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October 2021 INFORM

6 New essential dietary lipids?

Odd-chain fatty acids from full-fat dairy and intake of whole grains appear to have health benefits. Are they the next omega-3, and should we start taking supplements?



10 Screening method for quick detection of mineral oil hydrocarbons from lubricants used in the coconut oil supply chain

A new method developed for the quick detection and identification of lubricants in crude coconut oil can serve as an extra control step in identifying sources of contamination and preventing migration.

- **14 Phospholipids as natural emulsifiers in food and beverages** This quick review summarizes the molecular structure, properties, and uses of these important lipids in food, beverages, and food supplements.
- **19 Green processes for phospholipid recovery and fractionation** This article presents three greener alternatives to conventional processing technologies.
- 22 Canola quality by handheld NIR spectrometer Researchers give handheld NIR spectrometers a thumbs-up for predicting some, but not all, of the most important compounds that define canola quality.

DEPARTMENTS

- 5 Index to Advertisers
- 44 Classified Advertising
- 32 AOCS Meeting Watch
- Analysis/commentary
- 28 Olio
- **31** Regulatory Review
- 33 Member Spotlight
- **Publications and more**
- 34 Patents
- **39 AOCS Journals**
- 43 Extracts & Distillates



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New essential Rebecca Guenard dietary lipids?

Milkmen once delivered whole milk door to door; however, this delivery service became less common with each passing generation along with the practice of drinking full-fat milk. Saturated fats were deemed unhealthy when researchers determined that overconsumption increases total cholesterol in the bloodstream, which can result in blocked arteries due to the presence of the more harmful low-density-lipoprotein (LDL) cholesterol.

- Studies involving foods, like whole fat dairy, that contain odd-chain fatty acids show a correlation between good health and how much an individual consumes. There is a similar correspondence with whole grains intake, possibly an effect of odd-chain phenolic lipids in these foods.
- These bioactive compounds show antioxidant and anti-inflammatory capabilities in animal and *in vitro* studies, but clinical studies to determine efficacy and dosing have not yet been conducted.
- Are odd-chain fatty acids the next omega-3s, and should consumers consider taking supplements?

One way for consumers to reduce saturated fat intake was to switch to low-fat dairy products. In the United States, during the late 1970s, the government dissuaded adults from drinking full-fat milk in an effort to prevent heart disease. The World Health Organization established similar nutritional guidelines (https://www.who.int/news-room/fact-sheets/ detail/healthy-diet).

Now, scientists are gathering evidence that beneficial fatty acids may be lost when saturated fats are eliminated from dairy. "Odd-chain fatty acids are a minor component in dairy, breast milk, and some plants," says Farah Hosseinian, food science professor at Carleton University in Ottawa, Canada. They have not been given much attention in the past, but researchers have since realized the compounds are bioactive, she says.

In an effort to remove saturated fats, are we losing essential nutrients? Some researchers think so and are promoting supplementation of odd-chain fatty acids (OCFA) as a way to improve human health and extend life expectancy.

EVIDENCE OF BIOACTIVITY

Current interest in OCFA may have arisen in the mid-2010s, when type 2 diabetes research led to the conclusion that not all saturated fats behaved equally in the body. Researchers analyzed the plasma of a large group of participants across eight countries. They found that higher amounts of OCFA in plasma were associated with a lower occurrence of type 2 diabetes, while more even-chain fatty acids indicated higher risk for the disease (http://dx.doi.org/10.1016/S2213-8587(14)70146-9).

In a commentary for *The Lancet Diabetes & Endocrinology* at the time, Dariush Mozaffarian, cardiologist and professor of nutrition at Tufts University in Boston, Massachusetts, USA, argued that the findings bolster a growing cache of evidence that consuming dairy fat reduces insulin resistance and the chance of developing type 2 diabetes (https://doi.org/10.1016/S2213-8587(14)70166-4).

The majority of milk fat is comprised of the saturated fatty acids myristic, palmitic, and stearic, which range in chain length from C14 to C16 to C18. Oleic acid is the prominent unsaturated fatty acid. OCFA are present in much smaller amounts, even as low as 0.5% of the total fat.

HEALTH AND NUTRITION



Despite being minor components of dairy fat, OCFA continued to pique the interest of scientists, because both pentadecanoic acid (15:0) and heptadecanoic acid (17:0) are good biomarkers for dairy intake. Studies have shown that the concentration of these fatty acids in blood plasma increases when dairy products are consumed.

Researchers have since concluded that OCFA comes from other sources besides ruminant fat, based on measurable levels in the plasma of vegans which is comparable to that of omnivores (https://doi.org/10.1159/000118629). One possibility for the formation of OCFA in the body is through synthesis from propionic acid, generated by the gut microbiome after fermenting dietary fiber. Scientists have suggested other means of endogenous synthesis, but no conclusive pathway has been determined.

LABORATORY AND ANIMAL EXPERIMENTS

To understand why OCFA in blood plasma correlates with positive health effects, researchers have carried out dozens of studies on animals and cellular models in the laboratory. Their goal is to determine the biological function of these fats.

Hosseinian has spent a decade studying phenolic lipids, particularly alkylresorcinols, that have odd-chain fatty acids attached to their rings. The compounds react through a similar mechanism to OCFA. Therefore, her research often involves a comparison of the two. Using a device known as a liposofast, Hosseinian's team produces clusters of liposomes. The particles act as artificial cell membranes that the team uses to study how OCFA and phenolic lipids likely incorporate into human cells. Using a scanning electron microscope, they have observed that the fats incorporate uniformly into the cell membranes, increasing their functionality.

"At the surface of the cell there are lipoproteins, glycolipids, and glycoproteins," says Hosseinian. The OCFA scavenge free radicals, possibly protecting these important biological molecules involved in cell function. "Diseases like Alzheimer's or Parkinson's are related to lipid oxidation or protein oxidation," she says. OCFA could be repairing or preventing damage that might lead to these diseases if left unchecked.

Other research groups found OCFA were less likely to undergo beta-oxidation than even-numbered FA when isotopically labeled fatty acids were fed to mice, perhaps remaining intact to repopulate glycosphingolipids in the brain. In separate studies, mouse models created with impaired mitochondrial function showed improvement when treated with OCFA, indicating their necessity for glucose homoeostasis (Fig. 1, page 8). Finally, further mouse studies suggest that OCFA also serve a role in proper gastrointestinal function (https://doi. org/10.3945/an.115.011387).

A recent study of C15:0 administered to rats and New Zealand white rabbits existing on high-fat diets showed that

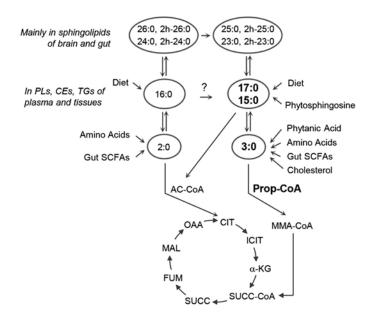


FIG. 1. Research indicates that C15:0 and C17:0 are involved in a number of human metabolic pathways. In particular, the fatty acids are believed to provide intermediates for the citric acid cycle, an important process for energy production in mitochondria. Source: Pfeuffer, M. and A. Jaudszus, *Adv. Nutr. 7*: 730–4, 2016.

after two weeks the animals had overturned the effects of the unhealthy diet, exhibiting lower glucose, cholesterol, inflammation, anemia, and dyslipidemia (https://www.nature.com/articles/s41598-020-64960-y). The same research group studied the effect of C17:0 concentration in the serum of bottlenose dolphins. They found an inverse relationship between C17:0 concentration and the development of a metabolic syndrome similar to prediabetes. When the dolphins were fed fish with higher C17:0 content, the incidence of this metabolic syndrome decreased (https://doi.org/10.1371/journal.pone.0132117).

DEVELOPING SUPPLEMENTS

Despite all the proof that has been gathered so far supporting OCFA status as beneficial to human health, Hosseinian says it is too soon to recommend supplements. She hopes that one day these molecules will be considered essential for the diet just like omega-3s, but says more research is necessary before that can happen. Yes, OCFA exhibit antioxidant activity and prevent inflammation, but she says these findings have only been gathered in laboratory experiments and human clinical trials are still needed.

"The mechanism of action is not as easy as we are making it sound here. Gender, age, polymorphism, many things should be considered before a product comes to market," says Hosseinian.

Not all researchers agree. The lead researcher of the previously mentioned dolphin studies, Stephanie Venn-Watson, claims she has discovered a new essential fatty acid and wants to make it available to consumers (https://tinyurl. com/23uy8ubb). She is now the cofounder and CEO of Seraphina Therapeutics, based in Andover, Massachusetts, USA. The company is focused on creating an OCFA supplement market. The company launched with a fatty15[™] supplement and earlier this year released the food ingredient FA15[™] that can be incorporated into various foods, including nondairy milks.

This summer several media outlets reported FA15[™], a powered form of C15:0, had received generally recognized as safe (GRAS) status (https://tinyurl.com/dfunvws2). However, a search of the US Food and Drug Administration (FDA) database did not produce any record of such a notice being issued. A Seraphina Therapeutics spokesperson clarified that the company self-certified using a panel of experts familiar with the FDA's GRAS requirements.

If omega-3 research is any indicator of the potential success of supplements, then OCFA have many obstacles to navigate before they will be adopted by the food industry. For 30 years, researchers published studies showing a correlation between consuming omega-3s and improved cardiovascular health. Nutritional guidelines for most countries recommend two servings of fish per week to achieve such health effects. However, clinical trials for the development of an omega-3 treatment have faced significant challenges.

Since there have not been any human studies of OCFA, Hosseinian says scientists really do not know how much the body needs or which dietary sources have the highest conversion rates in the body. She says scientists need to ensure that consuming too much does not actually result in negative health effects.

"Overall, we are talking about minor components people get through diet that can affect cell membrane functionality," says Hosseinian. "But these are dose-dependent, and we do not yet know the optimum amount."

The fats and oils community is eager to learn more about these bioactive compounds, and multiple calls for more research into OCFA have been published in scientific journals, including the *Journal of the American Oil Chemists' Society* (https://doi.org/10.1002/aocs.12507). As more researchers take up this challenge, the coming years should provide consumers with the information they need to determine how to get the most health benefits from OCFA.

Rebecca Guenard is the associate editor of Inform *at AOCS. She can be contacted at rebecca.guenard@aocs.org.*

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Control Contro

Efficacy of dietary odd-chain saturated fatty acid pentadecanoic acid parallels broad associated health benefits in humans: Could it be essential? Venn-Watson S., *et al.*, *Sci. Rep.* 10: 8161, 2020.

Pentadecanoic and heptadecanoic acids: multifaceted odd-chain fatty acids, Pfeuffer, M. and A. Jaudszus, *Adv. Nutr. 7*: 730–4, 2016.

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Screening method for Bram Dijke, Lorit Kampoutsi, Raquel de Llanos, Falk Bruse, Carlos Martin-Alberca, and Marian Steverink Mydrocarbons from hydrocarbons from lubricants used in the coconut oil supply chain

- Contamination by mineral oil hydrocarbons (MOH) affects all types of food and feed due to the ubiquitous presence of these compounds.
- Technical lubricants used in edible oil supply chains can be a source of contamination.
- This article describes a new method developed for the quick detection and identification of the presence of lubricants in crude coconut oil that can serve as an extra control step in identifying the source of contamination and preventing MOH migration.

Although the presence of mineral oil hydrocarbons (MOH) in foods and feed has been known since the late '80s, recently there have been substantive advancements in the understanding of this topic. Thus, today MOH are generally recognized as a contamination issue. MOH are a highly complex mix of components, usually categorized into two main groups: mineral oil saturated hydrocarbons (MOSH), which consist of linear and branched (cyclo)alkanes, and mineral oil aromatic hydrocarbons (MOAH), which refer mostly to highly alkylated mono-/poly-cyclic aromatic hydrocarbons.

As more insight into the toxicological aspects and health risks associated with these petrogenic compounds is obtained, authorities worldwide are beginning to establish specifications (i.e., maximum allowed levels). Meanwhile, the food industry is proactively working with every available means to prevent and eliminate the presence of MOH in their raw and processed products. One of the strategies is to localize the sources of contamination and to tackle the root cause of the problem by applying the best manufacturing practices possible. To date, it is known that the main three entry routes of MOH into food are: i) via food contact materials; ii) the use of mineral oil-based food additives and pro-



FIG. 1. Non-food-grade and food-grade lubricants

cessing aids; and iii) unintentional contamination by lubricants or exhaust gases from engines.

In the specific case of the complex and diverse supply chain of tropical edible oils, such as coconut oil (CNO), the technical lubricants used in the crushing and harvesting machinery and transport vehicles for raw materials have been described as possible sources of contamination. Therefore, keeping control on the type of lubricants and how they are used might prevent unintentional contaminations.

