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New developments in vegetable oil materials science

Rebecca Guenard

- Long before petrochemicals became the dominant starting material, manufacturers used vegetable oils to make coatings, adhesives, and other types of polymers. Researchers are now optimizing vegetable oil chemistry with an eye toward making sustainable products with properties that gain the same level of customer satisfaction as their petrochemical counterparts.
- Electrolysis and ozonolysis are manufacturing methods that reduce reagent use and unwanted side reactions. A major vegetable oil company has brought ozonolysis technology online to make plant-based products quicker and cleaner.
- Before the pandemic, the bio-based polymers market was projected to have huge growth in the next five years. COVID-19 has caused unemployment and plummeting petroleum prices; how will these factors affect the bio-based sector?

In 1941, Henry Ford produced a new type of car body. It was made of a metal frame surrounded by soybean-based plastic panels. The company never documented the formula for the panels on the Soybean Car, so there is no way to know if Ford made the bio-plastic by manipulating the oil's chemistry the way it is done today (https://tinyurl.com/soybeancar).

"Making natural resin is not a new idea," says Jonathan Curtis, associate professor and principle investigator at the University of Alberta's, Biorefining Conversions Network, in Edmonton, Canada. Protective coatings for furniture and wood floors were once sourced from conifer sap and insect exudate. But synthetic materials made from petrochemicals significantly outperformed natural polymers, and natural materials fell out of vogue.

Some materials scientists want to make natural products mainstream again. By addressing technical problems and considering the mechanical properties needed for a given application, they are developing novel chemical formulations to turn vegetable oils into a range of polymer materials.

Vegetable oils are a mixture of unsaturated fatty acids with double bonds that act as reactive sites where the molecules can be chemically modified. Oleic acid, linoleic acid, and linolenic acid contain one, two, and three double bonds, respectively. Since different vegetable oils have different fatty acid compositions, researchers have an opportunity to create different materials. The potential for a new variety of vegetable oil-derived materials coincides with growing consumer interest in environmentally friendly, sustainable products in the past decade. Will the post-COVID economy threaten their chance of success?

EPOXIDES

According to market research, the projected global market value for epoxy resins will exceed $11 billion in the next two years, a nearly 70% increase (https://tinyurl.com/resinsmarket). Petrochemical products currently dominate the market, but vegetable oil epoxides continue to battle for a share. Analysts believe that recent regulatory mandates for non-phthalate plasticizers, coupled with consumer demand for eco-friendly materials, will give naturally based polymers a boost.

"Producing vegetable oil epoxides is a low-cost, well-established process using simple chemistry that is relatively benign," says Curtis. The reaction uses formic or acidic acid and peroxide with only water as a waste product (Fig. 1) and is fairly clean, he says.

However, linking the epoxides together to form a hard plastic involves overcoming inherent challenges. The organic acids, commonly used as curing agents, do not mix with oil oxides and no reaction occurs. A solvent can be added, but that introduces toxicity to a reaction that is intended to be clean.

"There are more technical problems using vegetable oil-based epoxides because of their poor solubility with some of the more polar crosslinking agents," Curtis says.

In an effort to address these problems, scientists have studied the kinetics of common curing agents to understand how they influence the physical and mechanical properties of resulting resins (https://doi.org/10.1002/aocs.12260). Some researchers found that using bio-based curing agents (Fig. 2, page 8) improved the properties of a polymer made from epoxidized vegetable oil (https://doi.org/10.1016/j.cej.2017.06.039). Whatever the approach, materials scientists now understand that to create a valuable vegetable oil-based polymer they must consider each aspect of the polymer: the monomers, curing agents, and additives.

