taken from mice fed high fat diets rich in SFAs (lard or hydrogenated coconut oil) have shown exaggerated inflammatory cytokine responses (TNF-alpha gene expression and production) when stimulated with bacterial endotoxin. This effect may relate to altered membrane composition. Calder noted that it is uncertain whether SFAs trigger inflammatory responses. He suggested that they might increase susceptibility to inflammatory responses to endotoxin.

Brent Flickinger, a vice president at Archer Daniels Midland Company (Chicago, Illinois, USA) provided perspective about how food ingredient manufacturers consider replacing TFAs with SFAs in some foods needing solid fat structure. He noted that the process of interesterification, which causes a random rearrangement of the fatty acids on the glycerol backbone, can be used to prepare fats that are suitable for solid fat applications, such as margarines, spreads, or baking shortenings. In contrast, the use of fully hydrogenated soybean oil as a trans-fat replacement for such products could create undesirable textural properties, such as hardness or waxiness. Several human studies have shown no significant effects of interesterified fats on blood lipid and lipoprotein parameters. Overall, manufacturers of fat-containing products need to balance the supply of edible oils with functional and nutritional considerations of edible fats and oils ingredients.

The conclusion of the authors is that saturated fatty acids in foods can have unfavorable health effects if consumed at excessive dietary levels. In recognition of this relationship, the *2015–2020 Dietary Guidelines for Americans* maintains the recommendation that SFA intake not exceed 10% of total energy (1). On the other hand, it should be recognized that foods tending to be high in SFA (e.g., meats and dairy products) also can be important sources of energy and of essential nutrients, such as high quality protein, calcium, iron, and zinc. Physiological effects of SFA may depend, in part, on their chain lengths. In general, moderation is appropriate in considering dietary intakes of foods containing SFA.

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This report was prepared by the coauthors. The other session participants had the opportunity to review the draft and to offer comments and suggestions. This article should not imply endorsement or approval by the session participants.

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Professional Pathways

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

Jim Kenar is a research chemist at the US Department of Agriculture–National Center for Agricultural Utilization Research (USDA–NCAUR) in Peoria, Illinois, USA. Jim is an active member of AOCS and currently a senior associate editor for the *Journal of the American Oil Chemists' Society (JAOCS).* He has been selected to succeed Richard W. Hartel as the journal's next editor-in-chief starting in May, 2016. Jim's research interests focus on the modification and utilization of agricultural materials (vegetable oils and polysaccharides) for food and nonfood applications.

1. Why did you join AOCS?

I joined AOCS in 2000 at the suggestion of NCAUR colleagues as a way to present research and network. I quickly realized that AOCS provides a good forum to interact with professionals and keep up to date on scientific perspectives in fats and oils, and over the years it has allowed me an avenue to contribute to the Society.

2. Describe your career path.

After obtaining my bachelor's degree in chemistry from Bradley University in Peoria, Illinois, I obtained my Ph.D. in 1994 in physical organic chemistry from the Johns Hopkins University, Baltimore, Maryland, USA. My doctoral research focused on reactions of highly reactive carbenes (divalent carbon species). This interest in reactive intermediates led me to Dr. Ned Porter's laboratory at Duke University where, as a postdoctoral research associate, I studied mechanistic aspects of lipid hydroperoxides that are formed during oxidation. In 1996, I joined the US Naval Surface Warfare Center, Indian Head Division, as a federal employee conducting research and development on novel energetic materials. Three years later, I joined NCAUR, a USDA Agricultural Research Service (ARS) research center. My research initially focused on the chemical modification and applications of vegetable oils and their derivatives. More recently, it has involved examining interactions of fats, oils, and their derivatives with starch and other polysaccharides.

3. What do you love about your job?

I enjoy the flexibility to pursue various lines of research within the scope of our bigger research project, and coming up with solutions to some of the difficult problems we face.

4. How do you see the industry changing in the next five years?

Society needs practical answers to complex problems that are difficult to solve from the viewpoint of one scientific discipline. Collaboration and approaches from several scientific and other disciplines will be necessary to successfully overcome these problems. Also, computers, the internet, social media, and big data are useful tools that have dramatically changed the way scientists search and share scientific information. Being able to sort, filter, and use this information effectively will be very important in the future.

5. Describe memorable job experiences.

My most memorable job experiences have always come when I have helped others succeed.

6. Please describe a course, seminar, book, mentor, or speaker that has inspired you in ways that have helped you advance your career.

My wife, Kelly, is always an inspiration to me. My graduate advisor, Dr. Alex Nickon, is a mentor who shaped my thinking about chemistry and taught me the importance of writing well. As a member of the emergency response/hazmat team at NCAUR since 2001, I take yearly hazmat training. This training has not only taught me the importance of preparation but has also helped sharpen my communication and leadership skills, as making crucial decisions in potentially extreme situations can be challenging.

CONTINUED ON PAGE 22

Micronutrient loss during crushing and refining processes

Jennifer Régis, Florent Joffre, and Frédéric Fine

Tocopherols, phytosterols, and coenzymes Q are micronutrients naturally contained in oilseeds such as rape, soybean, and sunflower. Although they are minor constituents, these compounds are considered positive for health and play a major role in preventing lipid oxidation. However, their concentration in edible oils can vary significantly based on genetic factors, cultivation, and storage conditions. In addition, their amounts in a vegetable oil depend strongly on the industrial processes used to extract the oil, especially the processes that are used during crushing and refining.

- Micronutrients are compounds naturally found in oilseeds. Though minor constituents, these compounds are considered positive for health and play a major role in preventing lipid oxidation.
- Unfortunately, studies indicate that the crushing and refining processes have a negative impact on the micronutrient content of oils.
- This review describes the influence of different factors on micronutrient concentration and proposes alternative methods to improve their yield and preservation.

RAPE, SUNFLOWER, AND SOYBEAN SEEDS: NATURAL SOURCES OF MICRONUTRIENTS

Vegetable seeds contain micronutrients with properties that are both varied and useful.

Tocopherols, formed of a chromanol ring and a hydrophobic phytyl chain (Table 1), are known for their positive role in protecting human health and for their ability to inhibit lipid oxidation, depending on their allotropic form.

Sterols, composed of a 1,2-cyclopentano-phenanthrene skeleton with a hydroxyl group on C3, an alkyl chain on C17, and usually a double bond on C5 (Table 1), are well known for their cholesterol- and cardiovascular risk-lowering properties, and their role in membrane fluidity and mobility.

Phenolic compounds such as sinapine or vinylsyringol (Table 1) are a group of substances that contain at least one aromatic ring and an alcohol group. These compounds, which are responsible for giving products their color and flavor, are minor in canola oil, whereas rape seeds contain them in significant amounts. Hence, it appears that the traditional oil extraction process is inimical to these micronutrients.

Coenzymes and tocopherols possess similar properties, although their chemical structures are completely different (Table 1). Coenzymes Q9 and Q10 also play a role in respiratory chain activity.

THE DISAPPEARANCE OF MICRONUTRIENTS DURING THE CRUSHING PROCESS

Studies have been conducted to demonstrate the impact of the crushing process on the amounts of micronutrients in rapeseed, soybean, and sunflower oils, and to investigate their fate.

Researchers have not usually studied the influence of crushing processes on tocopherol contents in crude oils. However, according to Chu and Lin [1], pre-treatments applied before the extraction process have an impact on tocopherol content during the processing of soybean seeds.

No crushing parameters influencing the concentration of sterols have been identified in the literature. However, results obtained by Mouloungui et al. and Roche et al. [1] suggest that rapeseed oil contains 1.7 times more sterols than sunflower oil, and soybeans contain 2.2 times more sterols than soybean oil.

Szydlowska-Czerniak et al. [1] found that an increase in temperature during the rapeseed conditioning phase had a beneficial effect on the amounts of total phenolic compounds in its crude oil.

Finally, data on coenzymes are scant. We note, however, that the highest level of coenzymes Q9 and Q10 in crude soybean oil was recorded by Pregnolato *et al.* [2] and Rodriguez-Acuna et al. [3]. Sunflower and rapeseed oils contained almost exclusively coenzymes Q9 and Q10, respectively.

THE DISAPPEARANCE OF MICRONUTRIENTS DURING THE REFINING PROCESS

Through four steps, namely degumming, neutralization, bleaching and deodorization, the refining process removes minor constituents from oils to meet consumer demand.

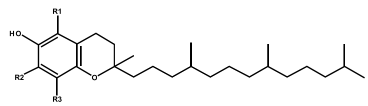
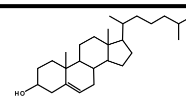
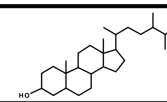
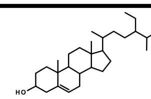
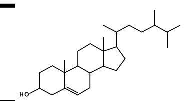
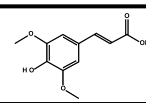
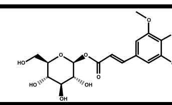
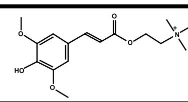
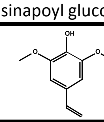
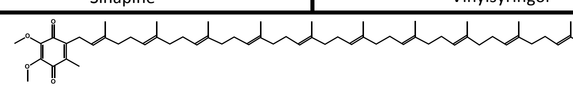
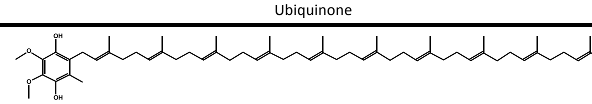
Micronutrient	Chemical structure	
Tocopherols		With R1=R2=R3=CH3 for α-tocopherol With R1=CH3, R2=H, R3=CH3 for β-tocopherol With R1=H, R2=R3=CH3 for γ-tocopherol With R1=R2=H, R3=CH3 for δ-tocopherol
Sterols	 Cholesterol	 Stigmasterol
	 B-sitosterol	 Campesterol
Phenolic compounds	 Sinapic acid	 1-O-β-D-glucopyranosyl sinapate (sinapoyl glucose)
	 Sinapine	 Vinylsyringol
Coenzymes Q9 and Q10	 Ubiquinone	
	 Ubiquinol	

TABLE 1. Chemical structures of micronutrients present in rape, soybean and sunflower seeds

Unfortunately, this step also causes some degree of micronutrient loss, depending on the variety of oil.

It has been demonstrated that the deodorizing step reduces the quantity of tocopherols by about 25% in both soybean and rapeseed oils [1]. Cmolik *et al.* [1] suggest that this decrease is correlated with the deodorization temperature. For sunflower oil, neutralization and deodorization were the most destructive steps, with losses of up to 16.3% and 20.3%, respectively [1].