The current analytical methodologies available for the analysis of MOSH and MOAH (e.g., DIN EN 16995 by liquid chromatography coupled to gas chromatography and flame ionization detection HPLC-GC-FID, or multidimensional gas chromatography GCxGC-based methods) still struggle to separate, quantify, and accurately identify the different groups of compounds within the two MOSH/MOAH fractions. In the specific case of edible oils, the challenge is even greater because of the chemical similarity of MOSH/MOAH and the naturally plant-derived hydrocarbons like odd-numbered n-alkanes or terpenoids. In addition, the sample preparation strategies are very demanding and complicated in the case of edible oils. These difficulties highlight the need for a new method for quickly detecting the presence of these contaminants in food samples and identifying the source of contamination.

Our research team is proposing an alternative analytical methodology that would allow the presence of technical lubri-

TABLE 1. Some examples of the lubricants analyzed in this study, as well as the use of each lubricant and their grade

Lubricant's code	Туре	Food grade	Non- food- grade
001	Compressor oil	Х	
002	Expeller/Conveyor		Х
003	Compressor oil	Х	
004	Gear oil	Х	
005	Expeller		Х
006	Mobil equipment		Х
007	Engine oil		Х
008	Conveyor		Х
009	Used engine oil		Х

cants in CNO to be detected quickly. This method is based on a simple sample preparation approach, and a screening and chemical fingerprinting analysis by GPC-HPLC-Fluorescence/ ELSD (gel permeation chromatography—high-performance liquid chromatography-fluorescence detection/evaporative light-scattering detector). It aims to detect contamination while also identifying the implicated lubricant, which would

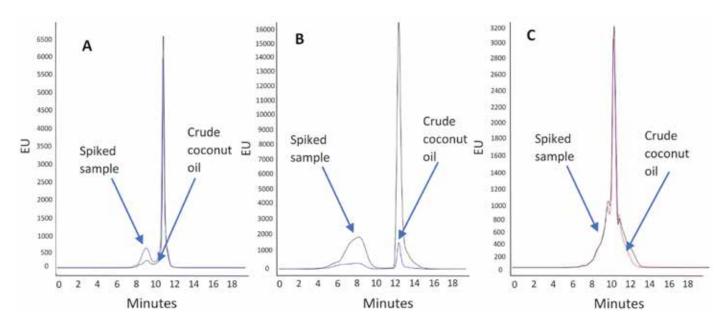


FIG. 2. GPC-fluorescence chromatograms at ex260nm/em330nm wavelength showing the effect of the three different sample preparation approaches on pure CNO samples and CNO samples spiked with one of the lubricants at 50 mg/kg; A. Sample prep method #1; B. Sample prep method #2; C. Sample prep method #3.

facilitate the determination of the source and prevention of the contamination.

Up to 30 different technical and food-grade lubricants were studied. Table 1 (page 11) shows a list of some of the lubricants analyzed, together with a short description of their use along the supply chain. Figure 1 (page 11) shows a few jars containing some of those lubricants. In addition, homemade cold press, contaminant-free CNO, and crude and refined CNO were analyzed to obtain their chemical profiles. These CNO samples were spiked with the lubricants at several concentrations (e.g., 50 mg/kg). After that, the obtained profiles from the contaminated samples were compared to ones from the non-contaminated samples.

In the first step of the study, three different sample preparation methodologies were examined to select the most suitable approach to be used together with the quick (i.e., 15-min run) screening method by GPC-Fluorescence/ ELSD for the separation of the MOSH/MOAH fractions from the triglycerides and other matrix compounds. The time needed to perform each method in its entirety and its simplicity were assessed.

As can be seen in Figure 2, the combination of the three sample preparation approaches with the screening method showed different results. Figure 2A shows that the screening method is able to separate two main peaks of the CNO sample. In addition, it can be observed that the spiked sample with a lubricant at 50 mg/kg shows a clear increase in the response of the first peak. The sample prep method #1 is one of Cargill's internal methods usually used for fatty acid methyl ester (FAME) analysis. The sample preparation time is 15 min, and it permits the recovery of both MOSH and MOAH compounds. Figure 2B shows an even clearer increase in the two peaks of the spiked sample. In this case, the sample prep method #2 consists of a simple dilution in heptane, which takes 2 min in total. This method focuses more on the MOSH compounds, although it is also able to recover part of the MOAH compounds. In Figure 2C, method #3, does not show a clear increase in the chromatogram of the spiked sample. Method #3 is another of Cargill's internal methods which concentrates on the MOAH compounds through the use of several solvents and clean-up steps that remove most of the interferences. Here, it should be mentioned that this methodology takes up to 90 min in total.

The results of this portion of the experiment demonstrated that the first two sample preparation methods can be useful to quickly screen CNO samples, aiming to discover a lubricant contamination.

In the next step, 2-minute chromatographic fractions of interest from the GPC separation were selected and directed to the HPLC column. The aim in this case was to obtain characteristic profiles of each lubricant by the so-called fingerprinting analysis based on HPLC with fluorescence and ELSD detectors. This part of the analysis takes up to 120 minutes (including re-equilibration time). However, this methodology is able to separate a large variety of peaks that might allow the profiling of the samples. Figure 3 shows the HPLC separation of the fraction eluting between minutes 8 and 10 (first peak in Figure 2A). As can be observed in Figure 3, the spiked CNO sample shows several peaks belonging to the lubricant at about minutes 55 and 65 that are not present in the non-contaminated CNO sample. When comparing this chromatogram with the one obtained with the sample prep method #2, it was observed that there were a lot of matrix peaks. The region between min-

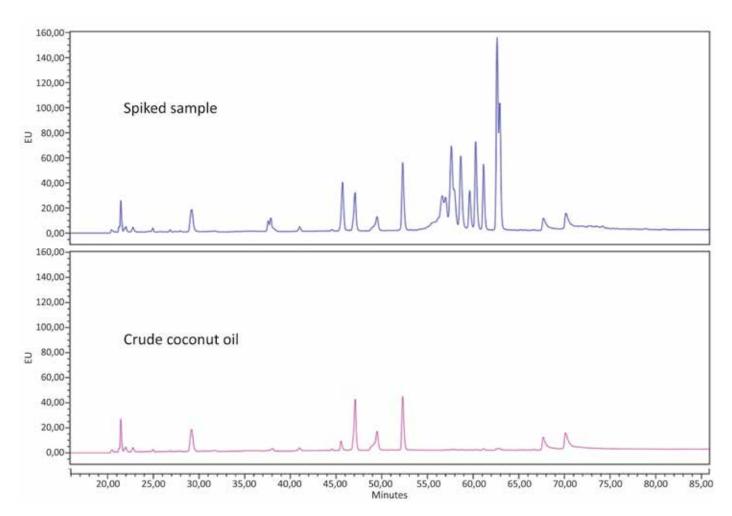


FIG. 3. Fingerprinting method by HPLC-Fluorescence/ELSD. These fluorescence chromatograms show the content of the GPC fraction between 8–10 min of a CNO sample and a CNO sample spiked with lubricant (at 50 mg/kg). Fluorescence detector conditions: Ex260nm/Em330nm wavelengths.

utes 55 and 65 showed a similar cluster of peaks, but with lower intensities and worse resolution (data not shown). Therefore, the sample preparation method #1 was selected as the best method to proceed with the analysis of the rest of lubricants.

All in all, this combined screening and fingerprinting methodology seems to enable the quick determination of CNO contamination by a lubricant, while at the same time it is a promising tool for obtaining unique chemical profiles of lubricants that would allow their identification in a coconut oil sample. The results presented above confirm the suitability of this technique for the detection of lubricant-derived MO contamination in crude coconut oil, and the prospects of its application for the determination of the source of lubricant contamination along the supply chain.

Bram Dijke, Lorit Kampoutsi, Raquel de Llanos, Falk Bruse, Carlos Martin-Alberca, and Marian Steverink are researchers at Cargill Global Edible Oils Solutions, Europe, R&D. Carlos Martin-Alberca can be contacted at Carlos_Martinalberca@cargill.com.

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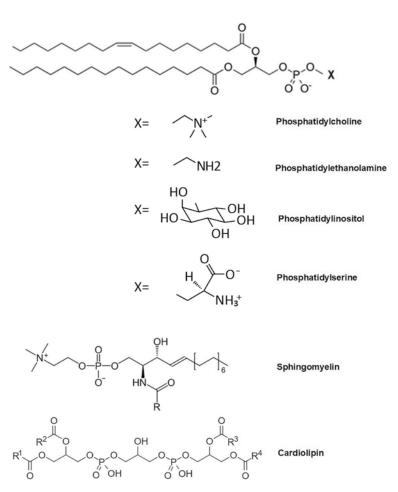
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Phospholipids as Ernesto M. Hernandez natural emulsifiers in food and beverages

Phospholipids, the main components of lecithins, are basically comprised of a phosphorus moiety, a polar and a non-polar portion attached to a glycerol or a sphingosyl backbone. This type of molecular structure makes them a special type of lipid with very important roles in many biological processes. The more common glycerol phospholipids are built up from glycerol, fatty acids, and a phosphate group. The phosphate group can be esterified with choline, ethanolamine, serine, or inositol (Fig. 1). The functionality and variety of phospholipids are reflected in a multitude of applications in food, cosmetics, and pharmaceuticals.

- Phospholipids play significant roles in numerous biological processes.
- They also have many applications in food, cosmetics, and pharmaceuticals.
- This article reviews the molecular structure, properties, and uses of these important lipids in food, beverages, and food supplements.



WANT MORE?

Ernesto M. Hernandez was an invited speaker at the 2021 AOCS Annual Meeting & Expo. His presentation, "New Modified Phospholipid for Stabilization of Beverages," can be viewed at https://www.eventscribe.net/2021/AOCS/ SearchByBucket.asp?f=TrackName&pfp=BrowseByBucketCustom3.

FIG. 1. Main structures of phospholipids

PHOSPHOLIPIDS

Lecithin is a relatively new ingredient in the human diet; it was discovered in the mid-19th Century from carp eggs and was later extracted from other animal products. Lecithin from egg yolk first started to be applied in food products in the beginning of the 20th Century and found wider global food applications with the introduction of soybean lecithin in the mid-1900s (Table 1). Most lecithins are obtained from the degumming of vegetable oils, such as soybean, sunflower, and canola. Degumming is usually the first step in processing vegetable oils, and it involves adding water to the crude oil to hydrate the more hydrophilic lecithin, which then separates into a distinct phase that can be removed by centrifugation. The resulting lecithin from the degumming step is usually a mixture of acetone-insoluble polar lipids and triacylglycerols, together with other minor components. Another minor commercial source of lecithin is derived from egg yolk.

The gums or wet lecithin resulting from centrifugation contain approximately 50% water, which must be quickly removed to avoid microbial spoilage. Bleaching agents such hydrogen peroxide are usually added before drying to less than 1% moisture. Commercial lecithins have been classified into at least six different grades, each of which is divided into natural, refined, and chemically modified categories. De-oiled lecithin is produced by removing residual oil with a solvent, usually acetone. It can also undergo alcohol fractionation to produce a fraction high in phosphatidylcholine (alcohol-insoluble) and a fraction high in phosphatidylinositol (alcohol-soluble). Commercial chemically modified lecithins include hydrogenated, hydroxylated, acetylated, sulfonated, and halogenated products The typical composition of commercial lecithin is shown in Table 2.

TABLE 2. Typical composition of commercial lecithin

Soybean Oil	34%
Phosphatidylcholine	17%
Phosphatidylethanolamine	15%
Phosphatidylinositol	11%
Phytoglycolipids	17%
Carbohydrates	5%
Moisture	1%

Lecithin is considered GRAS (generally recognized as safe) by the US Food and Drug Administration (FDA) under 21 CFR 184.1400. Chemically modified lecithins require special labelling; i.e., if hydroxylated, lecithin should be identified as "Hydroxylated Soy Lecithin" or "Hydroxylated Lecithin". When enzymatically modified, the phrase 'Enzymatically Modified Lecithin" should be included on labelling. These modifications with phospholipases are usually done to

TABLE 1. Phospholipids timeline

1850: The term lecithin (from the Greek lekithos or "egg yolk") is coined by T.N. Gobley from his work on a "phosphorus-containing substance" from carp eggs.

1884: J.L.W. Thudichum introduces the term phospholipids in the treatise, *Chemical Constitution of the Brain*: "Phospholipids are the centre of life, and chemical soul of all bioplasm..."

1902: Patent is issued to Reeser Margarine Fabrik on the use of lecithin to improve margarine properties.

1908: Egg lecithin is widely commercialized in Europe. The malted dairy drink Ovaltine is introduced with the slogan: "...contains active lecithin, the best brain and nerve tonic known". A patent is issued to H.C. Buer for the recovery of lecithin from oilseeds.

1925: Large-scale production of commercial of soy lecithin begins in Europe. De-oiled lecithin is introduced.

1925: H. Bollmann patents "Process of increasing the durability of pure salad or sweet oils" using lecithin (antioxidant properties of lecithin).

1929: Use of lecithin is introduced to improve the texture and appearance of chocolate. Process to bleach lecithin using hydrogen peroxide is introduced.