"Nobody is going to have great commercial traction on the vegetable oil epoxy materials front without addressing the whole package," Curtis says. "You cannot just work on the oil epoxides; you have to design the whole system." Part of working with natural products is thinking about the chemistry in a different way, he explains. A synthetic plastic is made from one monomer and leads to one type of polymer. When you start with a mixture of fatty acids from vegetable oils you cannot

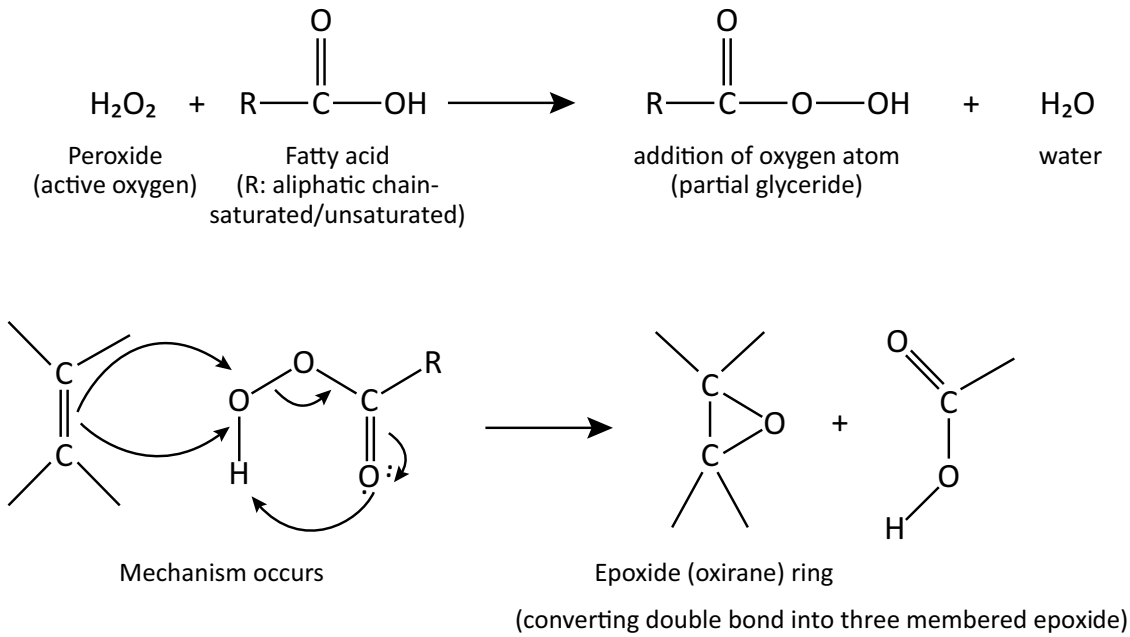

FIG. 1. The mechanism for epoxidation of fatty acids in vegetable oils. Source: Gamage, *et al.*, *J. Natl. Sci. Found. Sri Lanka 37*: 229–240, 2009.