Ferrari *et al.* and Gutfinger and Letan [1] have shown that most of the sterols are removed during bleaching and deodorization. The sterol content in bleached rapeseed oils fell by 18.5% versus 39.6% in deodorized rapeseed oils. The relationship between deodorization temperature and loss of sterols in sunflower oil was established by Jawad *et al.* [1].

Ghazani [4] observed that the degumming and neutralization steps caused most of the phenolic compound loss in rapeseed oil. Losses of 66% and 94.5%, respectively, were noted during these two stages.

However, according to Pregnolato *et al.* [2], refined sunflower oils were enriched by 46% in coenzymes Q9 and by 48% in coenzymes Q10.

POTENTIAL PROCESSES FOR THE FUTURE

As seen above, crushing and refining have a negative impact on micronutrient values in oils. Alternative processes are therefore being tested. Ghazani [4] has found that the substitution of sodium hydroxide by new alkaline agents, such as magnesium oxide or calcium hydroxide, during the neutralization step reduces total losses of tocopherols and sterols. Results obtained following the use of heat treatments also seem encouraging, as they show a better preservation of nutritional elements [1].

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Further reading

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CONTINUED FROM PAGE 19

7. Do you have any advice for young professionals who are trying to develop an effective network of other professionals?

Become involved in a professional society. Don't just attend meetings but present your research and actively seek out ways to be involved and contribute to societal activities. Always keep learning (personally and professionally), seek out mentors and opportunities to network, and expand your horizons.

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

I would take classes in materials science and biotechnology to become more multi-disciplinary. There is interesting research that takes place at the interface between organic chemistry, materials science, and biotechnology, and being able to understand and navigate between these disciplines is important in solving complex problems.

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

Opportunities for advancement really depend on what you desire out of your career and life. Advancement opportunities exist everywhere but you need to be looking for them with the proper attitude. I can't remember where I found it, but as a reminder to myself I have a quote on my wall, "Whiners and complainers see the world through crap-colored glasses."

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

Regardless of being well-rounded or a specialist, being able to work, cooperate, and communicate effectively with others is essential. Both attributes are valuable; one allows you to see a bigger picture while the other enables you to work out the little details. Whether you gravitate toward one end of the spectrum or the other, you must recognize your strengths and be able to surround yourself with others who compliment your skill set. Besides, who says that in being well-rounded you cannot also specialize?

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

In my case, completing a graduate degree allowed me a certain level of independence and opportunities to pursue research interests that might not have been possible otherwise. I learned much about myself and science during the endeavor. Whether you pursue a graduate degree or not, much like a professional athlete, investing time into your craft takes commitment, hard work, and a lot of hustle.

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The bonding of chemical giants

Laura Cassiday

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In the early days of December 2015, rumors were flying about a possible merger between two of the world's largest and best-known chemical companies, Dow (Midland, Michigan, USA) and DuPont (Wilmington, Delaware, USA). When the companies confirmed the merger, hailed by some as "the deal of the century," on December 11, experts immediately began speculating about the potential impact on employees, investors, and the chemical industry as a whole. Although many questions remain unanswered, details about the companies' merger and subsequent restructuring suggest both opportunities and challenges for those affected by the deal.

The newly formed company, DowDuPont, will have a combined value of more than $130 billion and about 100,000 employees worldwide. The deal is expected to close in the second half of 2016, pending approval by regulatory agencies and by Dow
and DuPont shareholders. Eighteen to 24 months after closing the deal, DowDuPont will separate into three publicly traded spin-off companies that focus on agriculture, materials science, and specialty products. The planned fragmentation of DowDuPont into these smaller spin-offs may alleviate anti-trust concerns that could block the mega-merger. Also, Dow and DuPont product lines are sufficiently diverse that they do not compete head-to-head with each other in many areas. Assets that do overlap, such as corn seeds, could be divested. Nonetheless, the proposed merger is certain to be closely scrutinized by the US Federal Trade Commission and may be subject to lawsuits by disgruntled investors and watchdog groups.

For now, at least, DowDuPont will retain the respective company headquarters in Midland, Michigan, and Wilmington, Delaware; Dow chairman and CEO Andrew Liveris will become executive chairman of the DowDuPont Board of Directors; while Edward Breen, chairman and CEO of DuPont, will take the helm as CEO of DowDuPont. The DowDuPont board will have 16 directors, eight from each company. Advisory committees will be established to oversee the spinoffs: Liveris will lead the materials science committee, and Breen the agriculture and specialty products committees. Dow shareholders can expect 1.00 share of DowDuPont for each Dow share that they own, whereas DuPont shareholders will receive 1.282 shares for each DuPont share they own, so that Dow and DuPont shareholders will each own about 50% of the new company.

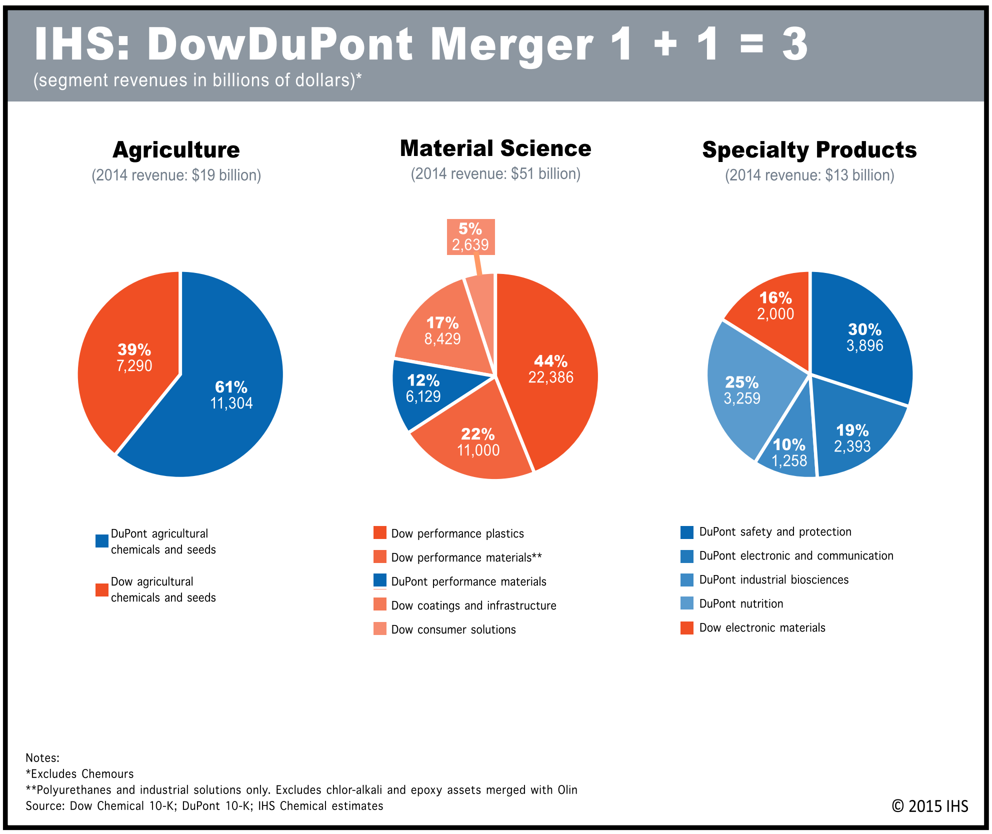
The merger and spin-off will create the world's largest agriculture company, with about $19 billion in estimated sales, ahead of current leader Monsanto (about $16 billion). The DowDuPont agriculture company will be number two in seeds, behind Monsanto, and tied for second place with Bayer, behind Syngenta, in crop protection chemicals. Some analysts predict that the DowDuPont merger will put pressure on other agriculture companies to consolidate, as Monsanto unsuccessfully tried to do with Syngenta in April 2015.

The greatest overlap in product portfolios occurs in agriculture. Although Dow's business focuses on agricultural chemicals and DuPont's on seeds, there are areas of head-to-head competition, such as in corn seeds and herbicides. However, some of these assets could be sold off if they attract the scrutiny of anti-trust regulators.

DuPont recently appointed James Collins as chief of DuPont's agriculture unit. Whether the new DowDuPont agriculture company will be headed by Collins, or by Tim Hassinger, CEO of Dow AgroSciences, had not been announced. when this issue of *Inform* went to press. Breen told the *Wall Street Journal* that leaders for the planned spin-off companies will be chosen at least six months before the separation, and decisions will be based on managers' performance during the transition. Also unknown is where the agriculture spin-off will be located, although speculation centers on areas of the United States with a wealth of agricultural universities, such as St. Louis or Kansas City, Missouri, or Omaha, Nebraska.

With about $51 billion in projected sales, the materials science company will be the largest of the spin-offs. It will consist mostly of legacy Dow businesses, such as performance plastics (e.g., olefins, polyolefins, polyethylene, and elastomers) and performance materials (e.g., polyurethanes, ethylene oxide, and vinyl) but will also leverage DuPont's engineering polymers business (e.g., advanced ethylene copolymers and nylon). Key areas include packaging, transportation, and building and construction. Dow and DuPont's portfolios in these areas are quite complementary, experts say. For example, in automotive applications, Dow offers polymers for interior and exterior uses, whereas DuPont makes polymers for under the hood. The companies also have complementary offerings in packaging, particularly in multilayered structures. The materials science business will be located in Midland, Mich.

The $13 billion specialty products spin-off will consist mostly of the former DuPont businesses of safety and protection (which makes the Tyvek and Kevlar brands), industrial biosciences (enzymes, peptides, biomaterials, and biofuels), nutrition and health (predominantly the former Danisco), and electronics and communication, with the addition of Dow electronic materials, which supplies a variety of chemicals to the electronics industry. Dow recently paid $4.8 billion to acquire full ownership of silicone products manufacturer Dow Corning, which will also likely join the specialty products firm. As with materials science, the specialty products offerings of DuPont and Dow are viewed as more complementary than overlapping. The specialty products business will be located in Delaware, although an exact site has not yet been specified.



The deal is expected to save $3 billion in operating costs in the first 24 months after the merger and add $1 billion in revenue. On the same day that the merger was made public, DuPont announced the company will be slashing $700 million in costs in 2016, and that 10% of its global workforce will be affected. In numbers, this means displacing 5,400 of DuPont's 54,000 employees worldwide. In addition, some offices and plants will be shut down in preparation for the merger. On December 29, Breen announced that the first round of layoffs will affect 1,700 DuPont employees (including researchers) in Delaware—28% of its current Delaware workforce. Similarly, Dow plans to make $300 million in cuts before the merger is complete.

Also in mid-December, DuPont announced that the company will be replacing its prestigious Central Research & Development with a new organization called Science and Innovation. Critics worry that this restructuring, in addition to the merger, will mean drastic cuts in R&D. However, Breen told investors that only about $300 million will be cut from DowDuPont's research budget, compared with the $2.1 billion that DuPont alone spent on R&D in 2015.