1930: American Lecithin Co. is incorporated in the US.

1934: ADM starts large-scale production of soy lecithin in the United States. Main applications are in chocolate and margarine manufacture.

1954: Introduction of centrifuges to separate the gums from soybean oil appreciably increases global production of lecithin.

1965: Bangham, *et al.*, first describe liposomes in "Diffusion of univalent ions across the lamellae of swollen phospholipids". This is the first feasible model for biological cell membranes.

1990s: Researchers gain a better understanding of the roles of phospholipids in many metabolic processes.

2000s: New phospholipid-based nutritional supplements and bioactive delivery systems (i.e., structured phospholipids, krill oil, micro and nano particles) are introduced.





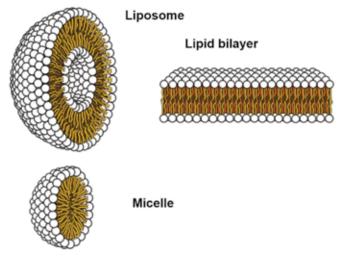


FIG. 2. Liposomes, bilayers, and micelles

modify emulsification properties and improve water dispersibility. More recently, phospholipases have been introduced in the degumming stage to facilitate the separation of phosphatides from the oil that is being refined.

PROPERTIES OF PHOSPHOLIPIDS

The amphiphilic character of phospholipids, which originates from their bipolar molecular structure, gives them a tendency to form bilayers, micelles, and liposomes (Fig. 2). Consequently, they find many applications in food, cosmetic, and pharmaceutical products as natural emulsifiers, wetting, and dispersing agents.

The tendency of phospholipids to form bilayers and liposome structures is used industrially to encapsulate specific food and beverage ingredients, such as flavors, bioactives, and other unstable food ingredients that might need protection against degradation. The dispersion and emulsion stabilization properties of lecithins have been directly associated with the charge of the phospholipids, as well as the composition of the fatty acids. The emulsification properties of lecithins are often characterized by their hydrophilic-lipophilic balance (HLB) value. This value varies between 1 and 20, where 1 is assigned to a surfactant that is oil-soluble and 20 is for emulsifiers that are completely soluble in water. Typical lecithins with 62% acetone-insolubles have an HLB of approximately 4. Oil-free or granulated lecithins have an HLB of about 7, and chemically modified lecithins, such as hydroxylated lecithins, have HLB values of more than 10.

These unique bioactive properties of phospholipids make it possible to design specific products for specific nutritional and pharmaceutical applications, such as the widely reported therapeutic uses of lecithin and specialized phospholipids in the prevention or treatment of neurochemical and cardiovascular disorders and other medical applications. The physiological properties of the major phospholipids are listed in Table 3. Moreover, phospholipids may provide an effective and versatile delivery system for other functional compounds, such as essential fatty acids, thereby increasing their bioavailability and chemo-preventive effects.

TABLE 3. Physiological properties of the major phospholipids

Phospholipid	Physiological properties
PC	Structural component of cell membranes, synthesis of neurotransmitters
PE	Nerve tissue structure, important role in membrane fusion
PS	Nerve cell processes, regulation of nerve impulses, apoptosis and platelet clotting
PI	Important role in the process of transmis- sion of messages in neural system
CL	Found in mitochondrial membrane, important role in mitochondrial bioener- getics processes
SM	Key components of nano domains in cell membranes, part of functional lipid rafts, role in membrane transport, structure and signal transduction.

Phospholipids are now being used to manufacture modified liposomes in combination with other surfactants, such as glycerol and mono- and diglycerides, for the delivery of bioactive lipids and other pharmaceutical and medical compounds.

PHOSPHOLIPIDS IN FOOD AND BEVERAGES

Lecithins are currently used in a wide range of food products and supplements as emulsifiers, stabilizers, control crystallization agents, viscosity modifiers, antioxidants, and reducers or replacers of fat. Interestingly, the earliest food applications for lecithin were in nutrition-oriented beverages. In 1908, egg lecithin, the only source at the time, was widely commercialized in Europe in popular nutritional supplement products like Ovaltine, which was advertised as a food drink made with eggs containing "active lecithin, the best brain and nerve tonic known".

Later, as more efficient processes were developed to extract lecithin from soybean oil, other applications were introduced. For example, in 1925, the use of lecithin to improve the rheological properties and appearance of chocolate was introduced. Another early application introduced in Europe was the use of lecithin in refined oils to prevent rancidity, prevent oxidation, and help preserve the quality of edible oils. Since then, the use of tocopherols and other antioxidants have made this application less popular.

Large-scale production of soy lecithin in the United States began in the 1930s with the introduction of hexane extraction of soybean oil. The main applications of these soy lecithins included stabilization of chocolate and preparation of the oil in water emulsions for margarine production.

Generally, the most widely used applications of lecithins in foods are as emulsifiers that assist in blending immiscible fluids and help in generating stable emulsions or dispersions. Common equipment used to generate these stable emulsions include high-speed blenders, high- and low-pressure homogenizers, and colloidal mills. Lecithins are utilized in both water-inoil emulsions (w/o) and in oil-in-water emulsions (o/w). Typical examples of applications of w/o emulsions with lecithins include dairy-based beverages, infant formulas, mayonnaise, and salad dressings. Water-in-oil emulsions in foods stabilized with lecithin include margarines, low-fat spreads, icings, and frostings.

Lecithins also play an important role as wetting and instantizing agents for food powders used in beverages. For example, food processing that involves dispersion of low-fat powders will require the use of lecithins with a low HLB value. Powders with a higher fat content will need a lecithin with a higher HLB to enable better dispersion in an aqueous system. Lecithins have also found applications as emulsifiers as well as viscosity modifiers in confectionery products, such as chocolates, caramels, and coatings. They are also used to control crystallization and improve the plasticity of several food products, as well as stabilizers and emulsifiers in many other food products, such as baked goods, cheeses, meat and poultry products, dairy, and imitation dairy products. Typical levels of lecithins used in foods vary between 0.2 and 1.0%. Table 4 shows some food and beverage applications of lecithins and phospholipids.

PHOSPHOLIPIDS AS FOOD SUPPLEMENTS

In the last few years, some phospholipids in foods and beverages have been studied for their bioactive properties. For example, it has been reported in human clinical trials that consumption of phospholipids is linked to improvements in cognitive performance and mood, especially during stressful times. Specifically, phosphatidylserine, used in several dietary supplements, has been reported as a potential treatment for Alzheimer's disease and other memory problems. Other studies with phosphatidylserine have shown improved cognitive abilities and behaviors. PS has also been shown to enhance cognitive function in the elderly.

The FDA has authorized two health claims for PS: "Phosphatidylserine (PS) may reduce the risk of cognitive dysfunction in the elderly" and "Phosphatidylserine (PS) may reduce the risk of dementia in the elderly". Phospholipids can also be restructured to enhance the biological activity of other functional lipids, such as conjugated linoleic acid and phytosterols.

Recently, a dairy product high in phospholipids was introduced into the market by Fronterra-NZMP. This product is being advertised to help manage the effects of stress, as a mood enhancer, and as a cognitive performance agent. It is marketed as a dairy-derived ingredient with a high concentration of milk phospholipids for applications in food and beverages, such as dairy-based beverages, nutritional bars, and ready-to-mix powders. Phosphatidylserine and sphingomyelin have been identified as the main components in these dairy phospholipids. The reported beneficial effects include balancing stress by reducing the release of the stress hormone cortisol and also stimulating the production of key neurotransmitters, such as acetylcholine, as well as improved nerve signaling.

A considerable amount of research has been published on the health benefits of the long-chain omega-3 fatty acids eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), such as protection against cardiovascular disease and prevention of

TABLE 4. Applications of Phospholipids in foods and beverages

Application	Functionality
Baked goods	Volume improvement Dough conditioner Fat dispersion Anti-staling Egg Yolk replacer
Chocolate	Viscosity modification Water uptake Ice coatings
Instant drinks	Wetting
Dairy	Dispersability
Cocoa/Chocolate	Dispersability Fat reducer Reduce viscosity Inhibit bloom
Margarine	Anti-spattering Emulsification Improve spreadability
Baking-pan release products	Anti-sticking
Flavors	Encapsulation
Release agents	Separation Wetting
Confectionary	Anti sticking
Vegetable Oils	Antioxidant

inflammation-related illnesses. Also, long-chain omega-3 DHA is generally associated with cell structure, particularly in neurologically related metabolism, such as brain and retina development. As a result, many foods, including several beverage products, are fortified with these fatty acids through the manufacture of emulsions that include the use of lecithin as one of the emulsifying ingredients.

In recent years, it has been suggested that a more beneficial way to deliver and metabolize long-chain omega-3s would be to administer them in their phospholipid form. Published research seems to indicate that long-chain omega-3 as phospholipids are more effective compared to either their triacylglycerol or ethyl ester forms. Research suggests that these omega-3 phospholipids seem able to cross the brain blood barrier more efficiently and substantially, where they increase brain polyunsaturated fatty acid (PUFA) and phospholipid content, thus providing additional benefits that can improve cognition and help prevent neurological disorders.

CONCLUDING THOUGHTS

The field of phospholipids research is rapidly moving from their use as emulsifying and texture improving agents to their use as bioactive agents, as well as in the stabilization and delivery of bioactive systems. As the demand for fortified foods and beverages with bioactive compounds continues to grow, this new generation of health-oriented foods and delivery systems will require more accurate analytical methodologies for quality assurance and product validation. In addition, the new fields of study they inspire, such as the use of nanotechnology for the production of lipids and phospholipid nanoparticles, may require new analytical techniques and implementation of appropriate regulatory and labeling guidelines.

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Green processes for Nurhan Turgut Dunford phospholipid recovery and fractionation

Phospholipids (PL) are the main components of all biological membranes. Their chemical structure is similar to that of triacylglycerides (TAG), except PL have a phosphorous group on the third carbon rather than three fatty acids on the glycerol backbone. Because of this amphiphilic structure, having two hydrophobic fatty acids and a hydrophilic phosphate group, PL play vital physiological roles in lipid bilayers and can be used to formulate many useful products. Indeed, they are commonly used as emulsifiers and surface-active wetting agents in many products, including foods, pharmaceuticals, and cosmetics. Various health benefits of PL, such as enhancements in brain functions and cardiovascular health, have also been reported (Küllenberg, *et al.*, 2012).

- Conventional phospholipid (PL) recovery and purification techniques use large volumes of fresh water, organic solvent, and alcohols.
- Due to diminishing fresh water resources and concerns about the use of organic solvents, the development of more environmentally benign processing techniques is vital to the sustainability of PL production.
- This article considers three greener alternatives to conventional processing technologies.

Glycerophospholipids [i.e., phosphatidylcholine (PC), phosphatidylethanolamine (PE), phosphatidylinositol (PI), phosphatidic acid (PA)] and sphingophospholipids [i.e., sphingomyelin (SM)] are the two subgroups of PL. Depending on the source, the amount of each group and composition of individual PL can be very different, affecting functionality of PL. This article focuses on oilseed-based PL.

Glycerophospholipids are the major PL in oilseeds. They are extracted along with neutral lipids, TAG, during oilseed processing. PL are not desirable in refined oils for several reasons. They impart a cloudy appearance and tend to precipitate out of crude oil during storage and transportation, making the oil handling quite difficult. Due to their emulsifying properties, they interfere with downstream processing and refining of crude oil and cause emulsion formation and foaming during cooking, such as frying. Hence, PL are separated out of crude oil during the first stage of refining in a process called degumming, which refers to the gummy consistency of PL.

The amount and composition of the PL vary significantly depending on the type of oilseed. For example, peanut (45–50% oil) and cottonseed (18–20% oil) oils contain relatively lower amounts of PL in crude oil (less than 0.5 and 1%, respectively) than those in soybean (18–20% oil) and canola (40–45% oil) oils, up to 3 and 4%, respectively. Canola PL have higher PC content, about 85% of total PL, than that of sunflower- and soybean-based PL. Both high oil and PC contents make canola a good choice for preparing PC-enriched PL fractions. Dietary PC supplementation is associated with enhanced memory and liver health (van der Veen, *et al.*, 2017). For edible applications, the non-allergenic nature of canola seeds is also an advantage over soy-based PL.

PL are separated from crude oil through water or acid degumming. Although hydratable PL can easily be removed through water degumming, nonhydratable PL separation requires acid degumming. Crude lecithin, a mixture of PL and neutral oil, is a byproduct of the water degumming process. Soybean oil-derived crude lecithin is usually further processed to produce food-grade lecithin and has been the most popular emulsifier for food applications. However, canola- and sunflower-based PL are gaining market share due to their non-allergenic properties.

Conventional PL recovery and purification techniques use large volumes of fresh water and organic solvent (i.e., acetone for de-oiling crude lecithin and alcohols for fractionation and enrichment of individual PL). Considering the diminishing fresh water resources and concerns about the potential adverse impacts of organic solvents on environment and human health, development of more environmentally benign processing techniques are vital for the sustainability of the PL production industry.