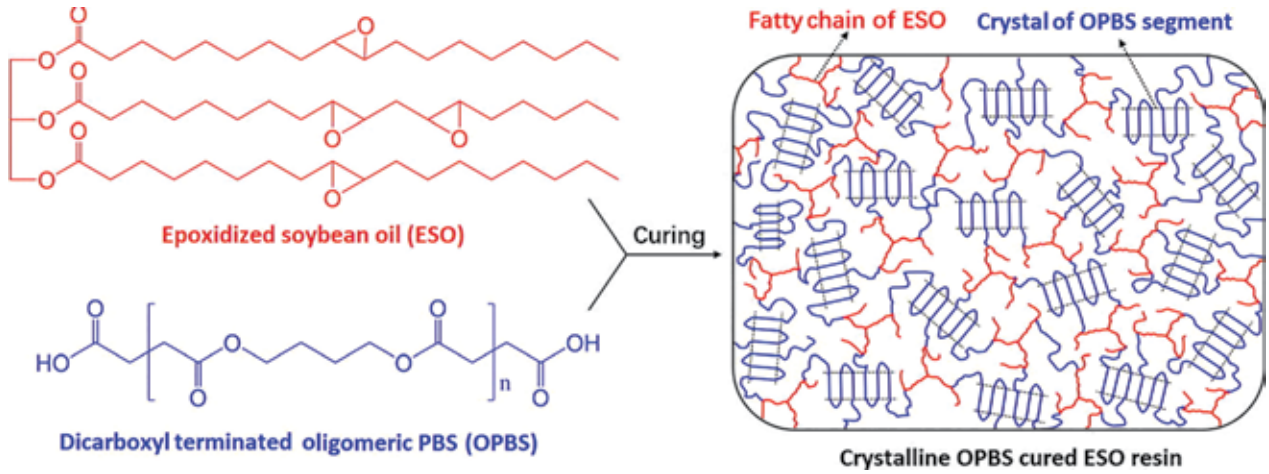

FIG. 2. Crosslinking of an epoxidized soybean oil with a bio-based curing agent. Source: Jian, *et al.*, *Chem. Eng. J. 326*: 875–885, 2017.

identify a single polymer, just the overall properties you want the end product to have.

Curtis says that genetic modification and controlled growth of oilseed plants can produce oils that are better suited for materials applications. "For example, castor oil naturally produces one hydroxyl fatty acid that is 95% of the oil," he says. "Instead of canola or soybean, where you have 10 different fatty acids, you essentially have a pure compound coming out of the plant." Biotechology can also produce high-purity designer oils that serve as better feedstocks for chemical processes.

Even if the challenges arising from different chemistries can be overcome, the existing manufacturing infrastructure of the petrochemical industry gives it an economic advantage over vegetable oil-based materials. To compete, natural materials manufacturers need innovative, low cost technologies that are easy to establish.

OZONOLYSIS

A simple, efficient way to produce oxidation products of free fatty acids is by reacting ozone across their double bonds. Manufacturers typically avoid this process because of the explosiveness of the bulk reagents. Now, a company has found a way to optimize the benefits of ozonolysis while avoiding the dangers.

FIG. 3. Liquid-cooled flow-through reactors inside the P2 Science ozonolysis plant. Source: P2 Science.

The company P2 Science (https://www.p2science.com/) in Woodbridge, Connecticut, USA, launched in 2011, with the mission of rethinking how to make specialty chemicals. Their goal: greener manufacturing using ozonolysis (Fig 3). "What we have done at P2 is make ozonolysis easier to control in an industrial setting," says the company's CEO, Neil Burns. Central to P2's strategy, Burns says, is its continuous-flow technology, patented with the European engineering firm Desmet Ballestra.

In P2's continuous-flow reactor system, a thin film of reactant combines with ozone as it runs down the walls of a liquid-cooled tube. "We use very little reactant at any given time compared to the traditional process where tons are being used," says Burns. "It is a continuous, controllable process where ozone enriched air and a vegetable oil or terpene flow concurrently."

This summer, P2 Science and ADM (http://www.adm.com/), based in Chicago, Illinois, announced a joint development agreement to commercialize plant-based monomers and polymers using ozonolysis. Initially, the companies will make high-value products like nylon and polyester for applications in paints and coatings, automotive, construction, and personal care and industrial cleaning industries, among others. Paul Bloom, ADM's vice president of sustainable materials, says the partnership provides an opportunity for his company to broaden its plant-based portfolio.

Bloom says that for many years ADM has been interested in ozonolysis as a way to modify vegetable oils cleanly. "The end products are used everywhere—from intermediates for the agricultural industry to cleaning and personal care products," he says. "These products are made more sustainably, because you can basically use air and electricity to generate ozone, and when you are done there is minimal waste."

Burns also points out that the flow-through design of the process means that it is easily scalable. "This is a modular process that can go from tens to hundreds to thousands of tons of product produced with minimal change in the characteristics of the reactor," he says. In that way, the process is more conveniently scaled than a process like fermentation where a complete recalibration would be necessary. "We will be able to adjust production effortlessly to meet demand," says Burns.