"It is too preliminary to comment on the broader impact of the merger at this time," Dow media relations representative Rachelle Schikorra told -"but each of the three created businesses will be a leader in its industry, will be able to allocate capital more effectively, apply its powerful innovation more productively, and expand its products and solutions to more customers worldwide." Schikorra notes that both Dow and DuPont have "a legacy and heritage of science and innovation," and that the combined company will be able to more effectively leverage those resources.

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Information

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PATENTS

Compositions and methods for increasing the suppression of hunger and reducing the digestibility of non-fat energy satiety

Arstrup, A., *et al.,* University of Copenhagen, US9066536, June 30, 2015

The present invention relates to methods for increasing the suppression of hunger and/or increasing the reduction of prospective consumption and/or increasing the reduction of appetite and/or increasing the feeling of satiety and/or reducing non-fat energy uptake in the gastrointestinal tract of a mammal in order to prevent a positive non-fat energy balance, weight gain, overweight and obesity, and to induce a negative non-fat energy balance and weight loss in subjects who wish to reduce their body weight. In particular, feed, food and/or beverages and dietary supplements of the present invention comprises mucilage such as flax seed mucilage and/or one or more active compounds of mucilage useful for increasing the suppression of hunger and/or increasing the reduction of prospective consumption and/or increasing the reduction of appetite and/or increasing the feeling of satiety and/or reducing the digestibility of non-fat energy in the gastrointestinal tract of a mammal.

Synergistic antioxidant composition

Jourdain, L. and L. Sagalowicz, Nestec S.A., US9068138, June 30, 2015

The present invention relates to an antioxidant composition comprising a galactolipid, ascorbic acid and/or a derivative thereof, and at least one further lipid. Further aspects of the invention are the method of manufacturing such an antioxidant composition as well as the use of galactolipids in combination with ascorbic acid and/or a derivative thereof for protecting a composition against oxidation. Particularly, the invention relates to a composition to be used in food products.

Modified-immobilized enzymes of high tolerance to hydrophilic substrates in organic media

Basheer, S., Trans Bio Diesel Ltd., US9068175, June 30, 2015

Disclosed are preparations of modified interfacial enzymes, particularly lipases and phospholipases, immobilized on a solid support, wherein the enzyme is surrounded by hydrophobic microenvironment, thereby protected from deactivation and/or aggregation in the presence of hydrophilic agents, substrates and/or reaction products. The enzyme may be protected by being covalently bonded with lipid groups which coat the enzyme, or by being immobilized or embedded in a hydrophobic solid support. Also disclosed are processes for the preparation of the hydrophobically protected enzymes. The enzymes may be efficiently used in the preparation of biodiesel.

High-melting-point sunflower fat for confectionary

Salas Linan, J.J. *et al.,* Consejo Superior de Investigaciones Cientificas (CSIC), US9072309, July 7, 2015

The present invention is based on the finding that stearin fats, obtainable by dry or solvent fractionation of sunflower high-stearic and high-oleic oils, optionally with seeding with tempered stearin crystals, have a high solid fat content at temperatures higher than 30 degrees centigrade, even higher than cocoa butter or other high saturated tropical fats with a similar disaturated triacylglycerol content due to the presence of disaturated triacylglycerols rich in stearic acid, and improved melting point due to the presence of arachidic and behenic acids in these disaturated triacylgiycerols, being at the same time healthier that actual fats made from palm, palm kernel and coconut oils, or hydrogenated and transesterified vegetable oils.

Nutritional products including monoglycerides and fatty acids

Lai, C.-S.; *et al.,* Abbott Laboratories, US9078846, July 14, 2015

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

Nutritional products including a novel fat system including monoglycerides

Lai, C.-S., Abbott Laboratories, US9078847, July 14, 2015

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

System and method for extracting vitamin E from fatty acid distillates

Raviyan, P. and K. Soinak, Thailand Research Fund, US9078850, July 14, 2015

Various systems and processes for extracting Vitamin E from a fatty acid distillate (FAD) having a Vitamin E component are disclosed. The process includes preparing a mixture of a FAD and a non-polar solvent (e.g., hexane). The mixture can be sequentially cooled to a series of pre-determined temperatures. As the mixture is sequentially cooled to each of the pre-determined temperatures within the series of pre-determined temperatures, non-Vitamin E components present in the FAD can form solid fractions within the mixture at the various pre-determined temperature stages. The process further includes removing the solid fractions from the mixture at each of the pre-determined temperature stages. After completion of a number of cooling and separation stages or cycles, the non-polar solvent can be removed from the remaining mixture to recover a Vitamin E extract.

Surfactants derived from epoxidized oils and compositions thereof

Lele, B.S., US9085709, July 21, 2015

Present invention relates to surfactants derived from epoxidized oils and compositions thereof. Particularly this invention describes surfactants derived from epoxidized oils covalently attached to water soluble polymers via thioether bond forming linker and formulations thereof.

Process for obtaining oleochemicals with reduced content of by-products

Horst, J., *et al.,* Cognis IP Management GmbH, US9085746, July 21, 2015

Processes for obtaining fatty acids with reduced content of by-products, wherein unrefined fats or oils are subjected to steam stripping in a counter-current column to produce a bottom fraction of de-acidified fats or oils, a first distillate consisting mainly of fatty acids and glycerides, and a second distillate comprising fatty acids and essentially all unwanted ketones, aldehydes and phenols, and the bottom fraction containing the de-acidified fats or oils is combined with the first distillate containing fatty acids and glycerides.

Heat stable, fat-based confections and methods of making same

Cross, J.C., *et al.,* Kerry Group Services Intl. Ltd., US9089153, July 28, 2015

New coated food products are provided. The products comprise a center food piece such as a nutmeat or cereal piece, and a coating surrounding the center food piece. The coating can be flavored with flavorings such as cheese, chocolate, or fruit. The coating comprises a substantially homogeneous mixture of a fat-based composition and a particulate material, which results in a stable coating that can tolerate higher temperatures when compared to prior art products while also having 50 percent or less of the fat content of prior art coatings.

Oil or fat composition

Moriwaki, J. *et al.,* KAO Corporation, US9095156, August 4, 2015

Provided is an oil or fat composition, which has an MCPD-FS content X (ppm) and a hydroxyl value Y (mg-KOH/g) satisfying a relationship of the following expression (1), the content X being measured by a Deutsche Gesellschaft fuer Fettwissenschaft (DGF) standard method C-III 18(09), and has a content (percent) of trans isomers of linoleic acids in constituent fatty acids of an oil or fat, of 2 or more. $Y \geq 1.25X+4$ where $Y \leq 88$ (1).

Stable creamer composition and method of making same

Napolitano, G.E. and L.J. Erickson, Nestec, S.A., US9101149, August 11, 2015

The present invention relates to a creamer composition, e.g. for use for addition into a coffee beverage, with improved oxidative and physical stability. The creamer includes oil with a high amount of oleic acids and a limited amount of linoleic acid and linolenic acid. The invention further relates to a method of producing the creamer, a beverage composition including the creamer and a method of producing a beverage composition.

Toughening cross-linked thermosets

Palmese, G.R., *et. al.,* Drexel University; US Army, US9102807, August 11, 2015

Grafted triglycerides comprising an acrylated triglyceride grafted with a fatty acid residue containing 4 to 28 carbon atoms. Also described are methods for making a grafted triglyceride and for curing a material selected from vinyl esters and unsaturated polyesters and mixtures thereof and optionally a reactive diluent. The method includes the steps of mixing a grafted triglyceride of the present invention with a material selected from vinyl esters, unsaturated polyesters and mixtures thereof to form a mixture, and curing the mixture to form a cured resin system. A cured resin system comprising a cured product obtained by the foregoing method and composites containing the cured product and a filler or reinforcing material are also disclosed. This method also includes use of the grafted triglycerides to make toughened resin and composite systems with reduced hazardous air pollutants without significantly reducing the glass transition temperature and significantly increasing the viscosity.

Emulsifier composition for shortening

Wassell, P., Dupont Nutrition Biosciences APS, US9107429, August 18, 2015

A method of producing a water-in-oil product is described. The method comprises the step of: admixing a hydrophobic component with a water-in-oil emulsion to form said water-in-oil product, wherein said hydrophobic component comprises a probiotic in a hydrophobic medium.

Chocolate or chocolate-like food and method of producing the same

Akahane, A., *et al.,* The Nisshin Oillio Group, Ltd, US9107431, August 18, 2015

Object: To provide a chocolate or chocolate-like food capable of inhibiting occurrence of low-temperature bloom. Means for solving problem: A chocolate or chocolate-like food of the present invention comprises an oil and fat, wherein the oil and fat comprises 50 to 85 percent by weight of triglyceride having an oleic acid at position 2 and saturated fatty acids having 16 or more carbon atoms at positions 1 and 3 (XOX type triglyceride); and 5 to 18 percent by weight of triglyceride in which two oleic acids are bound and one saturated fatty acid having 16 or more carbon atoms is bound (XO2 type triglyceride). According to such a chocolate or chocolate-like food, the low temperature bloom can be inhibited.

Bloom-retarding fat

Juul, B. and M.D. Anderson, AAK Denmark A/S, US9113644, August 25, 2015

The invention relates to a bloom retarding fat composition comprising a component A and a component B. Component A is present in an amount of 40–95 percent (w/w) of said bloom retarding fat composition, and component B is present in an amount of 5–60 percent (w/w) of said bloom retarding fat composition. Component A comprises a fat composition having a content of SatOSat TAGs of 60 percent (w/w) or higher. Component B comprises a fat composition having a content of saturated fatty acids of 30 percent (w/w) or less. In component B the total content of Sat2U TAGs of 18 percent (w/w) or less, and a content Sat3 TAGs of 8 percent (w/w) or less. Further in component B the content of SatSatU TAGs in Sat2U of component B is 1 percent (w/w) or more. Also the molar ratio of SatSatU:SatUSat TAGs is 1 or higher in component B and wherein Sat is a C16-20 saturated fatty acid, and wherein O stands for oleic acids; U stands for unsaturated fatty acids, including oleic acid. The invention relates to various applications of such a bloom retarding fat compositions.

Flavored and edible colored waxes and methods for precision deposition on edible substrates

Liniger, S. and A. Hutchison, Sensient Colors, LLC, US9113647, August 25, 2015

A method of imparting flavor to an edible substrate by precision depositing a first food grade flavored wax onto a surface of an edible substrate. A method of providing a flavored image on an edible substrate by ink jet printing a food grade colored fluid on an edible substrate to create an image and applying a food grade flavored wax onto the edible substrate. A method of imparting flavor to an edible substrate by applying a food grade flavored wax onto the edible substrate and applying a food grade flavored fluid onto the edible substrate.