Commercial enzyme producers are responding to the need for sustainable oil and oilseed processing techniques by developing new enzymes that could potentially reduce fresh water and organic solvent use. Indeed, enzymes for de-oiling crude lecithin and degumming crude oils are commercially available today. Lipases are particularly effective in degumming oils with high non-hydratable PL content (Al Sharqi, et al., 2014; Al Sharqi, et al., 2015a, 2015b). It is important to note that enzymatic processes break up the PL structure and produce lysophospholipids, limiting their applications to oil-in-water type emulsifiers. Only a few studies on the recovery of PL and other valuable products from enzymatic oil processing byproducts have been reported (Xie and Dunford, 2015, 2016, 2017). Further research is needed on the technical and economic potential of recovering high-value products from the by-products generated during enzymatic degumming.

Supercritical fluid (SCF) technology is an alternative to conventional processes and has the potential to reduce the adverse environmental impacts of oil and oilseed processing. The latter technique uses fluids, usually gasses, above their critical temperature and pressure, as solvent for processing. A unique feature of the SCF is that physical properties, such as the viscosity and density of the fluid, can be adjusted by simply changing the temperature and pressure of the system. This allows selective extraction of the desired components from a mixture. Solvent separation from the final product can be achieved by lowering the pressure to a level that solvent returns to the gas phase, leaving no solvent residue in the extract.

Supercritical carbon dioxide (SC-CO₂) offers several options for oil, oilseed, and lecithin processing. SC-CO₂ is an excellent solvent because it is readily available, inexpensive, has low critical temperature and pressure, and is non-toxic,

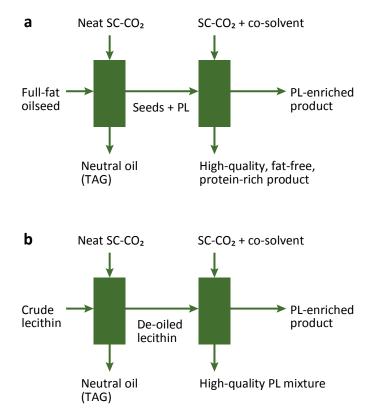


FIG. 1. Proposed SC-CO₂ processing options for phospholipid processing

non-flammable, and non-explosive. SCF technology, specifically SC-CO₂, has been utilized for PL recovery and lecithin fractionation (Dunford and Temelli, 1995; Temelli and Dunford, 1995, 1996). Since SC-CO₂ is a non-polar solvent, it is an excellent solvent for extraction of neutral lipids such as TAG from oilseeds, but it has low affinity to relatively polar PL. Hence, a two-step low temperature (lower than 50°C) process involving extraction of TAG from oilseeds using neat SC-CO₂ first, and then, recovering residual PL by adjusting the polarity of SC-CO₂ by adding a co-solvent, i.e. ethanol, produces two high-quality products: neutral lipids and PL concentrates (Fig. 1 a). Final products will have light color and no solvent residue. A similar two-step process can also be applied to crude lecithin. First, crude lecithin can be de-oiled using neat SC-CO₂, then, ethanol can be added to the system as a co-solvent to obtain a high-quality PC-enriched fraction (Fig. 1 b).

Obviously, the high capital cost of SCF technology would limit the applications of this technique to commodity products. However, SC-CO₂ could be a viable option for production of high-value and purity products for pharmaceutical and cosmetics industries.

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Canola quality V.J. Barthet, M.W.P. Petryk, and B. Siemens NIR spectrometer

- Handheld NIR spectrometers can be used to predict some of the most important compounds that define canola quality (high oil and protein contents, low glucosinolate and chlorophyll contents, and fatty acid composition).
- It is important to test models on true external verification sets instead of relying only on cross-validation for model stability.
- For handheld NIR spectrometers, the compact design necessarily has small sample windows, necessitating multiple measurements in intact seeds to reduce sampling error. Ground/milled seeds or flour samples have lesser sampling effect.
- It is important to understand how the limited wavelength range of some handheld NIR spectrometers makes NIR spectrometers unsuitable for the prediction of certain quality parameters.

Canola seeds are crushed to produce a protein-rich meal and a healthy edible oil. Canola seed quality is defined by: (1) oil content, the higher the better for crushers; (2) protein content to produce a good quality meal; (3) low glucosinolate content to be a true canola seed and not a rapeseed (<30 micromole/g of oil-free meal); (4) low content of chlorophyll, an unwanted component that is removed during oil processing; and finally (5) fatty acid composition, some canola varieties (low linolenic acid, or LL, and high oleic acid-low linolenic acid, or HO-LL) have been selectively bred to produce oil with high thermal stability (ideal for frying applications). All these analytes can be measured by well-known chemical reference methods.

Near-infrared (NIR) spectrometry is commonly used by the agriculture and food industry to predict quality parameters of major and minor components of various food products, especially grains such as wheat and oilseeds (Daun, *et al.*, 1994; Williams and Norris, 2001). Various types of benchtop NIR spectrometers (NIRs) can be found on the market with variable wavelength ranges, which include NIR (750–2500 nm) and visible range wavelengths (400–750 nm). Handheld NIR spectrometers are a relatively new technology (Béc, *et al.*, 2020) developed for on-thespot analyses to either fight fraud or assess product quality and authenticity. These on-the-spot analyses limit the sample processing that can be made (e.g., grinding). Portable handheld NIRs usually have smaller wavelength ranges compared to benchtop instruments (Fig. 1).

For all NIRs, the obtained scan is an average of several scans (20– 100) as defined by the user or the manufacturer. The most important difference between the two types of instruments is how the scans are collected. Benchtop instruments that are specialized for use in the agriculture and food industry typically scan the sample at several different points (set by the user/manufacturer ranging from 10–100 scans). However, with handheld NIRs the scanned part of the sample is limited to the window size of the instrument (1–2 centimeters wide), where sample points are selected by the positioning of the handheld NIRs. This

ANALYTICAL TECHNOLOGIES



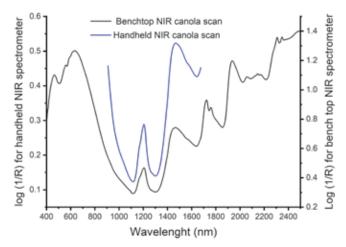


FIG. 1. NIR scan of intact canola seeds obtained by benchtop NIR spectrometer and handheld NIR spectrometer

can result in an important sampling effect associated with the handheld NIRs, especially with intact seeds.

Sampling effect can be reduced by taking replicate scans; however, the number of replicate scans is highly dependent on the type of sample, with the most important factor being the fraction of NIRs window in contact with the sample under analysis (the greater the contact area, the higher the signalto-noise ratio of the spectra). When we analyzed intact canola seeds, the optimal number of replicate scans to produce an average scan used for prediction and verification was five. This number jumped to 25, when we tried to develop models for intact soybean seeds. This is well understood in terms of seed size—intact canola seeds are 1.5–2.5 mm in diameter versus 5–11 mm diameter for intact soybean seed, which means that multiple seeds are in contact with the NIRs window in the case of intact canola seeds versus virtually a single seed in the case of intact soybean seeds.

DEVELOPING NIR MODELS

Developing NIR models requires running statistical predictions to associate the NIR spectra with the chemical data of the samples (obtained by traditional reference methods). It is important to have a good calibration sample set, but it is also essential to test these models with good external verification sets. Performing only a cross-validation is not enough to really test any prediction model and its robustness.

Models were developed using a calibration set containing samples collected from various Canadian canola growing locations over two harvest years. This was done to have not only compositional ranges but also location and year variations. Once developed and verified using cross-validations, the NIR models were then tested against a first external verification set. This external verification set included samples from the same year and location as the calibration set but were independent of each other. This first external verification set served as an important first step. If the models did not successfully predict the quality of these similar samples; it would likely not predict any other external samples properly. Finally, to assess the models' stability, the models were tested against a true external verification seed set containing samples that had no relation (year and location) with either the samples in the calibration or the first verification set.

Descriptive statistics for the canola seed composition (i.e., oil, protein, total glucosinolate, chlorophyll content, main fatty acids, iodine value, and total saturates) are presented in Table 1 (page 24) for all the canola sample sets (calibration and both external validation sets). As environmental factors influence the quality of the harvested canola seeds, it could happen that the quality parameters of a particular year could be out of range with the current calibration dataset, as was the case for our first verification set (Table 1). This can lead to situations where the calibration models must extrapolate the predictions

	Oil	Protein	Chlorophyll	Total Glucosinolate	Oleic Acid	α-Linolenic Acid	Total Saturates	Iodine Value of Oil
Units	%, dry basis	%, dry basis	mg/k, as is	µmol/g of seed, dry basis	%, in oil	%, in oil	%, in oil	Units, in oil
Calibration set (2016 & 2017 intact canola seed samples)								
Mean	48.7	22.2	13.8	11.3	63.9	8.52	6.66	111.1
St. Deviation	2.5	2.2	11.9	3.6	3.8	2.66	0.29	5.5
Median	48.8	22.1	11.1	10.5	62.8	9.35	6.63	112.8
Minimum	41.9	16.5	1.0	4.9	56.6	1.49	5.41	90.8
Maximum	54.9	28.5	96.8	23.7	79.4	12.27	8.01	121.9
Ν	181	168	167	77	181	181	181	181
External valida	tion set (2017 i	ntact canola see	ed samples)					
Mean	48.2	23.5	19.1	11.1	64.2	8.22	6.69	110.3
St. Deviation	3.8	3.8	24.0	4.2	4.8	2.91	0.30	6.4
Median	48.1	22.9	11.4	10.3	63.0	9.12	6.76	112.2
Minimum	40.3	16.4	1.5	4.4	54.3	1.68	5.97	94.2
Maximum	57.0	30.9	132.7	22.1	79.5	12.10	7.39	118.3
Ν	64	64	64	64	64	64	64	64
Final external	validation set (2	018 intact cano	la seed samples)				
Mean	48.54	22.80	24.80	9.70	64.5	8.00	6.67	111.0
St. Deviation	3.20	3.51	39.78	3.66	4.49	2.85	0.27	6.19
Median	48.37	22.46	9.74	8.86	64.06	8.49	6.69	111.01
Minimum	41.6	16.50	1.3	3.6	56.7	1.49	5.90	92.7
Maximum	55.1	31.50	210.9	21.1	77.9	12.40	7.30	119.38
Ν	69	69	69	69	69	69	69	69

TABLE 1. Sample description—reference method summary for canola calibration and verification sets

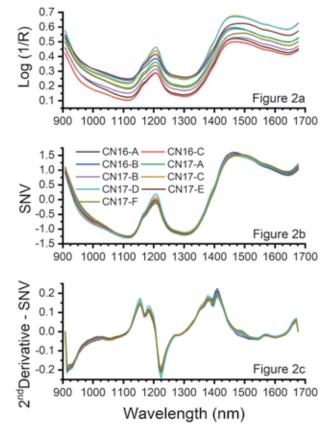


FIG. 2. Typical scans of intact canola seed samples from the canola calibration set with no spectral processing (a), after an SNV transform (b) followed by an S-G second derivative with four-point smoothing (c) for the external validation samples, potentially decreasing the validity of the NIR prediction models in the extrapolated regions. However, the robustness of the calibration models is important, as more often than not the models must be able to predict sample quality factors that might be slightly outside of the normal calibration range.

Models could be developed using raw NIR scans or processed scans. The mathematical pre-treatments applied on individual averaged spectra serve to normalize spectra and eliminate noise, baseline offset, and slope variations. For intact canola seeds, the spectral pre-processing was shown to be important to obtain good cross-validation statistics. Alternatively, intact soybean prediction models gave better cross-validation results when minimal spectra pre-processing was done. As mentioned, run scans repeated five times averaged into one sample scan was the optimal protocol to reduce sampling effect and predict intact canola seed quality. To develop our models, a baseline variation correction was performed on the raw average spectra by applying a standard normal variate (SNV) data transformation followed by a Savitzky–Golay smoothing second derivative with four-point smoothing on either side of the data point. These scan treatments are presented in Figure 2. Soybean scan treatment was only a SNV treatment.

Plotting the variance of the calibration as a function of number of factors (NF) allowed for the optimization of the NF values used to construct the predictive models. The optimal NF value was obtained when the addition of another factor in the calibration model did not increase the explained variance by more than 6% to the root mean square error (RMSE), while also giving satisfactory statistical results (standard error of cross-validation or SECV and R-square cross-validation or $R^2_{cross-val}$).

STATISTICAL RESULTS

Statistical results for canola quality prediction models are presented in Table 2 (page 26) and as a graphical linear relationship in Figure 3 (page 27). The prediction models for oil content, protein content, and iodine value showed excellent statistical results on both verification sets, with R²_{val} over 0.96, ratio of performance to deviation (RPD) over 5.1, and desirable standard error of prediction (SEP) and bias values (Table 2 and Figure 3). Iodine value prediction can help to differentiate canola type (i.e., conventional, LL, or HO-LL) as high-stability canola (LL or HO-LL), which usually has iodine values below 100 units (104 unit in some rare cases). The NIRs predictive models that were developed with the handheld spectrometer compare favorably to published models originally developed by benchtop instruments (Daun, *et al.*, 1994; Petisco, *et al*, 2010).