EXPLORING THE POTENTIAL OF ELECTROLYSIS

As AOCS members certainly know, water is a very stable liquid. But, with enough electricity running through it, water will break down into hydrogen and oxygen. A growing trend among chemical companies is testing whether renewable energy and cheap electrolysis can be used to make industrial chemicals from carbon dioxide and water. In late 2019, the German companies Evonik and Siemens built a facility together to test the technology (https://tinyurl.com/cenbusiness).

Now, there is a possibility that electrolysis could be used to epoxidize vegetable oils. Karthish Manthiram, engineering professor at Massachusetts Institute of Technology, in Cambridge, and his team recently published a paper in the *Journal of the American Chemistry Society*, describing the electrochemical epoxidation of olefins (https://doi.org/10.1021/jacs.9b02345). Manthiram says the conventional way of mak-

ing epoxides creates surplus carbon dioxide. "When you try to drive that reaction using temperature and pressure, there is a tendency for over oxidation," he says.

Manthiram's team set out to improve the mass balance of epoxide manufacturing while also developing a safe, sustainable process. They suspended electrodes in a mixture of water and acetonitrile that contained an olefin, and coated the negative electrode in magnesium oxide nanoparticles. Magnesium oxide is known to generate oxo-species during water oxidation, which they hoped would transfer to the olefin. The electrolysis reaction not only worked, it operated with an impressive 30% electron efficiency.

The group will work on improving the process and eventually expand the technology if it is successful. Currently, the reaction loses efficiency as the olefin chain length increases, but Manthiram sees a possibility for applying electrolysis to the epoxidation of vegetable oils in the future.

"We developed this process in the hopes of being able to apply it to starting material like ethylene and propylene," Manthiram says. With the right combination of catalyst and a method for dispersing the vegetable oil in the desired solvent, he says long-term development could include the selective epoxidation of vegetable oils.

AN ECONOMIC CURVE BALL?

Right now, demand for materials made from vegetable oils is uncertain. As with traditional polymers, the industry is cyclical, but the current market is unprecedented and difficult to read—although sustainability does seem to have a stronghold on consumer spending.

Plant-based polymers may only be a minor fraction of the industry, but they have enjoyed steady market growth as oil prices climbed, making them competitive against the price of synthetic polymers. At the same time, a growing number of major consumer goods companies partnered with green materials producers to create sustainable, eco-friendly packaging. Not every wrinkle had been smoothed out of vegetable oil-based polymers, but the industry was starting to gain traction. Now the coronavirus pandemic threatens to upend that progress.

The halt of transportation, global and domestic, from COVID-19 shutdowns resulted in a surplus of petroleum, and prices plunged. Meanwhile, the pandemic has sparked a demand for single-use packaging due to a rise in the number of people eating take out. In addition, retailers banned reusable bags fearing they could transmit the virus. Market analysts report that the natural plastics industry may be saved by these factors, but they indicate that the industry's future will be determined in the next few months (https://www.plasticstoday.com).

This summer, Genomatica, an ingredient company in San Diego, California, USA, commissioned an independent survey within the United States that contradicts any negative forecast for bio-based polymers (https://tinyurl.com/sustainabilitysurverygenomatic). In an interview with *Forbes*, Genomatica's co-founder and CEO, Christophe Schilling, explains how staying home made people realize their environmental impact more profoundly (https://tinyurl.com/sustainabilitysurveryforbes). Pollution cleared, and many people in the world experienced clean air and water for the first time in their lives.

Based on their survey results, Genomatica reports that amid the pandemic "sustainability has moved from a fringe preference to a core imperative across American life," according to the *Forbes* article. The study found that 85% of Americans reported they have been thinking about sustainability the same amount or more during COVID-19 quarantine. And a third of participants said they were willing to pay a little more for sustainable products, even during an economic downturn. When the participants were categorized by generation, nearly half of the younger consumers (25–40 years old) said replacing synthetic products with alternatives containing natural ingredients was important. We will have to wait to see how these responses compare to the reality of the world economy in the next decade, but they do indicate that consumers are prioritizing the environment.