Edible products having a high cocoa polyphenol content and improved flavor and the milled cocoa extracts used therein

Anderson, B.A., *et al.,* Mars, Inc., US9114114, August 25, 2015

Milling dry extracts containing cocoa polyphenols (CPs) to reduce the particle size improves the flavor of edible products (e.g., foods, medical foods, nutritional supplements, and pharmaceuticals) or additives containing the milled cocoa extracts. The products, e.g., chocolates, are less astringent and less bitter. The mean particle size after milling is less than about 15 microns, preferably less than about 10 microns, and most preferably less than about 5 microns. The total CP content of the milled extracts is at least about 300 milligrams and preferably about 300 to about 700 milligrams per gram of milled extract. The additives consist essentially of (i) the milled high CP cocoa extract and (ii) a fat (e.g., cocoa butter), an oil (e.g., vegetable oil), or a syrup (e.g., corn syrup).

Semi-continuous deodorizer comprising a structured packing

Galina, F., *et al.,* Alfa Laval Corp AB, US9114329, August 25, 2015

The present invention relates to a semi-continuous deodorizer comprising at least one stripping section, which stripping section comprises a feed buffer tray for liquids, a liquid flow regulating means, a liquid distributor, a structured packing, and a receiver tray, wherein the regulating means are regulating the liquid flow from the feed buffer tray to the distributor to distribute the flow of liquid over the structured packing. The liquid is in the packing contacted in counter-current flow to an already once-used stripping agent which is recovered from one of more of the trays installed in the apparatus. The invention relates further to a method for refining fats and oils in a semi-continuous deodorizer, a method for re-using stripping agent in a semi-continuous deodorizer, segregating the recovered distillate into high purity types matching the feed type, and a use of the semi-continuous deodorizer.

Method and apparatus for esterifying fatty acid

Jackson, R.D. and O.M. Davies, Renewable Holdings Ltd., US9120739, September 1, 2015

A method of esterifying free fatty acid in natural oil comprises heating the natural oil (2) to a first temperature above a reaction temperature, feeding the heated natural oil into an acid-resistant pipe reactor (5), providing a mixture of acid catalyst (6) and short chain alcohol (8) at a second temperature below the reaction temperature, and feeding the mixture of acid catalyst and short chain alcohol into the natural oil in the pipe reactor (5). The short chain alcohol and free fatty acid react at the reaction temperature to form an ester.

Process of reactive trituration directly on an oil cake

Dubois, J.-L. and A. Piccirilli, Arkema France, US9120996, September 1, 2015

A process including at least one reactive trituration step which includes putting an oil cake including from 3 percent to 30 percent oil in contact with an anhydrous light alcohol and an alkaline catalyst under temperature and time conditions that are sufficient to allow for the extraction and transesterification of the vegetable oil and lead to the production of a mixture including fatty acid esters and glycerol, and a de-oiled cake including less than 3 percent oil. Also, a detoxified de-oiled cake as well as to a mixture of fatty acid esters with improved stability and resistance to oxidation.

Edible fat continuous spreads

Bons, J.R., *et al.,* Conopco Inc., US9125425, September 8, 2015

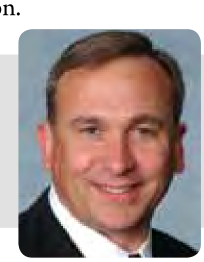
The invention relates to a method of preparing an edible fat continuous spread comprising a dispersed aqueous phase and crystalline non-esterified plant sterol, said method comprising the steps of preparing a fat continuous emulsion and mixing said emulsion with fast re-crystallized non-esterified plant sterol.

Compositions and methods for producing elevated and sustained ketosis

D'Agostino, D.P. *et al.,* University of South Florida, US9138420, September 22, 2015

Beta-hydroxybutyrate mineral salts in combination with medium chain fatty acids or an ester thereof such as medium chain triglycerides were used to induce ketosis, achieving blood ketone levels of (2-7 mmol/L), with or without dietary restriction. The combination results in substantial improvements in metabolic biomarkers related to insulin resistance, diabetes, weight loss, and physical performance in a short period of time. Further, use of these supplements to achieve ketosis yields a significant elevation of blood ketones and reduction of blood glucose levels. Use of these substances does not adversely affect lipid profiles. By initiating rapid ketosis and accelerating the rate of ketoadaptation, this invention is useful for the avoidance of glucose withdrawal symptoms commonly experienced by individuals initiating a ketogenic diet, and minimizes the loss of lean body mass during dietary restriction.

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.



EXTRACTS & DISTILLATES

Optimization of catalytic cracking process for upgrading camelina oil to hydrocarbon biofuel

Zhao, *et al., Ind. Crops Prod.* 77: 516–526, 2015, http://dx.doi.org/10.1016/j.indcrop.2015.09.019.

Catalytic cracking of camelina oil over Zn/ZSM-5 catalyst in a fixed-bed tubular reactor was investigated. An optimization study on the catalytic cracking conditions based on nine well-planned orthogonal experiments was carried out. Three main operation conditions including reaction temperature, liquid hourly space velocity and oil extraction press frequency were studied to examine their effects on the yield and quality of hydrocarbon biofuel produced. Characterization of the catalyst, hydrocarbon biofuel and non-condensable gas was conducted. There was no significant difference between the bulk structures of fresh Zn/ZSM-5 and used Zn/ZSM-5. Small ZnO particles dispersed well on the parent ZSM-5. Hydrocarbon biofuel contained 65.18% hydrocarbons and its properties including dynamic viscosity, density and higher heating value were improved after upgrading, compared to camelina oil. It was found that the oil extraction press frequency was the most important factor and liquid hourly space velocity was the least important factor for the hydrocarbon biofuel production. In addition, the optimum conditions for camelina oil upgrading were a combination of reaction temperature of 550 °C, a liquid hourly space velocity of 1.0 h^{-1} and an oil extraction press frequency of 15 Hz.

Synergistic effect of surfactants and silica nanoparticles on oil recovery from condensed corn distillers solubles (CCDS)

Fang, L., *et al., Ind. Crops Prod.* 77: 553–559, 2015, http://dx.doi.org/10.1016/j.indcrop.2015.09.031.

Most of the oil in condensed corn distillers solubles (CCDS) is in an emulsified form and centrifugation alone is not sufficient to recover the oil in high yield. The synergistic effect between non-ionic surfactants (Tween® 80 and Span® 80) and silica nanoparticles (hydrophilic and hydrophobic) on oil recovery was investigated using 3 batches of commercial CCDS. The use of surfactant mixture with Hydrophilic-Lipophilic-Balance (HLB) value of 9.7 led to the highest oil recovery. Tween® 80/silica and surfactant mixture (HLB 9.7)/silica recovered 5–10% more oil compared with the control groups. However, Span® 80/silica was not effective. Surfactant mixture/silica made the oil recovery by centrifugation more efficient by destabilizing oil-in-water emulsion and washing out free oil droplets. The use of surfactant and silica significantly affected the distribution of different types of oil, as well as centrifugation conditions, heating and shaking. About 20% of total oil remained in the unbroken cells or germ pieces in CCDS, which is unrecoverable without additional treatment.

Addition of cellulolytic enzymes and phytase for improving ethanol fermentation performance and oil recovery in corn dry grind process

Luangthongkam, P., *et al., Ind. Crops Prod.* 77: 803–808, 2015, http://dx.doi.org/10.1016/j.indcrop.2015.09.060.

Application of hydrolytic and other enzymes for improving fermentation performance and oil recovery in corn dry-grind process was optimized. Non-starch polysaccharide enzymes (BluZy-P XL; predominantly xylanase activity) were added at stages prior to fermentation at optimum conditions of 50 °C and pH 5.2 and compared with conventional fermentation (30 °C, pH 4.0). Enzyme applications resulted in faster ethanol production rates with a slight increase in yield compared to control. The thin stillage yield increased by 0.7–5% w/w wet basis with corresponding increase in solids content with enzyme treatment after liquefaction. The oil partitioned in thin stillage was at 67.7% dry basis after treatment with hydrolytic enzymes during fermentation. Further addition of protease and phytase during simultaneous saccharification and fermentation increased thin stillage oil partitioning to 77.8%. It also influenced other fermentation parameters, e.g., ethanol production rate increased to 1.16 g/g dry corn per hour, and thin stillage wet solids increased by 2% w/w. This study indicated that treatments with non-starch hydrolytic enzymes have potential to improve the performance of corn dry-grind process including oil partitioning into thin stillage. The novelty of this research is the addition of protease and phytase enzymes during simultaneous saccharification and fermentation of corn dry-grind process, which further improved ethanol yields and oil partitioning into thin stillage.

Recent advances in vegetable-oils-based environment friendly coatings: a review

Sharmin, E., *et al., Ind. Crops Prod.* 76: 215–229, 2015, http://dx.doi.org/10.1016/j.indcrop.2015.06.022.

The overarching goal worldwide for the scientific community is "sustainable development" today, for an everlasting sustainable and green tomorrow. The strategy includes (i) harvesting renewable resources instead of fossil fuels, (ii) using environment friendly routes, and (iii) engineering material degradation pathways operating under reasonable time frames. The concept revolves around the focal point of "Green" or "Sustainable" Chemistry. In the world of coatings, the idea has already made its debut in the form of environment friendly technologies-low or no solvent, high solids, hyperbranched, water borne and UV curable coatings, utilising monomers/polymers derived from renewable resources. Vegetable oils [VEGO] constitute Mother Nature's most abundant, cost-effective, non toxic, and biodegradable resource. They have been traditionally used for several non-food applications mainly coatings since primitive times. Today, the implementation of the modern technologies coupled with the full-fledged use of VEGO based monomers or polymers in the field as raw materials, is an excellent effort toward sustainable future in the world of coatings globally. The review highlights some state-of-the art-modifications of VEGO as environment friendly-low or no solvent, high solids, hyperbranched, water borne and UV curable coatings. The article provides a handy overall vision of VEGO based environment friendly coatings on a single platform. These approaches can be well employed on those oils that are non-edible, non-medicinal and are left unexplored, unutilised or underutilised to date, thus adding value to an unutilised or under-utilised sustainable resource.

Effects of tree nuts on blood lipids, apolipoproteins, and blood pressure: systematic review, meta-analysis, and dose-response of 61 controlled intervention trials

Del Gobbo, L.C., *et al., Am. J. Clin. Nutr. 102*: 1347–1356, 2015, http://dx.doi.org/10.3945/ajcn.115.110965.