The calibration set used to develop the total glucosinolate prediction model (N=77) contained about the same number of samples as each verification set, leading to a larger N in the verification (N = 64+69 = 133) than in the calibration. The statistical results obtained with the two verification sets were poor and could not accurately predict the total glucosinolate content in canola. However, the model still has the potential to differentiate canola from rapeseed. Benchtop instruments showed similar SEP but better R²_{val} (Daun, *et al.* 1994, Petisco, *et al.*, 2010). The optimum spectral region for total glucosinolates prediction were 700–1017, 1333–1837, and 2173–2355 nm (Daun, *et al.*, 1994; Kumar, *et al.*, 2010). The limited wavelength range of the handheld NIRs (908.1–1676.2 nm range) could be an additional factor for the poor results of the total glucosinolate prediction model. Improvement to the current calibration model requires the addition of new samples and testing against the same two verification sets. This should be done prior to determining if the instrument is inadequate in predicting the glucosinolate content in intact canola seeds.

The model for predicting chlorophyll content was poor (Table 2) and not usable for any application. Wavelengths in the visible region (600–770 nm) are essential to predict chlorophyll content in intact canola seeds (Tkachuck and Kuzina, 1982). This limitation is not restricted to handheld NIRs but specific to any NIRs that do not have the required wavelengths for this particular model of prediction.

Oleic acid and alpha-linolenic acid prediction model assessments gave results considered fair to good (Table 2). These results compared favorably to previously reported results using benchtop NIRs (Daun, *et al.*, 1994; Oblath, *et al.*, 2016, Siemens and Daun, 2005). However, our first verification set showed an outlier, which was in fact a high-erucic-acid rapeseed. The inclusion of a high-erucic-acid rapeseed sample led to an incorrect prediction for oleic acid content, suggesting that NIR erucic

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	De	velopm	ent	Internal Cross-Validation			Testing and Verification													
Content Parameter	NF⁵	SEC	R ² cal ^d	NF⁵	SECV ^e	R ² cross-val ^f	Verifica- tion set	SEP ^g	RPD ^h	R ² val ⁱ	Bias	Comments (Williams, 2001)								
Oil	20	0.27	0.988	4	0.49	0.961	1	0.45	7.7	0.986	-0.06	V. good - Process control								
(%, dry basis)	20	0.27	0.988	4	0.49	0.961	2	0.59	7.1	0.985	0.20	Good - Quality control								
Protein	20	0.26	0.985	5	0.48	0.953	1	0.55	6.8	0.984	0.29	V. good - Process control								
(%, dry basis)	20	0.20	0.985	5	0.48	0.953	2	0.50	7.1	0.980	-0.24	V. good - Process control								
Total glucosinolates	20	0.82	0.948	3	2.9	0.359	1	3.5	1.2	0.310	-0.97	V. poor - Not recommended								
(µmol/g seed)	20	0.02	0.540	5	2.3	2.5	2.9	2.3	2.3	0.335	2	2.5	1.5	0.202	-2.0	V. poor - Not recommended				
Chlorophyll	20	5.75	0.764	6	11.0	11.0	0.144	1	21	0.9	0.271	1.6	V. poor - Not recommended							
(mg/kg, as is)	20	5.75	0.764	D			11.0	11.0	11.0	11.0	11.0	11.0	11.0	11.0	11.0 0.144	1.0 0.144	2	7.3	5.4	0.116
Oleic acid ^a	20	0.50	0.983	6	1.0	0.926	1	1.2	3.7	0.935	0.32	Fair - Screening								
(%, in oil)	20	0.50	0.983	σ	5 1.0	0.926	2	1.0	4.2	0.937	0.49	Fair - Screening								
α-Linolenic acid	20	0.46	0.970	6	0.98	0.867	1	0.77	3.8	0.929	-0.01	Fair - Screening								
(%, in oil)	20	0.40	0.970	D	0.98	0.867	2	0.96	3.0	0.883	-0.42	Poor - Rough screening								
Total saturates	20	0.13	0.809	8	0.23	0.399	1	0.26	1.2	0.290	0.04	V. poor - Not recommended								
(%, in oil)	20	0.15	0.809	0	0.25	0.23 0.399	2	0.25	1.1	0.202	0.10	V. poor - Not recommended								
lodine value	20	0.35	0.996	6	0.00	0.00	0.974	1	1.10	6.6	0.968	-0.31	V. good - Process control							
(units, in oil)	20	0.55	0.990	0	0.90	0.90 0.974	2	0.98	6.3	0.987	-0.83	V. good - Process control								

TABLE 2. Calibration statistics for the prediction models. Model developed with standard normal variate (SNV) transformation followed by a second derivative using the Savitzky-Golay (S-G) algorithm, 4 points data smoothing on either side of the central data point, and a second order polynomial.

¹External verification set 1: 2017 canola samples

² External verification set 2: 2018 canola samples

^a An outlier (high erucic acid rapeseed) was found in the validation dataset and removed from the validation dataset for oleic acid testing only ^bNF, the number of factors used as components in the PLSR model ^c SEC, Standard Error of Calibration

acid spectral features may overlap significantly with oleic acid spectral features. This highlights one of the limitations of NIR spectroscopy (NIRS), either handheld or benchtop: Chemical constituents with similar spectral features may lead to an inaccurate analysis if a broad range of constituent concentrations (of oleic acid, alpha-linolenic acid, and erucic acid in this case) are not included in developing the model. The presence of an unusually high concentration of erucic acid (not found in canola) such as in the rapeseed would cause the model to fail with respect to oleic acid and alpha-linolenic acid content.

The handheld NIRs prediction model performance for total saturates in intact canola seed was very poor and unreliable. The total saturate range was 5.4–8.0%; however, most of the samples fell within the 6.0–7.0% range. As for any NIRs, the range and the distribution of the analyte to be predicted limited the quality of the model.

Handheld NIRs, with limited wavelength range (908.1– 1676.2 nm) can be used on intact canola seeds to predict various quality parameters. Some predicted parameters (e.g., oil content, protein content, and iodine value) could be used for any application, whereas other predicted parameters (e.g., fatty acid composition) could only be used for screening (e.g., standard error of prediction of 0.96 for a minimum alpha-linolenic acid for 1.49%). The wavelength range (e.g., visible range ${}^{d}\,R^2_{\,cal}$, Coefficient of determination for the calibration set ${}^{e}\,SECV$, Standard Error of Cross validation

^gSEP, Standard Error of Performance

^h RPD, Residual Prediction Deviation

 $^{i}R^{2}_{\ val}$, Coefficient of determination for the validation set

for chlorophyll model) and the type of sample (small seed size versus big seed size) will limit the application of these instruments for on-the-spot analysis of some intact grains (e.g., soybean) to certain quality parameters (e.g., chlorophyll).

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Bert Siemens has an M. Sc. from the University of Manitoba, where he is a faculty member in Agriculture and Food Sciences. He has been with the Canadian Grain Commission (Oilseeds Chemist) for three decades and focuses on NIR spectroscopy quality analysis on oilseeds.

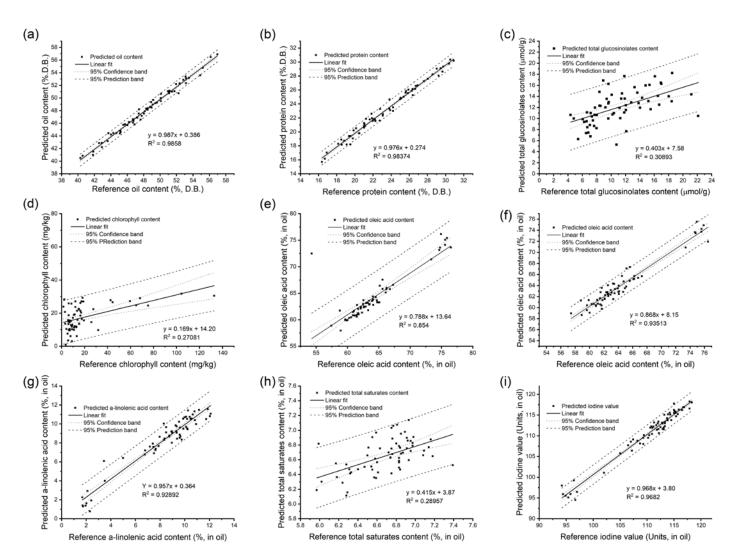


FIG. 3. Scatter plots of reference data and NIR predicted values form the external validation set 1 for the estimation of oil (a), protein (b), total glucosinolates (c), and chlorophyll content (d), plus oleic acid (e–f), alpha-linolenic acid content (g), total saturates (h), and iodine value (i) of intact canola (*B. napus*) seeds.

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Cleansing oils, the new personal care trend

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

Rebecca Guenard

Starting in adolescence, when overactive glands produce an abundance of oil, clogging pores and promoting acne, most of us adopt cleansing routines that intentionally strip oil away. Oil-free products have ruled the personal care market for decades. This year, the industry has done an about-face, touting the benefits of oil-based cleansers that heal the skin.

According to Grand View Research, Inc., the global cosmetic oil market is poised to experience a 5.2% increase in revenue growth and reach \$72 billion by 2025 (http://www. grandviewresearch.com). The popularity of these products comes following the acknowledgment in the industry that some cleansers contain harsh surfactants that damage the skin.

By design, surfactants interact with and solubilize dirt and oil, but that also means they remove lipids from the outermost layer of the skin, the stratum corneum (SC). Depending on the functionality of a surfactant's head group, it may also cause damage to the skin's structural proteins. Personal care product manufacturers have developed mild surfactants and reformulated cleansers to minimize this type of damage. Yet, the new consumer trend is to forgo foaming water-based cleansers altogether.

Most experts credit Korean beauty manufacturers for this trend, since the products produced by Korean companies tend to be designed to preserve the skin barrier using natural ingredients. Now, the practice has become popular worldwide.

Oil cleansers are not new. There is evidence that they have been used as far back as the ancient Egyptians, 5,000 years ago. Public baths in Ancient Greece and in Rome were a place for people to douse themselves in extra virgin olive oil (EVOO) as a way to clean their bodies (https://tinyurl.com/ytckyust). The cleansing product eventually evolved into bars of soap made by heating olive oil with water and baking soda that solidified when cooled.

The anti-oxidant, anti-inflammatory properties of the phenols in EVOO make it a popular food product, and these traits make it beneficial for the skin as well. The oil also contains bacteria-fighting microbes and skin-nourishing vitamins. In addition, when applied topically the oil acts as an emollient, restoring skin lipids.

The results of a study out this summer on eczema patients showed that using lipid-rich cleansers reduced the severity of the skin disease by restricting water loss and improving hydration (https://doi.org/10.1111/dth.14970). In the double-blind clinical trial, patients given a lipid-based cleanser containing fatty acids from ingredients like safflower seed oil were compared to patients given a placebo.

Typical treatments for eczema include clinical strength moisturizers and creams, but scientists are finding that these products most often just act as a superficial barrier to temporarily prevent water loss from the SC. Together with cholesterol and free fatty acids, ceramides form the multilayered structures affecting skin permeability and thus water modulation. Researchers believe instead of a temporary fix, lipid-based cleansers (and moisturizers) permeate the SC, resolving the structural abnormalities that cause eczema.

The study authors wrote, "physiologic lipids, including ceramides, permeate the SC and are synthesized in the keratinocytes, processed in lamellar bodies, and secreted back into the SC to become a part of the dermal matrix." As a result, after 14 days patients given oil-rich cleanser reported less itching and dryness along with more softness and smoothness. Although this clinical research was conducted on patients with a compromised skin barrier, consumers with healthy skin experience similar benefits.

In fact, the products have become so popular that most well-known cosmetic companies, from drugstore to designer brands, offer an oil cleanser in their product lineup. The base oil for these products is often a common edible oil, such as almond, sunflower, or soybean. Including these sources of linoleic acid in their formulations provides an essential omega-6 fatty acid known to help build ceramides. Pricier blends contain more exotic sounding ingredients, like coix lacryma-jobi seed oil, a common anti-inflammatory used in Chinese medicine (https://www.allure.com/gallery/best-cleansing-oil).

Of course, plain old EVOO is still an option (https://tinyurl. com/488c6zhn). In fact, a luxury olive oil producer in Joshua Tree, California called Wonder Valley sells EVOO for both consumption and topical use. Although the bottles look similar, their facial cleanser formulation incorporates a few extra skinenhancing ingredients (https://welcometowondervalley.com/ index). However, EVOO acts as an effective cleanser without any added ingredients.

According to dermatologists, oil cleansers work because they dissolve the dirt and oil on the skin without disturbing the skin's microbiome. They remove waxy makeup, such as eyeliner or waterproof mascara, better than water-based cleansers while dissolving excess sebum—the oil produced by skin glands that can lead to acne. This is in addition to the antioxidant, anti-aging benefits mentioned earlier (https://tinyurl.com/488c6zhn).