Curtis says, more and more researchers see the value in the extra effort required to make vegetable oil systems suitable for new applications; they just need manufacturers who are dedicated to sustainability. "If you can replace some portion of petrochemicals with vegetable oils, then that is a win for the people growing the beans and that is a win for the environment," Curtis says.

In terms of performance and cost, petrochemical materials still outweigh vegetable oil materials. The petrochemical industry has had a 150-year head start. Its development and manufacturing infrastructure are well-established. But, Curtis says, it does not really make sense to directly compare products from the two industries. Just as milk jugs and grocery bags made of polyethylene were inconceivable before that material was developed, there are possible applications for vegetable oil materials that remain unknown. "Polymers are widely used in all kinds of places," he says. "There are so many applications that surely we can find ones that work well with vegetable oil-based products."

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

Information

AOCS journal

Isothermal curing kinetics of epoxidized fatty acid methyl esters and triacylglycerols, Li, Y., *et al.*, *J. Am. Oil Chem. Soc. 96*, 9: 1035–1045, 2019.

Epoxidation of cyclooctene using water as the oxygen atom source at manganese oxide electrocatalysts, Jin, K., *et al.*, *J. Am. Chem. Soc. 141*: 6413, 2019.

Curing of epoxidized soybean oil with crystalline oligomeric poly(butylene succinate) toward high performance and sustainable epoxy resins, Jian, X.Y., *et al.*, *Chem. Eng. J. 326*: 875–885, 2017.

Epoxidation of vegetable oils: A review, Tayde Saurabh, *et al.*, *Int. Adv. Eng. Technol. 2*: 491–501, 2011.

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Evaluation of avocado oil sold in the United States

Hilary S. Green and Selina C. Wang

Avocado oil has grown in popularity due to its mild flavor and health benefits, such as high oleic acid and antioxidant contents. Sales continue to increase in the United States; however, the projected growth of the avocado oil market is being stifled by one major barrier: There are no standards. This means there is no standardized way to test if an avocado oil is pure and of the quality advertised on the bottle. This lack of standards can give rise to economically motivated adulteration, or to the addition of cheaper or lower-quality oils to products labeled as pure avocado oil. This is done to decrease selling price and unfairly increase competition in the market.

- **There is an urgent need to develop standards for avocado oil to ensure that consumers receive the quality and authentic products they choose to purchase based on the labels. Standards are also needed to establish a level playing field for genuine producers to support continuing growth of the avocado oil industry.**
- **Adulteration with soybean oil at levels near 100% was confirmed in two "extra virgin" and one "refined" sample. Nine other samples had values outside proposed limits for avocado oil purity.**
- **The majority of commercial avocado oil samples, both extra virgin samples and refined, were of poor quality and oxidized before reaching the expiration date listed on the label. Best practices for harvesting, post-harvesting, processing, and storage of avocado oils need to be developed and followed.**

The olive oil industry has dealt with similar problems for years; an *Inform* article, "Questioning the virginity of olive oils" by Watkins, summarized our group's research results in 2010. A key difference in testing the quality and purity of avocado oil is that, unlike with olive oil, there are no established standards and limits for avocado oil. Without standards, consumers, bulk buyers, food service professionals, and genuine producers are unprotected from fraud, and there is no way to ensure that avocado oil is pure, of adequate quality, and safe. To help support the development of standards in the avocado oil industry, we determined the quality and purity of 22 avocado oils currently sold in the United States. We believe this snapshot of the current market demonstrates the importance of enforceable standards, regulations, and education.

The samples in this study were separated into three categories based on their label: extra virgin (EV), refined (R), and unspecified (U). Extra virgin oils are produced from high-quality fruit and extracted only via mechanical methods, without the use of solvent. Refined oils have been through processes where acids, alkalis, and heat are applied for bleaching and deodorization. Oils that had ambiguous labels or no specifications were placed in the (U) category. Table 1 summarizes

Editor's Note: A Virtual 2020 AOCS Annual Meeting & Expo presentation on a related topic by Selina Wang, "Introduction: Authentication of high-value oils, including olive oil," can be accessed at annualmeeting.aocs.org until 2021.

the sample information for each oil, including the purchasing method, expiration date, product origin, cost, and packaging type. Purity is black and white: If an oil is pure, there are no additives or oils present in the bottle other than what is labeled on the ingredient list. Quality is less black and white: There are many variables that affect the quality of an oil, and quality continues to change as oxidation takes place over time. Damaged fruits, improper extraction processes, and storage can all lead to a poor-quality product.