The effects of nuts on major cardiovascular disease (CVD) risk factors, including dose-responses and potential heterogeneity by nut type or phytosterol content, are not well established. We examined the effects of tree nuts (walnuts, pistachios, macadamia nuts, pecans, cashews, almonds, hazelnuts, and Brazil nuts) on blood lipids [total cholesterol, low-density lipoprotein (LDL) cholesterol, high-density lipoprotein, and triglycerides], lipoproteins [apolipoprotein A1, apolipoprotein B (ApoB), and apolipoprotein B100], blood pressure, and inflammation (C-reactive protein) in adults aged ≥18 y without prevalent CVD. We conducted a systematic review and meta-analysis following Preferred Reporting Items for Systematic Reviews and Meta-Analyses guidelines. Two investigators screened 1301 potentially eligible PubMed articles in duplicate. We calculated mean differences between nut intervention and control arms, dose-standardized to one 1-oz (28.4 g) serving/d, by using inverse-variance fixed-effects meta-analysis. Dose-response for nut intake was examined by using linear regression and fractional polynomial modeling. Heterogeneity by age, sex, background diet, baseline risk factors, nut type, disease condition, duration, and quality score was assessed with meta-regression. Publication bias was evaluated by using funnel plots and Egger's and Begg's tests. Sixty-one trials met eligibility criteria (n = 2582). Interventions ranged from 3 to 26 wk. Nut intake (per serving/d) lowered total cholesterol (−4.7 mg/dL; 95% CI: −5.3, −4.0 mg/dL), LDL cholesterol (−4.8 mg/dL; 95% CI: −5.5, −4.2 mg/dL), ApoB (−3.7 mg/dL; 95% CI: −5.2, −2.3 mg/dL), and triglycerides (−2.2 mg/dL; 95% CI: −3.8, −0.5 mg/dL) with no statistically significant effects on other outcomes. The dose-response between nut intake and total cholesterol and LDL cholesterol was nonlinear (*P*-nonlinearity < 0.001 each); stronger effects were observed for ≥60 g nuts/d. Significant heterogeneity was not observed by nut type or other factors. For ApoB, stronger effects were observed in populations with type 2 diabetes (−11.5 mg/dL; 95% CI: −16.2, −6.8 mg/dL) than in healthy populations (−2.5 mg/dL; 95% CI: −4.7, −0.3 mg/dL) (*P*-heterogeneity = 0.015). Little evidence of publication bias was found. Tree nut intake lowers total cholesterol, LDL cholesterol, ApoB, and triglycerides. The major determinant of cholesterol lowering appears to be nut dose rather than nut type. Our findings also highlight the need for investigation of possible stronger effects at high nut doses and among diabetic populations.

Natural inhibitors of lipase: examining lipolysis in a single droplet

del Castillo-Santaella, T., *et al., J. Agric. Food Chem. 63*: 10333–10340, 2015, http://dx.doi.org/10.1021/acs.jafc.5b04550.

Inhibition of lipase activity is one of the approaches to reduced fat intake with nutritional prevention promoting healthier diet. The food industry is very interested in the use of natural extracts, hence reducing the side effects of commercial drugs inhibiting lipolysis. In this work we propose a novel methodology to rapidly assess lipolysis/inhibition in a single droplet by interfacial tension and dilatational elasticity. The evolution of the interfacial tension of lipase in simplified duodenal fluid in the absence and that in the presence of the pharmaceutical drug Xenical are the negative (5 ± 1 mN/m) and positive (9 ± 1 mN/m) controls of the inhibition of lipolysis, respectively. Then, we correlate the inhibition with the reduction of the interfacial activity of lipase and further identify the mode of action of the inhibition based on dilatational response (conformational changes induced in the molecule/blocking of adsorption sites). This work provides new insight into the lipase inhibition mechanism and a rapid methodology to identify the potential of new natural inhibitors.

Rosemary plant (*Rosmarinus officinalis* L.), solvent extract and essential oil can be used to extend the usage life of hazelnut oil during deep frying

Tohma, S. and S. Turan, *Eur. J. Lipid Sci. Technol. 117*: 1978–1990, 2015, http://dx.doi.org/10.1002/ejlt.201400382.

In this study, the effects of rosemary (*Rosmarinus officinalis* L.) plant (RP), its alcoholic extracts (REE and RME) and essential oil (REO) on the oxidative stability of hazelnut oil (HO) during deep frying were investigated. HOs containing rosemary additives had lower conjugated diene (CD) content, p-anisidine value (AV) and polar material content compared to control values. Additionally, changes in L*, a*, and b* color values and viscosity were lower than control values. RP (50 g/kg) caused the highest retention of α-tocopherol. At the end of the frying period, the induction periods (IPs) of HOs containing rosemary additives were between 9.43 and 35.01 min, much higher than the control IP (4.28 min). The ratios of C18:1/C16:0 and C18:2/C16:0 were 7.6 and 1.2, respectively, in HOs containing 50 g/kg RP, values which were higher than those of the control. The study indicated that HO containing 50 g/kg RP had the best frying performance. Rosemary additives had no adverse effects on sensory acceptability according to results of a sensorial analysis of French fries. The rosemary additives also had no significant effect in terms of texture and color of French fries ($P > 0.05$). Consequently, this study shows that rosemary plant or extracts can be used to extend the usage life of hazelnut oil for frying.

Enhancing nutraceutical bioavailability from raw and cooked vegetables using excipient emulsions: influence of lipid type on carotenoid bioaccessibility from carrots

Zhang, R., e*t al., J. Agric. Food Chem. 63*: 10508–10517, 2015, http://dx.doi.org/10.1021/acs.jafc.5b04691.

The influence of the nature of the lipid phase in excipient emulsions on the bioaccessibility and transformation of carotenoid from carrots was investigated using a gastrointestinal tract (GIT) model. Excipient emulsions were fabricated using whey protein as an emulsifier and medium-chain triglycerides (MCT), fish oil, or corn oil as the oil phase. Changes in particle size, charge, and microstructure were measured as the carrot–emulsion mixtures were passed through simulated mouth, stomach, and small intestine regions. Carotenoid bioaccessibility depended on the type of lipids used to form the excipient emulsions (corn oil > fish oil >>MCT), which was attributed to differences in the solubilization capacity of mixed micelles formed from different lipid digestion products. The transformation of carotenoids was greater for fish oil and corn oil than for MCT, which may have been due to greater oxidation or isomerization. The bioaccessibility of the carotenoids was higher from boiled than raw carrots, which was attributed to greater disruption of the plant tissue facilitating carotenoid release. In conclusion, excipient emulsions are highly effective at increasing carotenoid bioaccessibility from carrots, but lipid type must be optimized to ensure high efficacy.

Detection of corn adulteration in Brazilian coffee (*Coffea arabica*) by tocopherol profiling and near-infrared (NIR) spectroscopy

Winkler-Moser, J.K., *et al., J. Agric. Food Chem. 63*: 10662–10668, 2015, http://dx.doi.org/10.1021/acs.jafc.5b04777.

Coffee is a high-value commodity that is a target for adulteration, leading to loss of quality and causing significant loss to consumers. Therefore, there is significant interest in developing methods for detecting coffee adulteration and improving the sensitivity and accuracy of these methods. Corn and other lower value crops are potential adulterants, along with sticks and coffee husks. Fourteen pure Brazilian roasted, ground coffee bean samples were adulterated with 1–20% of roasted, ground corn and were analyzed for their tocopherol content and profile by HPLC. They were also analyzed by near-infrared (NIR) spectroscopy. Both proposed methods of detection of corn adulteration displayed a sensitivity of around 5%, thus representing simple and fast analytical methods for detecting adulteration at likely levels of contamination. Further studies should be conducted to verify the results with a much larger sample size and additional types of adulterants.

Distribution of lipids in the grain of wheat (cv. hereward) determined by lipidomic analysis of milling and pearling fractions

González-Thuillier, I., *et al., J. Agric. Food Chem. 63*: 10705–10716, 2015, http://dx.doi.org/10.1021/acs.jafc.5b05289.

Lipidomic analyses of milling and pearling fractions from wheat grain were carried out to determine differences in composition that could relate to the spatial distribution of lipids in the grain. Free fatty acids and triacylglycerols were major components in all fractions, but the relative contents of polar lipids varied, particularly those of lysophosphatidylcholine and digalactosyldiglyceride, which were enriched in flour fractions. By contrast, minor phospholipids were enriched in bran and offal fractions. The most abundant fatty acids in the analyzed acyl lipids were C16:0 and C18:2 and their combinations, including C36:4 and C34:2. Phospholipids and galactolipids have been reported to have beneficial properties for breadmaking, whereas free fatty acids and triacylglycerols are considered detrimental. The subtle differences in the compositions of fractions determined in the present study could therefore underpin the production of flour fractions with optimized compositions for different end uses.

CONTINUED ON PAGE 36

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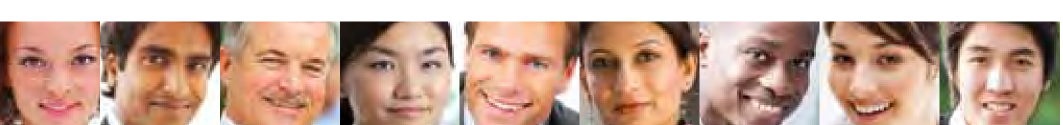

All members contribute to the success of the Society while furthering their professional goals.

CONTINUED FROM PAGE 34

Performance and emission characteristics of a diesel engine fueled with palm, jatropha, and moringa oil methyl ester

Rashed, M.M., *Ind. Crops Prod. 79:* 70–76, 2015, http://dx.doi.org/10.1016/j.indcrop.2015.10.046.

This paper aims to investigate the diesel engine performance and emission characteristics fueled with moringa biodiesel and compare those with the performance and emission characteristics of palm biodiesel, jatropha biodiesel, and diesel fuel. In this study, only 20% of each biodiesel (described by MB20, PB20, and JB20, respectively) was tested in diesel engine, given that open literature indicates the possible use of biodiesel of up to 20% in a diesel engine without modification. The physical and chemical properties of all fuel samples are also presented and compared with ASTM D6751 standards. A naturally aspirated multi-cylinder, four-stroke direct-injection diesel engine was used to evaluate their performance at different speeds and full load condition. All biodiesel fuel samples reduce brake power (BP) and increase brake-specific fuel consumption (BSFC) than diesel fuel. Engine emission results indicated that blended fuel reduces the average carbon monoxide (CO) and hydrocarbons (HC) emissions except nitric oxides (NO) emissions than diesel fuel. Among the biodiesel-blended fuel, Palm biodiesel showed better performance and minimal emission than jatropha and moringa biodiesel fuel. Although PB20 showed better performance, but performance of MB20 biodiesel blend is comparable with other fuels. Correspondingly, 20% of moringa biodiesel can be used in a diesel engine without any engine modification.