Experts do note that facial skin is more delicate than other areas of the body, and a wide range of skin types exist. Therefore, they recommend that consumers with acneprone skin use cleaning oils sparingly and make sure not to leave behind any excess. They also suggest using high-quality, unrefined oils if consumers are not purchasing a formulated cleanser from a beauty brand. Oils that contain impurities could lead to breakouts.

Information

A daily regimen of a ceramide-dominant moisturizing cream and cleanser restores the skin permeability barrier in adults with moderate eczema: a randomized trial, Spada, F., *et al., Dermatologic Therapy 34*: e14970, 2021.

Why you should ignore the "oil-free" hype, Ellenberger, J., *Finweek 5*, 2019.

Cleansing without compromise: the impact of cleansers on the skin barrier and the technology of mild cleansing, Ananthapadmanabhan, K.P., *et al.*, *Dermatologic Therapy 17*: 16–25, 2004.

Beyond that, the cleaning process is simple. Just massage one or two teaspoons of oil all over the face for a minute and gently wipe away with a warm washcloth. For acne prone skin, use a mild cleanser afterward to ensure the cleaning oil has been removed (https://tinyurl.com/52ujamp9).

The dry, tight feeling of skin after washing with soap is no longer associated with cleanliness; today's consumer wants to experience the soft, smooth feeling left behind by oil cleansers.

Rebecca Guenard is the associate editor of Inform *at AOCS. She can be contacted at rebecca.guenard@aocs.org.*

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Expanded TSCA reporting obligations could open companies to fines

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

New Toxic Substances Control Act (TSCA) reporting rules could have "profound implications" for companies in possession of information that reflects a substance poses a "substantial risk" to human health or the environment, according to an industry attorney.

And the US Environmental Protection Agency (EPA) has confirmed that any such information will "be carefully considered".

The potential to run afoul of TSCA's section 8(e) "substantial risk" reporting requirements adds an additional layer of complication and potential liability as companies all along the supply chain work to understand their compliance obligations amid an influx of new and proposed reporting obligations in the United States.

The EPA has proposed a sweeping reporting rule for per- and polyfluoroalkyl substances (PFASs) that would require industry to submit data on substances' health and environmental effects. It also finalized an 8(d) rule requiring submission of health and safety studies on 50 substances, and floated plans to implement tiered data reporting (TDR) to call in information on additional substances.

Concerns have already surfaced that requirements to submit health and safety studies could create issues where REACH data is involved.

But such mandates could also lead to fines if the EPA determines that information submitted under the new reporting rules should have been immediately reported under TSCA section 8(e) at the time it was originally generated, Arnold & Porter partner Lawrence Culleen wrote in a *Chemical Watch* expert briefing last week.

A company that submits previously unnotified information that shows a substance could present a substantial risk to human health or the environment could face "stiff penalties for violations of a provision of the statute which the EPA vigorously enforces," said Culleen.



SECTION 8(e)

Section 8(e) of TSCA is "intended to serve as an early warning system for substantial risks posed from chemical substances", the EPA said.

The universe of information that may trigger the immediate reporting requirement is "vast", said the agency, and includes:

- incomplete studies;
- periodic testing of employees;
- water samples;
- data collected from accidental injuries or spills;
- research and development information;
- underlying data from investigating chemical risks;
- information known by company headquarters in another country; and
- information collected since 1977 continues to be reportable today.

The 8(e) notifications must be made within 30 days of a company obtaining the information, and the EPA can pursue civil or criminal penalties for noncompliance.

Penalties for failing to comply with the requirement can be stiff, with the potential for fines to exceed \$40,000 per day, starting from the time the information was first obtained.

In 2010, for example, the EPA fined DuPont \$3.3m after the company voluntarily notified the agency that it had failed to report more than a hundred inhalation toxicity studies it conducted, 57 of which the agency decided met criteria for reporting under section 8(e).

If a company discovers information that should have been notified under 8(e), they "should immediately report the information to the agency", the EPA said.

AOCS MEETING WATCH

October 12–14, 2021. Plant Protein Science and Technology Forum, online.

May 1–4, 2022. AOCS Annual Meeting & Expo, Atlanta, Georgia, USA, and online.

April 30–May 3, 2023. AOCS Annual Meeting & Expo, Denver, Colorado, USA, and online.

For in-depth details on these and other upcoming meetings, visit http://aocs.org/meetings or contact the AOCS Meetings Department (email: meetings@aocs.org; phone: +1 217-693-4831). "Regulated entities of any size who voluntarily discover, promptly disclose, expeditiously correct and take steps to prevent recurrence of potential violations may be eligible for a reduction or elimination of any civil penalties that otherwise might apply," it said.

ACTION FROM THE EPA OR NGOs

According to the agency, "any information regarding "substantial risks" associated with a chemical will be carefully considered in light of our responsibilities under TSCA to protect against potential risks to health and the environment irrespective of whether the agency receives that information under TSCA section 8(e) or through other means.

"Potential reporting violations would be dealt with on a case-by-case basis by coordination between the receiving office and the enforcement office," it said.

Enforcement pressure could also come from NGOs, as was recently seen over alleged chemical data reporting (CDR) rule violations and in a request earlier this year for the EPA to pursue a \$434m fine for alleged 8(e) noncompliance.

Culleen said it would be prudent for businesses subject to the reporting rules "to review their files very, very carefully". Even at the proposed rule stage, he said it's not too early for companies to ask themselves if they have information that might be within the scope of section 8(e).

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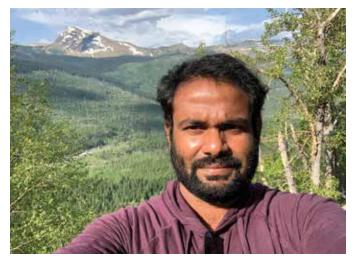
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Meet Hari Kiran Kotapati

Member Spotlight is a slice of life that helps AOCS members get to know each other on a more personal level.



Hari Kiran Kotapati in August 2020 at Glacier National Park Mountain in Montana (USA)

PROFESSIONAL

What's a typical day like for you?

My day starts very early and I am usually at work by 8:15 A.M. at the latest. I spend the first half-hour catching up on emails, science news, and just-published research papers in my field. Then I get back to the workbench in the lab unless I am working on writing. If weather permits, I usually take a 15-minute walk after lunch, and then it is back to the lab to finish things up before the end of the day.

My favorite part of my job is...

The problem-solving aspect and, most importantly, learning something new almost every day.

Flash back to when you were 10 years old. What did you want to be when you grew up?

Growing up in India, I always wanted to be an officer in the civil administrative services, which is very difficult to get into. I gave up on that dream when I discovered my true passion for science during my high school years.

Why did you decide to do the work you are doing now? I just started my new job at the United States Department of Agriculture–Methods and Application of the Food Composition Laboratory to work on the analysis of lipids from cows' milk. There are two main reasons I would like to emphasize for choosing my current job. First, I grew up on a farm in rural India and spent my holidays working on the farm and helping

Fast facts

Name	Hari Kiran Kotapati
Joined AOCS	2018
Education	Ph.D. from the University of Southern Mississippi (Hattiesburg, Mississippi, USA)
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Employer	University of Maryland College Park (College Park, Maryland, USA)
Current AOCS involvement	Secretary, Young Professional Common Interest Group

my parents in any way I could. So, research related to agriculture always has a special place in my heart. Second, I am super excited to be working under the guidance of Craig Byrdwell, a world-renowned expert in the field of lipid analysis.

What event, person, or life experience has had the most influence on the direction of your life?

My maternal grandmother Srimati Rukma Bai, who passed away about four years ago right before my Ph.D. defense. At one point, she sat me down and convinced me to go to college after I had decided not to pursue college education after high school.

PERSONAL

How do you relax after a hard day of work?

Regardless of how busy my day is at work, I try to finish off by either working out or going for a quick run. I find running to be a great way to relieve stress, especially after a hectic workday.

What is the most impressive thing you know how to do? Backing up a tractor with an attached trailer. I learned this when I was about 19 years old on my dad's farm. It took almost three years for me to learn how to do it perfectly. Although I have not driven a tractor in over a decade, I think I could still do it.

What skill would you like to master?

Cooking authentic biriyani (a South Indian dish). I have been cooking (mostly Indian food) for the last 15 years or so, and I can cook a decent biriyani.

PATENTS

Lipid formulations for delivery of messenger RNA

Heartlein, M., et al., Translate Bio, Inc. and Massachusetts Institute of Technology, US10959953, March 30, 2021

The present invention provides, among other things, methods of delivering mRNA *in vivo*, including administering to a subject in need of delivery a composition comprising an mRNA encoding a protein, encapsulated within a liposome such that the administering of the composition results in the expression of the protein encoded by the mRNA *in vivo*, wherein the liposome comprises a cationic lipid of formula I-c: ##STR00001## or a pharmaceutically acceptable salt thereof.

Capsules containing high doses of krill phospholipids

Hupfeld, S., *et al.*, Aker BioMarine Antarctic AS, US10960016, March 30, 2021

Oral capsules have been dismissed as a dosage form for delivering high dosages of krill phospholipids, but by purifying these to high levels it is indeed possible to use this dosage form to deliver, for example, 700 mg or more phospholipids per capsule.

Tire with tread of carbon black reinforced rubber composition containing specialized styrene/ butadiene elastomer

Brace, L.E., The Goodyear Tire & Rubber Co., US10961373, March 30, 2021

The invention relates to a tire having a tread of carbon black reinforced rubber composition containing a specialized styrene/ butadiene elastomer with molecular weight profile containing a limited low molecular weight content and triglyceride vegetable oil.

Modification of fats and oils for fuel and lubricating applications

Rissio, J., 21st Century R & D, LLC, US10961472, March 30, 2021

A bio-organic composition includes residues of a fatty acid glyceride-containing composition, residues of a first epoxide or glycol, and the residues of a second epoxide. The fatty acid glyceride-containing composition is characterized by the viscosity at room temperature. The first epoxide or glycol and second epoxides are present in a sufficient amount that the room temperature viscosity of the bio-organic composition is lower than the room temperature viscosity of the vegetable oil prior to formulation and/or the first epoxide or glycol and second epoxides are present in a sufficient amount that the pour point of the bio-organic composition is lower than the pour point of the fatty acid glyceride-containing composition prior to formulation.

Method for preparing 2-monoacylglycerides

Rongione, J., Stepan Co., US10961483, March 30, 2021

Methods for preparing and purifying 2-monoacylglyceride compounds are disclosed. In one method, an unsaturated triglyceride is reacted with water, a C.sub.1-C.sub.8 alcohol, or a mixture thereof in the presence of a lipase to produce a mixture comprising a 1,3-dihydroxy-2-monoacylglyceride and fatty esters or acids. Reaction of the 1,3-dihydroxy-2-monoacylglyceride with an aldehyde or ketone gives a mixture comprising a 2-monoacylglyceride acetal or ketal. Fatty esters or acids are removed from the mixture as an overhead product by distillation or wiped-film evaporation to isolate a purified 2-monoacylglyceride acetal or ketal. The inventive methods provide a 2-monoacylglyceride protected at the 1and 3-positions such that the acyl unit remains at the 2-position. The products are enriched in unsaturated fatty acid content when compared with the unsaturated fatty acid content of the original unsaturated triglyceride. Each method utilizes a practical purification scheme that avoids the scale-up or toxicity issues of commonly employed purification strategies.

Method for freezing olive oil

Volonakis, A., inventor, US10966444, April 6, 2021

Method for freezing olive oil which achieves the sustaining of the polyphenols until the time of its consumption. The method is performed immediately after the collection of the fruit olives from the tree, with the addition of nitrogen, the cold extraction of the oil and the remaining of the oil pulp in water of 27° C temperature at the softening for 30 minutes. The product is being transferred in stages at storage tanks with nitrogen supply and gradual at stages reduction of the temperature by 5° C and remaining in each storage tank for 12 hours until its temperature reaches $6-7^{\circ}$ C, after which it is being packaged and frozen with slow in stages freeze to -18° C to -23° C. Due to this method, the product maintains during the whole internal of freezing the color and aroma and the taste it had during its transformation into oil, organic characteristics that reappear exactly the same after it has been defrosted.

Skin-mimicking emulsion

Ormancey, X., Counter Brands, LLC, US10966914, April 6, 2021

The present invention is directed to a skin-mimicking emulsion comprised of polyosides, minerals, inositol polyphosphate or phytic acid, polysaccharides, triglycerides, polyols, alpha or beta hydroxy acids, phospholipids, phytosterols, vitamins, fatty alcohols, essential lipids, amino acids, and water. All of the inventive ingredients in combination are design to be similar to those found in skin and are combined to form a lamellar structure for maximum efficacy. The composition does not include any of the components usually found in conventional emulsions, such as synthetic preservatives, surfactants, fragrances, colorants, acrylic polymers, gelling agents, sequestrants, and EDTA, as such ingredients would be irritating to the skin.