Oil quality was determined using three main parameters: free fatty acids content (FFA), peroxide value (PV), and specific extinction in UV (shown as ΔK) as seen in Figure 1 on the next page, parts a, b, and c, respectively. The presence of free fatty acids in an oil is caused by the separation of fatty acids from a triacylglycerol (TAG); a higher FFA value indicates poorer quality. Figure 1a shows that extra virgin samples had higher FFA than refined and unspecified samples; this is expected, as the refining process removes free fatty acids. Although there are

TABLE 1. Sample information for the 22 avocado oils tested in this study

Sample code	Purchasing method	Expiration date (month-year)	Product origin	Cost/fl oz ($)	Packaging type
EV1	Online	Oct-21	USA	2.23	Dark glass
EV2	In store	Jun-21	USA	1.29	Dark glass
EV3	In store	Feb-21	Mexico	0.65	Dark glass
EV4	In store	Sep-20	USA	1.53	Dark glass
EV5	Online	Jul-21	USA	1.57	Dark glass
EV6	Online	NA	Brazil	0.49	Clear plastic
EV7	Online	Jun-21	USA	2.35	Dark glass
R1	Online	Jun-21	Spain or Mexico	0.44	Dark plastic
R2	In store	Aug-20	Mexico	0.74	Dark glass
R3	In store	Nov-20	Mexico	0.43	Dark glass
R4	Online	Dec-20	Mexico	0.35	Clear plastic
R5	In store	May-20	Mexico	0.25	Dark plastic
R6	In store	Jul-20	Mexico	0.77	Dark glass
R7	Online	Dec-19	Mexico	0.80	Dark glass
R8	In store	Apr-21	Mexico	1.44	Clear glass
R9	In store	Apr-21	Mexico, USA, or Spain	0.29	Clear plastic Clear plastic
U1	In store	NA	Mexico	0.29	Dark plastic
U2	In store	Apr-21	Mexico, USA, or Spain	0.66	Tin Bottle
U3	In store	Mar-21	Mexico, USA, or Spain	0.71	Tin Bottle
U4	In store	May-21	Mexico	0.47	Dark glass
U5	In store	Jun-21	Mexico	0.79	Dark glass
U6	Online	Feb-21	Mexico	0.34	Clear plastic