Lipid Oxidation

Food-grade nanoparticles for encapsulation, protection, and delivery of curcumin: comparison of lipid, protein, and phospholipid nanoparticles under simulated gastrointestinal conditions

Zou, L., *et al*, *RSC Adv. 6*: 3126–3136, 2016 (online in 2015), http://dx.doi.org/10.1039/C5RA22834D.

The potential of three nanoparticle-based delivery systems to improve curcumin bioavailability was investigated: lipid nPs (nanoemulsions); protein nPs (zein nanosuspensions); and, phospholipid nPs (nanoliposomes). All three nanoparticle types were fabricated from food-grade constituents, had small mean diameters ($d < 200$ nm), and had monomodal particle size distributions. The loading capacity of curcumin depended strongly on nanoparticle composition: protein nPs (11.7%); phospholipid nPs (3.1%); lipid nPs (0.40%). The curcumin-loaded nanoparticles were passed through a simulated gastrointestinal tract (GIT) consisting of mouth, stomach, and small intestine phases, and curcumin bioaccessibility and degradation were measured. Nanoparticle composition influenced their ability to protect curcumin from chemical degradation (lipid nPs ≈ protein nPs > phospholipid nPs) and to increase their solubilization within intestinal fluids (lipid nPs > phospholipid nPs > protein nPs). This latter effect was attributed to the enhanced solubilization capacity of the mixed micelle phase formed after digestion of the lipid nanoparticles. Overall, the lipid nanoparticles (nanoemulsions) appeared to be the most effective at increasing the amount of curcumin available for absorption (at an equal initial curcumin level). This study shows that different types of nanoparticles have different advantages and disadvantages for encapsulating, protecting, and releasing curcumin. This research will facilitate the rational selection of food-grade colloidal delivery systems designed to enhance the oral bioavailability of hydrophobic nutraceuticals.

An overview of encapsulation of active compounds used in food products by drying technology

Ray, S., *et al., Food Biosci.* (in press, 2016; online in 2015), http://dx.doi.org/10.1016/j.fbio.2015.12.009.

Drying is an important process parameter for preservation of food components and it is widely applicable in food sectors. Nowadays, encapsulation by drying technology is of growing interest to provide many useful effects in food industry. Encapsulation of several drying techniques (spray drying, freeze drying, fluidized bed coating) is a challenge to incorporate food component, antioxidant, colorant, cells and enzymes in powder form in food products. By drying, encapsulation achieves excellent properties of protection, stabilization, solubility and controlled release of the bioactive compounds. There are many reasons to apply encapsulation technology by drying, so recent developments of encapsulation are discussed in this review. Controlled release of food component at the right place at right time is a key functionality that can be provided by encapsulation. Drying improves effectiveness of food additives, broadens the application range of food ingredients, enhances shelf life of the food and lowers cost of the food products.

Multivariate optimization of a synergistic blend of oleoresin sage (*Salvia officinalis* L.) and ascorbyl palmitate to stabilize sunflower oil

Upadhyay, R. and H.N. Mishra, *J. Food Sci. Technol.* (accepted, currently online only, 2015). http://dx.doi.org/10.1007/s13197-015-2157-9 2016.

The simultaneous optimization of a synergistic blend of oleoresin sage (SAG) and ascorbyl palmitate (AP) in sunflower oil (SO) was performed using central composite and rotatable design coupled with principal component analysis (PCA) and response surface methodology (RSM). The physicochemical parameters viz., peroxide value, anisidine value, free fatty acids, induction period, total polar matter, antioxidant capacity and conjugated diene value were considered as response variables. PCA reduced the original set of correlated responses to few uncorrelated principal components (PC). The PC1 (eigen value, 5.78; data variance explained, 82.53 %) was selected for optimization using RSM. The quadratic model adequately described the data ($R 2 = 0. 91$, $p < 0.05$) and lack of fit was insignificant ($p > 0.05$). The contour plot of PC 1 score indicated the optimal synergistic combination of 1289.19 and 218.06 ppm for SAG and AP, respectively. This combination of SAG and AP resulted in shelf life of 320 days at 25 °C estimated using linear shelf life prediction model. In conclusion, the versatility of PCA–RSM approach has resulted in an easy interpretation in multiple response optimizations. This approach can be considered as a useful guide to develop new oil blends stabilized with food additives from natural sources.

Enzymatic synthesis of tyrosol-based phenolipids: characterization and effect of alkyl chain unsaturation on the antioxidant activities in bulk oil and oil-in-water emulsion

Pande, G., *et al., JAOCS 1*: 1–9, 2016 (online in 2015), http://dx.doi.org/10.1007/s11746-015-2775-4.

Oxidative stability of lipids is one of the most important parameters affecting their quality. Lipase-catalyzed lipophilic tyrosyl esters with an equivalent carbon alkyl chain but different degrees of unsaturation (C18:0 to C18:4*n*3) were prepared, characterized, and used as antioxidants. Free fatty acids and fatty acid ethyl esters (substrate molar ratio tyrosol: acyl donor, 1:10) were used as acyl donors and immobilized lipase from Candida antarctica was the biocatalyst (10 %). The phenolipids were isolated and characterized using ESI–MS, 1H-NMR, and 13C-NMR. Peroxide value (PV) and para-anisidine value (p-AV) were measured to evaluate their antioxidant activities in bulk oil structured lipid (SL) and in an oil-in-water emulsion (SL-based infant formula). No significant difference was found in yield and reaction time between the two types of acyl donors. However, as the unsaturation of the fatty acids increased the reaction time also increased. In SL, tyrosyl esters exhibited lower antioxidant activity than tyrosol whereas the addition of an alkyl chain enhanced the antioxidant efficiency of tyrosol in infant formula. Tyrosyl oleate was the most efficient antioxidant in the emulsion system followed by tyrosyl stearate and tyrosyl linoleate. These results suggest that the synthesized phenolipids may be used as potential antioxidants in lipid-based products.

Generation, fractionation, and characterization of iron-chelating protein hydrolysate from palm kernel cake proteins

Zarei, M., *et al., J. Food Sci.* (online only, 2015), http://dx.doi.org/10.1111/1750-3841.13200.

Palm kernel cake protein was hydrolyzed with different proteases namely papain, bromelain, subtilisin, flavourzyme, trypsin, chymotrypsin, and pepsin to generate different protein hydrolysates. Peptide content and iron-chelating activity of each hydrolysate were evaluated using O-phthaldialdehyde-based spectrophotometric method and ferrozine-based colorimetric assay, respectively. The results revealed a positive correlation between peptide contents and iron-chelating activities of the protein hydrolysates. Protein hydrolysate generated by papain exhibited the highest peptide content of 10.5 mM and highest iron-chelating activity of 64.8% compared with the other hydrolysates. Profiling of the papain-generated hydrolysate by reverse phase high performance liquid chromatography fractionation indicated a direct association between peptide content and iron-chelating activity in most of the fractions. Further fractionation using isoelectric focusing also revealed that protein hydrolysate with basic and neutral isoelectric point (pI) had the highest iron-chelating activity, although a few fractions in the acidic range also exhibited good metal chelating potential. After identification and synthesis of papain-generated peptides, GGIF and YLLLK showed among the highest iron-chelating activities of 56% and 53%, whereas their IC50 were 1.4 and 0.2 μM, respectively.

Industrial Applications

Microbial oils as food additives: recent approaches for improving microbial oil production and its polyunsaturated fatty acid content

Bellou, S., *et al., Curr. Opin. Biotechnol. 37*: 24–35, 2016, http://dx.doi.org/10.1016/j.copbio.2015.09.005.

In this short review, we summarize the latest research in the production of polyunsaturated microbial oils that are of interest in food technology. The current research targets the productivity of oleaginous microorganisms, as well as the biosynthesis of particular polyunsaturated fatty acids (PUFAs). The most important efforts target the efficiency of the oleaginous machinery, via overexpression of key-enzymes involved in lipid biosynthesis, as well as the minimization of lipid degradation, by repressing genes involved in the β-oxidation pathway. The production of specific PUFAs is approached by homologous or heterologous expression of specific desaturases and elongases involved in PUFA biosynthesis in oleaginous microorganisms. New perspectives, such as the production of triacylglycerols of specific structure and the employment of adaptive experimental evolution for creating robust oleaginous strains able to produce PUFAs are also discussed.

A novel process for preparing low-fat peanuts: optimization of the oil extraction yield with limited structural and organoleptic damage

Nader, J., *et al., Food Chem.197*: 1215–1225, 2016, http://dx.doi.org/10.1016/j.foodchem.2015.11.079.

The main purpose of this study was to extract the maximum amount of oil from peanuts without causing major damage and preserving their organoleptic quality after defatting. Accordingly, a successful, healthy, eco-friendly and economic defatting process for peanuts was implemented using mechanical oil expression, which was optimized by means of Response Surface Methodology. The results demonstrated that maximum extraction yields were obtained at a low initial moisture content (5–7% d.b.). Defatting and deformation ratios were mostly affected by the pressure and water content with high correlation coefficients (98.4% and 97.5%, respectively), and overall acceptability decreased following higher oil extraction yields. It was concluded that the optimum values for the product moisture content, pressure, and pressing duration were 5% d.b., 9.7 MPa and 4 min, respectively, with a defatting ratio of 70.6%. This resulted in an insignificant irreversible deformation ratio (<1%) and an overall acceptability of 7.6 over 10.

Influence of shear on fat crystallization

Tran, T. and D. Rousseau, *Food Res. Int.*, online January, 2016, http://dx.doi.org/10.1016/j.foodres.2015.12.022.

Processing conditions greatly impact fat crystallization kinetics and growth mechanisms. Recently, there has been increased interest in elucidating the role of shear on fat crystallization. This review provides an overview of fat crystallization under static conditions followed by a summary of the current body of work pertaining to the effects of shear on fat crystallization. The role of shear on different aspects of fat crystallization is reviewed: its effects on nucleation and growth, crystal network formation, polymorphic transitions, and microstructure modification at different length scales. The effects of shear are usually tied to critical shear rates but shear generally enhances nucleation and growth, accelerates crystallization and polymorphic transition times, and can cause orientation and structuring of fat crystal networks.

Improvements in the quality of sesame oil obtained by a green extraction method using enzymes

Ribeiro, S.A.O., *et al., LWT–Food Sci. Technol. 65*: 464–470, 2016, http://dx.doi.org/10.1016/j.lwt.2015.08.053.