Deodorant compositions

Sturgis, D.A., *et al.*, The Procter & Gamble Co., US10966915, April 6, 2021

A deodorant stick comprising: at least about 25% of a liquid triglyceride; at least one antimicrobial; a primary structurant with a melting point of at least about 50°C; and less than 8% of secondary structurants having a melting point of at least about 60°C; said stick being free of an aluminum salt; and said stick having a hardness from about 80 mm*10 to about 140 mm*10, as measured by penetration with ASTM D-1321 needle.

Compositions and methods for delivery of polyunsaturated fatty acid derivatives and analogs

Brostrom, L., et al., Cytometix, Inc., US10966937, April 6, 2021

The present invention provides a system enabling the oral delivery of therapeutics derived from polyunsaturated fatty acids (PUFAs), their metabolites and derivatives, including, eicosa-noids, prostaglandins, prostacyclins, leukotrienes, resolvins, endocannabinoids, thromboxanes, epoxyeicosa-trienoic acids (EETs), hydroxyeicostetraenoic acids (HETEs), and CMX-020. The delivery system includes a vehicle comprising a purified doco-sahexaenoic acid (DHA) in triglyceride or ester form; a purified eicosapentaenoic acid (EPA) in triglyceride or ester form; a combination of DHA, EPA in either triglyceride or ester forms; or a modified DHA, EPA, or omega-3 fatty acid analog; and optionally, an antioxidant, a surfactant, a solubilizer, a stabilizer, a lubricant, or a pH/tonicity adjustment agent.

Process of preparing vitamin E concentrate

Gee, P.T., Palm Nutraceuticals Sdn. Bhd., US10967037, April 6, 2021

The present invention discloses a process for modifying the natural composition of tocotrienol-rich fraction to achieve a product with reduced alpha-tocopherol content, enhanced beta- and delta-tocotrienol content, and also with an enriched total tocotrienol concentration.

Shea butter-containing rubber compositions and related methods

Galizio, B., Bridgestone Americas Tire Operations, LLC, US10968331, April 6, 2021

Disclosed herein are rubber compositions comprising shea butter and a rubber, and tires (and tire components) made from the rubber compositions. Also disclosed are methods of improving the wear resistance of a rubber composition by utilizing shea butter.

Oil-enhanced polymer modified asphalt adhesive compositions and methods of making

Croteau, C.R., *et al.*, Owens Corning Intellectual Capital, LLC, US10968376, April 6, 2021

Oil-enhanced polymer modified asphalt adhesive compositions, membrane materials including the oil-enhanced polymer modified asphalt adhesive composition, and methods for making are provided. The oil-enhanced polymer modified asphalt adhesive compositions have improved elongation, recovery, heat resistance, and cold adhesion and may include a polymer modified asphalt and an oil or oil derivative additive. The polymer modified asphalt may include an elastomeric copolymer. The oil or oil derivative additive may include one or more of vegetable oils, nut oils, and seed oils. A membrane including the oil-enhance polymer modified asphalt is also provided. A multi-layered roofing membrane is also provided.

Compositions comprising omega-3 polyunsaturated and medium-chain fatty acids

Jackson, M., *et al.*, Hill's Pet Nutrition, Inc., US10973244, April 13, 2021

Disclosed herein are pet food compositions for treating or preventing inflammation or an inflammatory disorder in an animal comprising an effective amount of at least one medium-chain triglyceride and an effective amount of at least one omega-3 fatty acid, wherein the at least one medium-chain triglycride and the at least one omega-3 fatty acid are present in an amount effect to a provide a synergistic decrease in the amount of circulating cytokines in the companion animal. Also disclosed herein are methods for treating or preventing inflammation or an inflammatory disorder in a companion animal, comprising administering the pet food compositions disclosed herein to the companion animal in need thereof.

Heterologous production of 10-methylstearic acid

Shaw, Arthur J., *et al.*, Ginkgo Bioworks, Inc., US10975398, April 13, 2021

Nucleic acids and cells comprising a methyltransferase gene and/or a reductase gene are disclosed. These nucleic acids and cells may be used to produce branched (methyl)lipids, such as 10-methylstearate.

Patent information was compiled by Scott Bloomer, a registered US patent agent and Director, Technical Services at AOCS. Contact him at scott.bloomer@aocs.org.





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3 of 3

AOCS JOURNALS

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Calculating bibliometrics can sometimes be complicated and confusing. This handy table from Scholarly Commons of the University of Illinois Library (https://guides.library.illinois. edu/c.php?g=621441&p=4328607) illustrates what these metrics measure, how they are calculated, and what databases and resources are available for each metric. Authors can find many other resources at the Scholarly Commons website (https://www.library.illinois.edu/sc/).

Metric	Website	Calculation		Meaning
Impact Factor	Journal Citation Reports (https:// bit.ly/3xalte9)	Use a two-year per cited by the number Example: 200 = the number of were cited by index 73 = the total numb 2019. 200/73 = 2.73 2020 impact factor	Impact factor reflects only on how many citations on a specific journal there are (on average). A journal with a high impact factor has articles that are cited often.	
h-index	Web of Science Google Scholar Scopus	scending order, base 2) Look down throu number of times a than the line (or particular Articles 1 2 3 4 5 6 7 8 *please remember ber; this is only if yo often find calculato *graphic republishe (2012). The h-index:	of your publications. organize articles in de- ed on the number of times they have been cited. ugh the list to figure out at what point the publication has been cited is equal to or larger per) number of the publication. Citation numbers 33 30 20 15 7 6 = h-index 5 4 that many databases will give you this num- ou'd like to calculate it manually. You can also ors online. d from Ireland, T., MacDonald, K., & Stirling, P. What is it, how do we determine it, and how n it? In A. Tokar, M. Beurskens, S. Keuneke, M.	The h-index focuses more specifically on the impact of only one scholar instead of an entire journal. The higher the h-index, the more scholarly output a researcher has.
		Can we keep up with Mahrt, I. Peters, C. F Science and the inte		

Metric	Website	Calculation	Meaning
g-index	Harzing's Publish or Perish (https:// harzing.com/ resources/pub- lish-or-perish)	Given a list of articles ranked in decreasing order of the number citations that they received, the g-index is the largest unique number to the extent that the top g articles received together is at least g2 citations.	The g-index can be thought of as a continu- ation of the h-index. The difference is that this index puts more weight on highly cited citations. The g-index was created because scholars noticed that h-index ignores the number of citations to each individual article beyond what is needed to achieve a certain h-in- dex. This number often complements the h-index and is not necessarily a replacement.
Eigenfactor score	Eigenfactor.org	The Eigenfactor score is calculated by eigenfactor.org. Howev- er, their process is very similar to calculating impact factor and they pull their data from the JCR as well. The major difference is that the Eigenfactor score deletes references from one article in a journal to another in the same journal. This eliminates the problem of self-citing. The Eigenfactor score is also a five-year calculation. More information can be found through Journal Citation Reports.	A high Eigenfactor score signals that the journal does not self-cite and con- trols the network of that discipline. It is useful to look at scholar's h-index as well as the Eigenfactor score of the journals they publish in in order to get a broad sense of their impact as a researcher.
Altmetric	Altmetric.com	Altmetric scores are usually calculated by companies. This means that they cannot be calculated manually. To see an explanation of how this metric is caculated, visit https://help.altmetric.com/ support/solutions/articles/6000233311-how-is-the-altmetric-at- tention-score-calculated.	Different sources go into altmetrics calculations, depending on the com- pany and the information that they are using. But in general, a high altmetric score indicates that an item has received a lot of attention and it has also received what that compa- ny has decided is "quality" attention (i.e. a news post might be more valuable than a twitter mention). Remember that attention does not necessarily indi- cate that the article is im- portant or even of quality. That is why it is useful to use altmetrics and impact factor together.

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S&D Surfactants and Detergents

Review Articles

ANA Advanced methodologies for trace elements in edible oil samples: a review

Shah, N.S. and M. Soylak, *Crit. Rev. Anal. Chem.* 1–20, March 15, 2021, https://doi.org/10.1080/10408347.2021.1895710.

Thousands of tons of fruit seeds are discarded every year worldwide as agro-industrial byproducts. Fruit seeds have a high oil content, are rich in monounsaturated fatty acids (FA) and in n-6 and n-3 polyunsaturated essential FA. Sterols, phospholipids, glycolipids, carotenoids, tocopherols, and polyphenols are other seed phytochemicals that make them interesting from a commercial viewpoint. Fruit seeds have high potential as raw material for several industries, but their lipid profile remains poorly studied. Current analytical approaches for the analysis of lipids that are based on high-performance liquid chromatography and high-resolution mass spectrometry allow the separation and analysis of compounds with the accurate identification and structural characterization of molecular species in very small quantities. Even though lipidomic analysis of fruit seed lipids is still in its infancy, it will bring a new look over these value-added byproducts. This review covers the following topics: (a) the lipid content of various fruit seed oils; (b) their lipid composition (FA, triacylglycerol, sterol, phospholipid, and glycolipid profiles); (c) current and future analytical methodologies for the analysis of lipids in fruit seeds; (d) biological activities of fruit seed extracts; and (e) potential biotechnological applications of fruit seed oils for their commercial valorization based on lipids.

BIO H&N PRO Using green alga *Haematococcus pluvialis* for astaxanthin and lipid co-production: advances and outlook

Ren, Y., et al., Bioresour. Technol. 340, November 2021, https://doi.org/10.1016/j.biortech.2021.125736.

Astaxanthin is one of the secondary carotenoids involved in mediating abiotic stress of microalgae. As an important antioxidant and nutraceutical compound, astaxanthin is widely applied in dietary supplements and cosmetic ingredients. However, most astaxanthin in the market is chemically synthesized, which is structurally heterogeneous and inefficient for biological uptake. Astaxanthin refinery from Haematococcus pluvialis is now a growing industrial sector. H. pluvialis can accumulate astaxanthin to ~5% of dry weight. As productivity is a key metric to evaluate the production feasibility, understanding the biological mechanisms of astaxanthin accumulation is beneficial for further production optimization. In this review, the biosynthesis mechanism of astaxanthin and production strategies are summarized. The current research on enhancing astaxanthin accumulation and the potential joint production of astaxanthin with lipids are also discussed. It is conceivable that with further improvement on the productivity of astaxanthin and by-products, the algal-derived astaxanthin would be more accessible to low-profit applications.

EAT IOP Gurum seeds: a potential source of edible oil

Karrar, E., *et al., Eur. J. Lipid Sci. Technol.* 123: 2000104, 2021, https://doi.org/10.1002/ejlt.202000104.

Cucurbitaceae family seeds are mostly discarded as agro-industrial wastes. Gurum (Citrullus lanatus var. colocynthoide) is an underutilized wild cucurbit plant, closely related to desert watermelon, which is grown abundantly in some African countries. Gurum seeds can play a significant role in health and nutrition due to their high oil content. This review describes the nutritional composition of gurum seeds and their oil profile. Gurum seeds are a good source of oil (27-35.5%), fiber (26-31%), crude protein (15-18%), and carbohydrates (14–17%). Gurum seed oil is extracted by supercritical CO₂ (SFE), screw press, and solvent extraction techniques. The oil is composed of unsaturated fatty acids with a high proportion of linoleic acid (C18:2) and oleic acid (C18:1). It also contains various bioactive compounds, such as tocopherols, phytosterols, and polyphenols. Solvent extraction has been reported to gives a higher yield than the screw press and SFE, but SFE is preferred due to safety issues. More studies are required for producing better quality gurum seed oil by using novel extraction techniques that can increase oil yield.

EAT HEN Fabrication, interaction mechanism, functional properties, and applications of fish gelatinpolysaccharide composites: a review

Shi, X.-D., *et al.*, Food Hydrocoll. *122*: 107106, 2022, https://doi.org/10.1016/j.foodhyd.2021.107106.

Fish gelatins (FGs), which have been considered as excellent candidates to substitute mammalian gelatins, are important food hydrocolloids isolated from aquatic resources. However, some undesirable sensory and physicochemical properties limit their applications. Over the past decade, many studies have reported the modification of FGs using polysaccharides, and the FG-polysaccharide composites show great potentiality in the food industry. This review summarizes and discusses fabrication methods, interaction mechanisms, functional properties, and applications of FG-polysaccharide composites. Various approaches, including physical mixing, chemical cross-linking, and enzymatic cross-linking have been put forward to prepare FG-polysaccharide composites. Therefore, non-covalent and covalent bonds occurred between the two types of macromolecules. Mechanical properties (gel strength and barrier property) and biological functions (mainly antioxidant and antimicrobial activity) of FGs are improved after the addition of polysaccharides. The FG-polysaccharide composites show wide varieties of applications in food packaging, wound dressing, and drug delivery. Further sci-



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cs@newera-spectro.com www.newera-spectro.com Quality and value you can rely on!® entific studies are needed to determine the structural and physicochemical properties of FGs and polysaccharides, optimize reaction conditions, identify interaction mechanisms, and expand applications in food, cosmetics, and medical industries.