The quality of vegetable oils is related to the presence of bioactive compounds, in which its contents may vary according to the extraction process. This study aimed to evaluate a clean technology for sesame oil extraction by enzymatic aqueous extraction, comparing to conventional extraction methods, such as pressing and solvent, in relation to the composition of bioactive lipophilic compounds. Two enzymes were used: Pectinex Ultra SPL and Alcalase 2.4L, and three factors were evaluated: concentration of enzymes (mL 100 mL^{-1}), sample/water ratio (g mL^{-1}) and extraction time (hours) through a 2^3 factorial design with center point in triplicate. The results showed variations in extraction yield and composition. The sesame oil extracted using enzymes showed the highest antioxidant capacity in the DPPH and L-ORAC (against peroxyl radical) assays, 128,54 and 349,98 μmol Trolox g^{-1} of oil, respectively, as well a higher content of total phytosterols (249 mg 100 g^{-1} of oil), total polyunsaturated and omega-6 fatty acids. No significant difference in γ-tocopherol content was observed, by Tukey test ($p < 0.05$), among the extraction methods. The enzymatic aqueous extraction improved the quality of sesame oil using a green methodology, free of toxic solvents.

Hydrothermal treatment for enhancing oil extraction and hydrochar production from oilseeds

Popov, S., *et al., Renewable Energy 85*: 844–853, 2016, http://dx.doi.org/10.1016/j.renene.2015.07.048.

A novel integrated oil extraction process that includes hydrothermal pretreatment and oil extraction (HPOE) from whole oilseeds followed by hydrothermal carbonization (HTC) of the extracted seedcake to hydrochar was developed. Five different types of oilseeds including cotton-, flax-, mustard-, canola-, and jatropha seeds were used in the study. The seeds were subjected to hydrothermal pretreatment in the range of temperatures from 120 to 210 °C for 30 min. Oils were extracted from the pretreated seeds using *n*-hexane in a Soxhlet apparatus for 120 min. The crude oil yields from the pretreated seeds at 180 °C and 210 °C were significantly higher (up to 30 wt%) than those from the respective untreated ground seeds. The seedcake after oil extraction was subjected to HTC at 300 °C with the recycled aqueous phase collected from the pretreatment step. The produced hydrochar had higher heating value of 26.5 kJ/g comparable to that of bituminous coal. BET surface area and pore volume analysis showed that the pretreated seeds had larger surface area and pore volume/size than the respective raw seeds, which resulted in better extractability of oil, shorter extraction time, and overall efficiency of HPOE process. Analyses of the crude oil did not show significant signs of degradation after the hydrothermal pretreatment of oilseeds. The study is the first of its kind where integrated oil extraction and hydrochar production from oilseeds have been studied with the objective of minimizing feedstock preparation and maximizing oil extraction and overall energy conversion using environmentally benign hydrothermal processes.

Ultrasound-induced green solvent extraction of oil from oleaginous seeds

Sicaire, A.-G., *et al., Ultrason. Sonochem.*, online 2016, http://dx.doi.org/10.1016/j.ultsonch.2016.01.011.

Ultrasound-assisted extraction of rapeseed oil was investigated and compared with conventional extraction for energy efficiency, throughput time, extraction yield, cleanness, processing cost and

CONTINUED ON PAGE 48

Fats and oils processing in Brazil and Mercosur: trends and challenges

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

Leslie Kleiner

In August 2015, the International Symposium "Processing of fats and oils: trends and challenges" was held in Florianópolis, Santa Catarina, Brazil. The Brazilian Fats and Oils Society (Sociedade Brasileira de Óleos e Gorduras—SBOG) organized the event, which had ~200 attendants from various industries and universities from Latin American and worldwide. To learn more about the topics covered, I interviewed Professor Jane Mara Block, from the Federal University of Santa Catarina (UFSC) and SBOG's President. I also interviewed Dharma R. Kodali, a bioproducts and biosystems engineering professor at the University of Minnesota, Minneapolis, USA, who presented on trans-fats replacement solutions during the event.

Q: What were the main discussion topics for Brazil and Mercosur?

A: (Block): Among the members of Mercosur (Brazil, Argentina, Uruguay, Paraguay, and Venezuela), Brazil and Argentina combined account for 56% of the world's soybean production. Over the last three decades, soybean has become the most important Brazilian agricultural product. Therefore, the conference mainly addressed the oil markets in Brazil and Argentina, as well as applications and challenges. According to Daniel Furlan from ABIOVE (Brazilian Vegetable Oils Industries Association), who presented on "Vegetable oils market in brazil," Brazil is second only to the United States in soybean production, producing 94.4 million metric tons (MMT) a year. Soybean represents 17.5% of total Brazilian exports, and soybean processing leads to a production output of 7.95 MMT of oil and 3 MMT of biodiesel on a yearly basis.

In Argentina, where soybean production has increased from 12 MMT in 1996 to 39 MMT in 2005, soybean was responsible for 50% of the grains produced. Adrian Gomez (President of ASAGA, the Argentine Fats and Oils Association) presented on the oilseeds industry chain in Argentina. According to Gomez, the oilseeds industry chain is the main exporting block and one of the pillars of the Argentine economy, representing 28% of total exports.

Q: What perspectives on trans fats were presented, given that they are not currently banned in the Mercosur region?

A: (Kodali): Based on scientific evidence, trans fats have no nutritional benefit and have been proven harmful to health. Currently, industrially produced trans fats are still allowed in food products. However, new FDA regulations ban the use of partially hydrogenated oils, the major source of trans fats, from food products by the year 2018. Local trans-fats regulations around the world may differ in degree and time, but they are likely to follow suit to FDA regulations on the topic. This paradigm shift provides an opportunity to researchers around the world to find ideal trans fat replacement solutions. Since important functional benefits provided by trans fats include improved oxidative stability (improved shelf-life) and solid fat functionality in foods, finding trans fats replacements that impart both characteristics without increasing saturates content can be a challenge. In order to develop these trans fats replacements, it is key to understand the relationship between structure, property, and functionality of fats. This allows using key ingredients with combination of technologies, such as interesterification and fractionation, to find solutions to the challenge.

Q: What are the perspectives on biodiesel in the Mercosur region?

A: (Block): The two main biofuels in Brazil are biodiesel and ethanol. The raw materials for biodiesel production are mainly soybean oil (over 70%), beef tallow, and cotton. The International Energy Agency (IEA) has projected that the production of biofuels in Brazil is expected to grow over 200%, from 1.3 million to 4.1 million barrels (65 billion liters) by 2035. For the next 20 years, it is estimated that Brazil will almost quadruple its generation of renewable energy and account for 40% of world export of biofuels. However, according to specialists, due to the lack of technological investment in the sector, Brazil is still far from reaching its full potential. Existing equipment and processes for biodiesel production need to improve in order to do so.

Q: What are the perspectives on algae oil in the Mercosur region?

A: (Block): During a symposium presentation, Eelco Blum, product line director from Solazyme Food Oils & Fats, shared that a microalgae refinery (joint venture between Solazyme and Bunge), had been built in São Paulo, Brazil. The oil production from microalgae started in 2014, and the plant's production capacity is 100,000 MMT of microalgae oils per year. The oil, which has high stability and high oleic acid content, can be used for frying, baking, and for applications such as margarines, spreads, sauces, dressings, among others. Consumers in Brazil already have a positive perception about microalgae as a food ingredient (~ 80% of the population have a very positive or somewhat positive opinion). Education on the topic showed to be key to engage and drive acceptance for these kinds of oils.

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.

Tips from inform|connect

Tips from inform|connect is a regular Inform *column that features tips and other discussion highlights from the community forum board at http://www.informconnect.org/home.*

Q: A scientist at a research and development center in Argentina asked, "Which is the best method for determining fatty acids composition in processed foods, primarily cereals, cereals, and prepared egg and vegetable dishes: extraction (or hydrolysis) of fat and methylation, or direct methylation for gas chromatography (GC) analysis?

A: Community members identified direct methylation as the best way to conduct fatty acid methyl ester (FAME) analysis by GC.

A professor at Iowa State University (Ames, Iowa, USA) advised: "If you need to extract fat, make sure to use a correct solvent system that will extract all of the lipids from the food."

Another professor, this one from Laramie, Wyoming, USA, shared his experience: "I have very good results using direct transesterification of fatty acids of the dried sample using BF3 in methanol, which can be purchased already prepared, or using 1.09 N HCL in methanol. The sample must be dry and finely ground. Drying must be done using a lyophilizer. I derivitize with 4 mL of reagent at 85˚C in borosilicate tubes with Teflon-lined caps with frequent vortex-mixing for 60 minutes. Enough sample for 30–50 mg of total lipid would be quite sufficient. Extract in high performance lipid chromatography (HPLC)-grade hexane and transfer hexane phase to GC sample vials. Dry the hexane with anhydrous sodium sulfate.

A senior technology manager at a company that makes nutritional lipids noted that "running a thin layer chromatography before conducting FAME may meet needs for special measurements, such as a free fatty acid profile."

AOCS Chief Science Officer Richard Cantrill wrote: "AOCS recommends the use of either Ce 2b-11 or Cd 2c-11 for FAME preparation in a "one-pot" approach followed by chromatography using Ce 1j-07. All three official AOCS methods have been collaboratively studied and give excellent results when used appropriately. The methods may be found with a useful spreadsheet for calculations in the AOCS Store at http://tinyurl.com/nbko2lw.

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Teruyoshi Yanagita
Bryan V. Yeh
Martin P. Yurawecz

30–34

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Casimir C. Akoh
David J. Anneken
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Philip A. Bollheimer
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—MICHAEL F. COX

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40–44

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45–49

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This list was compiled in January 2016.

50+

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Ram Blau
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LeRoy Dugan, Jr
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Thomas B. Hilton
Robert J. Hlavacek
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US FDA creates dietary supplement office

Josh Long

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

The US Food and Drug Administration (FDA) announced the creation of the Office of Dietary Supplement Programs (ODSP), a development that could bolster the agency's ability to police the dietary supplements industry.

The supplement division was elevated to an office on the recommendation of the US Department of Health and Human Services and following notification to the US Congress.

The new office could raise the visibility of FDA's dietary supplement activities and bolster its ability to target unlawful activity in an industry that has more than quintupled in size since the passage of the 1994 Dietary Supplement Health and Education Act (DSHEA), industry sources said.

"In the 20 years since the establishment of the dietary supplement program, the industry has grown from about $6 billion to more than $35 billion in annual sales," FDA said in a constituent update (http://tinyurl.com/zjdk2hc). "Elevating the program's position will raise the profile of dietary supplements within the agency, and will enhance the effectiveness of dietary supplement regulation by allowing ODSP to better compete for government resources and capabilities to regulate this rapidly expanding industry."

Five trade associations that represent the dietary supplement industry welcomed Monday's development.

"Overall, the industry views the elevation of DDSP not only as a demonstration of the government's commitment to eliminating the illegal activity and leveling the playing field for the responsible companies already following the law, but also as an important step for increasing consumer safety by cracking down on rogue manufacturers who sell illegal products," the associations said in a news release.

In a December 2, 2015 letter to Congress, the associations expressed support for elevating the supplement division to an office. The letter was signed by the leaders of the American Herbal Products Association (AHPA), Consumer Healthcare Products Association (CHPA), Council for Responsible Nutrition (CRN), Natural Products Association (NPA), and United Natural Products Alliance (UNPA).

FDA said it will continue to use its resources to remove from the market dangerous products, enforce cGMPs (current good manufacturing practices), and target "claims in cases involving serious risk of harm to the consumer (such as egregious claims of benefit in treating serious diseases) or widespread economic fraud."

Two Democrats in the US Senate who have criticized the industry said the creation of the dietary supplement office falls short of needed reforms.

"Just as dietary supplements have outgrown their current spot in FDA's organizational chart, dietary supplements have also outgrown the two-decades old law that was supposed to oversee this industry and protect consumers," said Sens. Richard Blumenthal of Connecticut and Dick Durbin of Illinois in a press release. "Comprehensive new legislation is needed to provide the necessary oversight of this $35 billion industry, and we intend to introduce legislation in the new year."

Added the senators: "As the FDA seeks to establish permanent leadership for this new office, we urge the agency to identify a strong leader that will aggressively pursue wrongdoing, vigorously enforce the law, and use all the tools at its disposal to protect consumers and push for increased transparency by the industry on the purity, quality, and identity of the products they manufacture."

A former regulator who oversaw FDA's dietary supplement activities said the creation of ODSP could result in a greater focus on enforcement. The supplement division has been a part of the Office of Nutrition, Labeling and Dietary Supplements. While that office has been focused on policy issues, the statutes governing GMPs—an integral part of FDA's dietary supplement regulations—are "enforcement-minded," explained Daniel Fabricant, Ph.D., executive director and CEO of NPA, in a phone interview.

"That is an enforcement shop," Fabricant said, referring to FDA's dietary supplement activities. "It should be. It shouldn't be buried under, 'Did we eat too much salt this week?' Not that that's not important, but the two shouldn't be the same."

CONTINUED ON PAGE 48

CONTINUED FROM PAGE 46

The creation of ODSP also curtails the risk that labeling and nutrition issues at FDA will overshadow matters that predominantly affect dietary supplements. Considering that supplements are taken by 180 million Americans, the industry should be a priority for the agency, Fabricant said.

Fabricant left FDA in the spring of 2014 to rejoin NPA, and the agency hasn't had a permanent director in its Division of Dietary Supplement Programs since that time. In the constituent update, FDA said it is in the process of identifying permanent leadership for the new dietary supplement office. In the meantime, a long-time FDA official who is trained as a pharmacist and lawyer, Bob Durkin, will serve as Acting Office Director. Durkin is one of a number of FDA officials who have temporarily headed up the division since Fabricant's exodus.

"We are hopeful they will move quickly to name a permanent director," CRN President and CEO Steve Mister said in a phone interview. "I think there is a backlog of things that need to happen in the dietary supplement area that have been kind of stymied for almost the last two years without a permanent director."

For example, the industry has long been awaiting the release of new draft guidance governing new dietary ingredient (NDI) notifications. Under DSHEA, the manufacturer or distributor of an NDI must submit a safety-related notification to FDA at least 75 days before introducing the supplement into interstate commerce.

FDA issued draft guidance on NDIs in July 2011, but the document met strong resistance from the dietary supplement industry. Mister acknowledged he was not surprised FDA hasn't released fresh guidance because he is aware the issue is controversial and was not likely to be tackled by an interim director. A permanent director is likely in a better position than a temporary one to defend a major decision to Congress, industry, and others.

Interim directors, Mister explained, have "less freedom to do the really innovative things that need to be done, the bold kind of decision making."

While he complimented the actions of interim directors who have spearheaded FDA's dietary supplement activities since his departure, Fabricant cited the importance of the agency laying out a concrete agenda, such as a plan for improving compliance with cGMPs.

"The bigger thing now is, let's get a plan folks," the former regulator said.

Josh Long is a senior legal correspondent for Informa (Phoenix, Arizona, USA) and a contributor to Natural Products Insider. *He can be contacted at josh.long@informa.com.*

Reproduced with permission from *Natural Products Insider,* December 21, 2015, http://www.naturalproductsinsider.com/blogs/insider-law/2015/12/fda-creates-dietary-supplement-office.aspx.

CONTINUED FROM PAGE 38

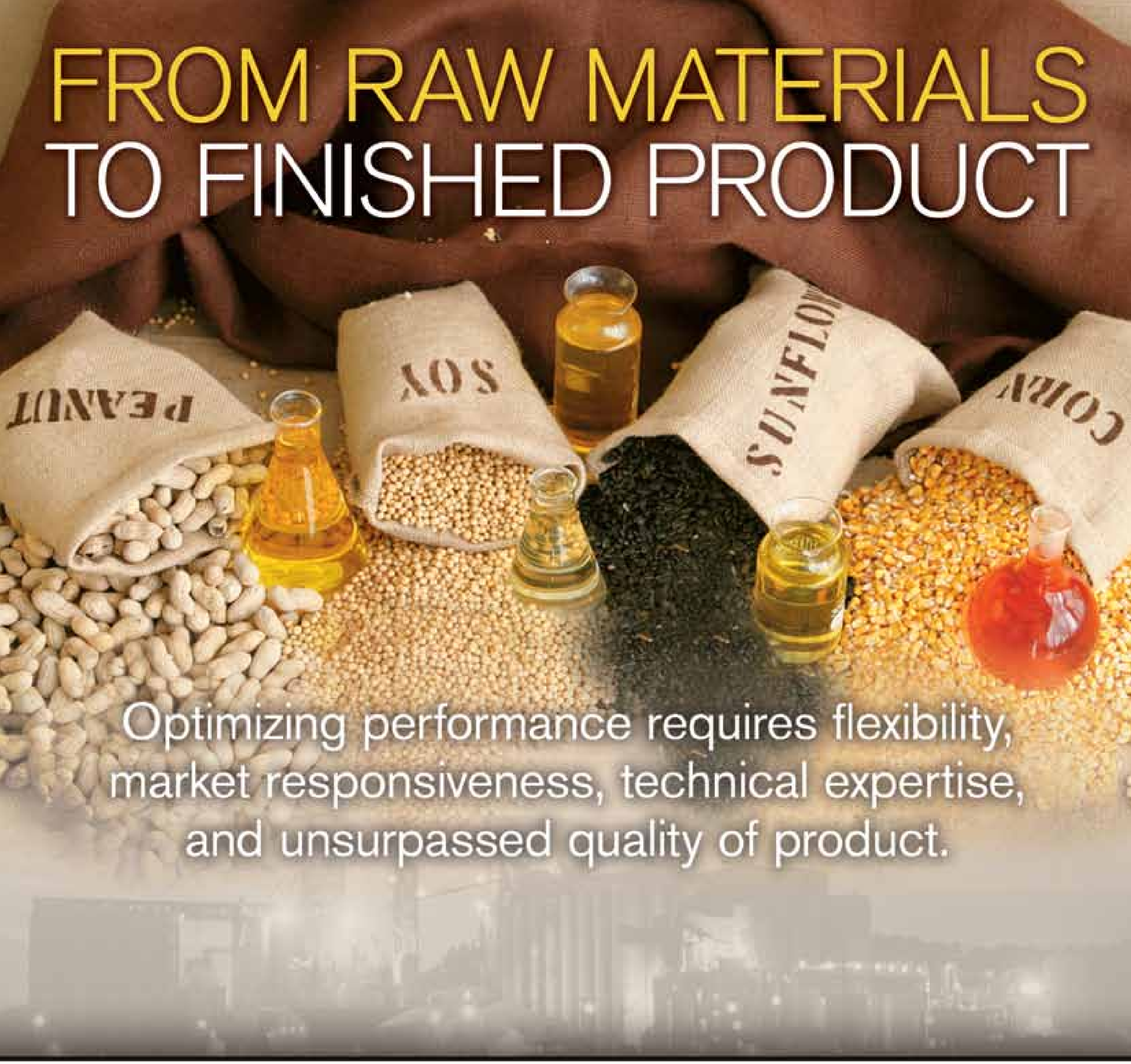
product quality. A multivariate study enabled us to define optimal parameters (7.7 W/cm^2 for ultrasonic power intensity, 40 °C for processing temperature, and a solid/liquid ratio of 1/15) for ultrasound-assisted extraction of oil from oilseeds to maximize lipid yield while reducing solvent consumption and extraction time using response surface methodology (RSM) with a three-variable central composite design (CCD). A significant difference in oil quality was noted under the conditions of the initial ultrasound extraction, which was later avoided using ultrasound in the absence of oxygen. Three concepts of multistage cross-current extraction were investigated and compared: conventional multistage maceration, ultrasound-assisted maceration and a combination, to assess the positive impact of using ultrasound on the seed oil extraction process. The study concludes that ultrasound-assisted extraction of oil is likely to reduce both economic and ecological impacts of the process in the fat and oil industry.

Synthetic Biology

Camelina sativa: An ideal platform for the metabolic engineering and field production of industrial lipids

Bansal, S. and T.P. Durrett, *Biochimie 120*: 9–16, 2016, http://dx.doi.org/10.1016/j.biochi.2015.06.009.

Triacylglycerols (TAG) containing modified fatty acids with functionality beyond those found in commercially grown oil seed crops can be used as feedstocks for biofuels and bio-based materials. Over the years, advances have been made in transgenically engineering the production of various modified fatty acids in the model plant *Arabidopsis thaliana*. However, the inability to produce large quantities of transgenic seed has limited the functional testing of the modified oil. In contrast, the emerging oil seed crop *Camelina sativa* possesses important agronomic traits that recommend it as an ideal production platform for biofuels and industrial feedstocks. Camelina possesses low water and fertilizer requirements and is capable of yields comparable to other oil seed crops, particularly under stress conditions. Importantly, its relatively short growing season enables it to be grown as part of a double cropping system. In addition to these valuable agronomic features, Camelina is amenable to rapid metabolic engineering. The development of a simple and effective transformation method, combined with the availability of abundant transcriptomic and genomic data, has allowed the generation of transgenic Camelina lines capable of synthesizing high levels of unusual lipids. In some cases these levels have surpassed what was achieved in Arabidopsis. Further, the ability to use Camelina as a crop production system has allowed for the large scale growth of transgenic oil seed crops, enabling subsequent physical property testing. The application of new techniques such as genome editing will further increase the suitability of Camelina as an ideal platform for the production of biofuels and bio-materials.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