Microbial biodiesel production from lignocellulosic biomass: new insights and future challenges

Uthandi, S., et al., Crit. Rev. Environ. Sci. Technol. 1–30, February 11, 2021, https://doi.org/10.1080/10643389.2021.1877045.

In many countries, biodiesel production is obstructed because of a high production cost accounting for raw materials, the large acreage needed for the cultivation of oil-yielding vegetable crops, and competition between food and feed. Therefore, biodiesel production requires new approaches for which microbial oils offer a potential solution. Among several microorganisms available, oleaginous microorganisms (yeast and fungi) accumulate more than 20-70% oils inside their cells when grown in specific environmental conditions. Moreover, microbial oils or single cell oils (SCOs) offer numerous advantages over vegetable oils or animal fats such as similar fatty acid profile, short life cycles of the microbes, relatively lower environmental impact, reduced labor demand, and convenient scalability. Microbial lipids production using lignocellulosic biomass (LCB), which are naturally available in abundance, as a renewable raw material for producing second-generation biodiesel, has become a fundamental approach for tackling the challenges of higher energy costs, protection of the environment, and rapid depletion of crude oil reserves. This review compares and examines the extent to which different microbes can accumulate a productive level of lipids using LCB as substrates, pretreatment strategies used for converting LCB into SCOs, and future challenges in using LCB for biodiesel production.

Original Articles

ana eat h&n Krill oil

microencapsulation: antioxidant activity, astaxanthin retention, encapsulation efficiency, fatty acids profile, *in vitro* bioaccessibility, and storage stability

Ortiz Sánchez, C.A., *LWT* 147, July 2021, https://doi.org/10.1016/j.lwt.2021.111476.

Krill oil (KO) emulsions with a ratio of KO/arabic gum of 1:4 (w/w) were prepared and then spray dried. The effect of drying temperatures on antioxidant activity, astaxanthin retention, and microencapsulation efficiency was evaluated and microcapsules with better results were employed for further analysis. The encapsulation of KO in arabic gum was confirmed by using Fourier Connecting talented scientists and technologists with leading companies and universities.



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transform infrared spectroscopy. KO microcapsules showed eicosapentaenoic and docosahexaenoic content of 21% of the total fatty acids, low ω -6/ ω -3 and SF/unsaturated FA ratios, and a bioaccessibility of 53.8% after 2 h of *in vitro* digestion. The effect of a_w on color change and degradation of astaxanthin contained in KO microcapsules was evaluated during storage at 35°C. Monolayer value was 3.7241 g H₂O/100 g d.s. which corresponds to 0.4 of a_w . The astaxanthin degradation rate increased considerably when the KO microcapsules were stored at an a_w of 0.743, under these conditions the microcapsules showed drastic changes in the color parameters.

ANA Han A new HPLC-MS/MS method for the simultaneous determination of 36 polyphenols in blueberry, strawberry, and their commercial products and determination of antioxidant activity

Mustafa, A.M., et al., Food Chem. 367: 130743, 2022, https://doi.org/10.1016/j.foodchem.2021.130743.

Berry fruit consumption has increased in recent years because berries are rich sources of polyphenols with reported health benefits. The aim of the present work was to develop a new comprehensive and fast HPLC-MS/MS method for simultaneous determination of 36 phenolic compounds (7 anthocyanins, 9 flavonols, 4 flavan-3-ols, 2 dihydrochalcones, 2 flavanones, and 12 phenolic acids) present in blueberry, strawberry, and their fruit jam. Blueberry fruits showed higher contents of anthocyanins, flavonols, and phenolic acids, while strawberry fruits exhibited higher contents of flavan-3-ols, dihydrochalcones, and flavanones. Anthocyanins were the main phenolic constituents in both berries. Furthermore, the higher total phenolic content in the blueberry fruit and jam justified their greater antioxidant capacity measured by DPPH free radical assay, compared to strawberry. In conclusion, this new HPLC-MS/MS method is useful and reliable for quality control and authentication analyses of blueberry and strawberry fruits and their commercial food products, such as jams.

BIO IOP PRO A probabilistic economic and environmental impact assessment of a cyanobacteria-based biorefinery

Beattie, A., *et al.*, *Algal Res*. 59, November 2021, https://doi.org/10.1016/j.algal.2021.102454.

Microbial-based biofuels represent a potential promising solution as an environmentally favorable transportation fuel. Cyanobacteria have many of the same advantages as microalgae: ability for rapid growth in otherwise non-arable regions, suitability for genetic engineering, and simple nutritional needs. Additionally, cyanobacteria can be engineered to secrete valu-

able co-products that can be harvested independent from the produced biomass. However, little work has been done to identify the processes and the economic and environmental impacts associated with a large-scale cyanobacteria-to-fuels facility. The present study is a concurrent techno-economic and life cycle assessment of a facility that generates fuels and methyl laurate, an oleochemical, from the cyanobacterial species Synechocystis sp. PCC 6803. The biorefinery model includes all aspects of cultivation, separation of the secreted methyl laurate, biomass harvesting, and fuel processing via hydrothermal liquefaction (HTL) of the dewatered biomass. The assessments leverage Monte Carlo analysis (MCA) to address uncertainty and variability inherent in the most significant input parameters, replacing them with probabilistic functions. For the facility configuration producing both fuels and the oleochemical co-product, the MCA average minimum fuel selling price (MFSP) is \$2.47 per decimeter (dm3) or \$9.34 per gallon of gasoline equivalent (gge) with the corresponding average global warming potential determined to be 118 g CO₂-eq-MJ-1. The case producing only fuels results in an MCA average MFSP of \$2.01-(dm3)-1 (\$7.60-gge-1) and an average environmental impact of 100 g CO2-eq-MJ-1. These results are compared to static optimistic and conservative scenario analysis estimates, illustrating the over- and under-estimation of outcomes associated with non-stochastic methods. Suggested facility improvements include increases in pond productivity of both the biomass and methyl laurate oil production, as well as improvements to carbon utilization and bio-crude yield from HTL processing.

EAT Revisiting a model to predict pure triglyceride thermodynamic properties: parameter optimization and performance

Seilert, J., et al., J. Am. Oil Chem.' Soc. 98: 1–14, 2021, https://doi.org/10.1002/aocs.12515.

In 1990, a well-known model to predict pure component properties of triglycerides was presented by Wesdorp in "Liquidmultiple solid phase equilibria in fats: theory and experiments" and has been shown to perform well despite making thermodynamically inconsistent predictions for certain test cases. In this study, the underlying parameter set is improved to deliver more physically consistent predictions, i.e., increasing melting point and enthalpy of fusion with increasing stability of the polymorphs, without deterioration of the primary model quality to describe the available experimental data. Interestingly, when a curated dataset containing only thermodynamically consistent data is compared to a broader dataset, it appears that the model's efficacy is highly dependent on the quantity of data, specifically the number of unsaturated triglycerides data. Quality and thermodynamic consistency of model predictions and the condition of a reliable description of monoacid triglycerides as a subset is discussed, addressing a potential interdependence.



EAT LOQ Encapsulation of olive leaf extract (*Olea europaea* L.) in gelatin/tragacanth gum by complex coacervation for application in sheep meat hamburger

Oliveira, F.M., *et al.*, *Food Control* 131: 108426, 2022, https://doi.org/10.1016/j.foodcont.2021.108426.

The aim of the study was to encapsulate the phenolic extract of the olive leaf by using gelatin and tragacanth gum as wall materials, followed by complex coacervation or lyophilization. The extract was applied in sheep meat hamburger to reduce oxidative degradation. The gelatin and gelatin/tragacanth matrices showed higher values of encapsulation efficiency and antioxidant activity than the matrix with only tragacanth, independent of the encapsulation technique used. The encapsulation was confirmed through DSC and FTIR analyses, and the particles obtained presented an irregular shape. The application of the particles in hamburger demonstrated that the particles of gelatin and gelatin/tragacanth showed greater capacity to reduce oxidative reactions than those of tragacanth, they provided the highest percentages of phenolic compounds release in the first two months of storage. These particles proved to have higher antioxidant efficiency than sodium erythorbate; however, vitamin C showed the highest antioxidant capacity.

EAT LOQ Oleogelation of extra virgin olive oil by different oleogelators affects the physical properties and the stability of bioactive compounds

Marilisa Alongi, M., *et al., Food Chem.* 368: 130779, 2022, https://doi.org/10.1016/j.foodchem.2021.130779.

Extra virgin olive oil (EVOO) was gelled with 10% monoglycerides, (MG), rice wax (RW), gamma-oryzanol, and beta-sitosterol (PS), or ethylcellulose (EC). The oleogel structure and the stability of bioactive compounds were investigated during storage up to 120 days at 20, 30, and 40°C.

All samples were self-standing but presented different structures. PS produced the firmest gel, whereas EC caused the lowest firmness and rheological values. Structural properties did not change during storage, except for EC oleogel. Structuring triggered a depletion in phenolic content and alpha-tocopherol, which was more pronounced when a higher temperature was required for oleogel preparation (MG ~ RW < PS < EC). However, during storage phenolics and alpha-tocopherol decreased following zero-order kinetics with a higher susceptibility in unstructured oil, suggesting in all cases a protective effect of the gel network.

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LOQ HEN Comparative study of the anti-obesity and gut microbiota modulation effects of green tea phenolics and their oxidation products in high-fat-induced obese mice

Liu, Z., et al., Food Chem. 367: 130735, 2022, https://doi.org/10.1016/j.foodchem.2021.130735.

Green and black teas are regarded to possess therapeutic potential for the treatment of obesity, but it is not clear which tea performs better in body weight control. In this study, aiming to eliminate cultivar variation, green tea phenolics (GTP) were oxidized by tyrosinase to obtain oxidized tea phenolics (OTP). Thereafter, their anti-obesity effect on high-fat diet induced obese mice were compared. The results showed that despite their distinctive phenolic profiles, GTP and OTP exerted similar anti-obesity properties after 12 weeks of dietary intervention. Furthermore, cecal microbiota profiling exhibited comparable modulatory effects of GTP and OTP on multiple bacterial taxa, including Parabacteroides distasonis, Bifidobacterium, Prevotella, and Akkermansia muciniphila, which were strongly associated with obesity-related indexes. Putative bacterial function profiling implicated that both GTP and OTP might regulate the lipid metabolism similarly. Collectively, the oxidation of GTP did not influence the anti-obesity and gut microbiota modulatory effects to any large extent.

PRO EAT Ultrasound-assisted sequentially precipitated nickel-silica catalysts and its application in the partial hydrogenation of edible oil

Lim, M.S.W. et al., Ultrason. Sonochem. 73: 105490, 2021, https://doi.org/10.1016/j.ultsonch.2021.105490.

Sequentially precipitated Mg-promoted nickel-silica catalysts with ageing performed under various ultrasonic intensities were employed to study the catalyst performance in the partial hydrogenation of sunflower oil. Results from various characterization studies showed that increasing ultrasonic intensity caused a higher degree of hydroxycarbonate erosion and suppressed the formation of Ni silicates and silica support, which improved Ni dispersion, BET surface area, and catalyst reducibility. Growth of silica clusters on the catalyst aggregates were observed in the absence of ultrasonication, which explained the higher silica and nickel silicate content on the outer surface of the catalyst particle. Application of ultrasound also altered the electron density of the Ni species, which led to higher activity and enhanced product selectivity for sonicated catalysts. The catalyst synthesized with ultrasonic intensity of 20.78 Wcm⁻² achieved 22.6% increase in hydrogenation activity, along with 28.5% decrease in trans-C18:1 yield at IV = 70, thus supporting the feasibility of such technique.

IOP PRO Microbial biodiesel production from lignocellulosic biomass: new insights and future challenges

Uthandi, S., et al., Crit. Rev. Environ. Sci. Technol. 1–30, February 11, 2021, https://doi.org/10.1080/10643389.2021.1877045.

In many countries, biodiesel production is obstructed because of a high production cost accounting for raw materials, the large acreage needed for the cultivation of oil-yielding vegetable crops, and competition between food and feed. Therefore, biodiesel production requires new approaches for which microbial oils offer a potential solution. Among several microorganisms available, oleaginous microorganisms (yeast and fungi) accumulate more than 20-70% oils inside their cells when grown in specific environmental conditions. Moreover, microbial oils or single cell oils (SCOs) offer numerous advantages over vegetable oils or animal fats such as similar fatty acid profile, short life cycles of the microbes, relatively lower environmental impact, reduced labor demand, and convenient scalability. Microbial lipids production using lignocellulosic biomass (LCB), which are naturally available in abundance, as a renewable raw material for producing second-generation biodiesel, has become a fundamental approach for tackling the challenges we face of higher energy costs, protection of the environment, and rapid depletion of crude oil reserves. This review compares and examines the extent to which different microbes can accumulate a productive level of lipids using LCB as substrates, pretreatment strategies used for converting LCB into SCOs, and future challenges in using LCB for biodiesel production.

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