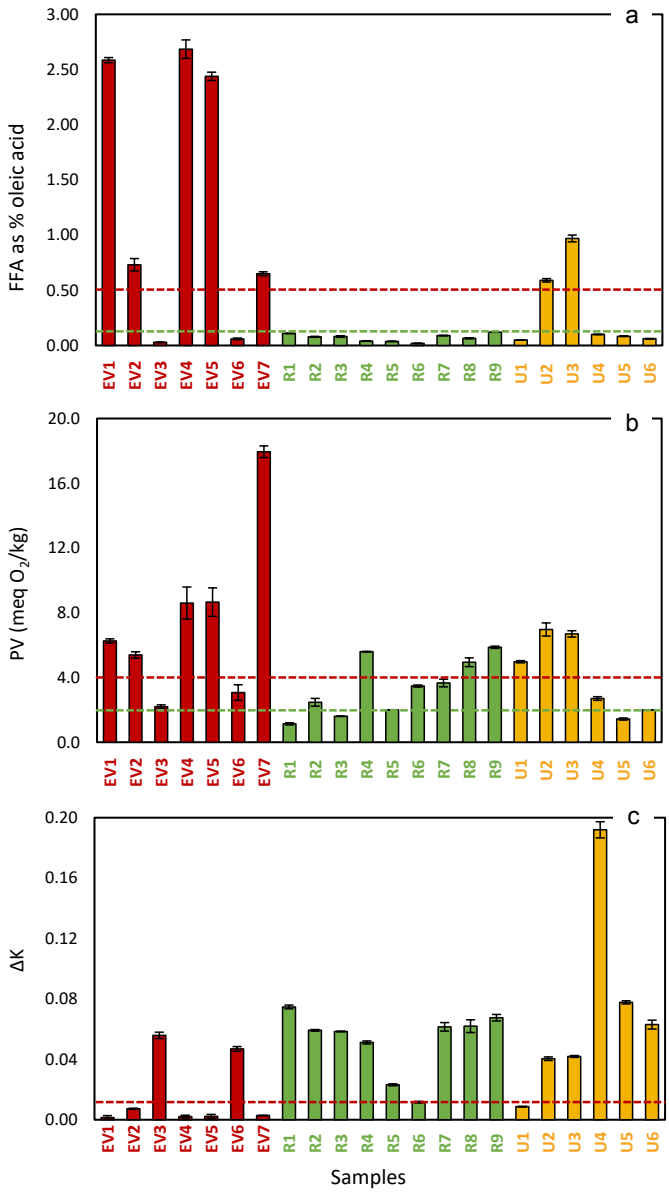

FIG. 1. (a) Free fatty acid content reported as % oleic fatty acid. (b) Peroxide values reported as milliequivalents of O_2/kg of oil. (c) Values for ΔK. Bars are an average of triplicate measurements and error bars show standard error of the mean. EV stands for extra virgin (red), R for refined (green), and U for unspecified avocado oil (gold). Dashed lines indicate the proposed limits for avocado oil with the red dashed line corresponding to limits set for extra virgin avocado oils and green dashed line indicating limits for refined avocado oils.

no official standards for avocado oil, Allan Woolf and coworkers proposed a set of standards for avocado oils. In these standards, refined avocado oils should have an FFA<0.1% (shown on Figure 1a with green dashed line) and for extra virgin oils FFA<0.5% (red dashed line). The refined oils were all at or under the proposed 0.1% FFA limit as are most of the unspecified oils. However, five of the seven extra virgin oils had FFA values higher than 0.5%, with three samples reaching 2.5%, indicating very poor quality. This could be caused by overripe, damaged, bruised, pest-infected fruits, or improper post-harvesting and processing practices.

The peroxide value (PV) measures the primary oxidation products which can be formed if an oil is exposed to oxygen, light, or from extended storage times. Woolf and coworkers proposed the maximum acceptable PV for extra virgin avocado oils should be 4.0 meq O_2/kg. Figure 1b shows five of the seven extra virgin samples are over this limit, where only EV3 and EV6 had low PV and FFA. Woolf's maximum PV limit for refined oils is 0.5 meq O_2/kg. The international standard development agency CODEX Alimentarius also has a proposed set of standards for avocado oils, which sets the maximum PV for refined avocado oils at 2.0 meq O_2/kg. Going by the more lenient CODEX standards (shown as green dotted line in Figure 1b), only three refined oils and two unspecified oils were under the limit. It is possible the high PV seen in the samples resulted from long storage time and clear packaging, which promote oxidation. The refined oils with the highest PV (R4, R8, and R9) were stored in clear packaging instead of tinted bottles, which helps to protect against oxidation from light.

The indicator ΔK, determined through UV-Vis spectroscopy, is used to differentiate an extra virgin/unrefined oil from one that is refined. There are currently no proposed avocado oil standards for ΔK; therefore, the limit used by the International Olive Council (COI/T.15/NC No 3/Rev. 14) for extra virgin olive oil (ΔK< 0.01) is used for comparison. Figure 1c shows all the extra virgin oils were below 0.01 except EV3 and EV6. This is interesting, as these samples had the lowest FFA and PV values. This indicates that EV3 and EV6 are not

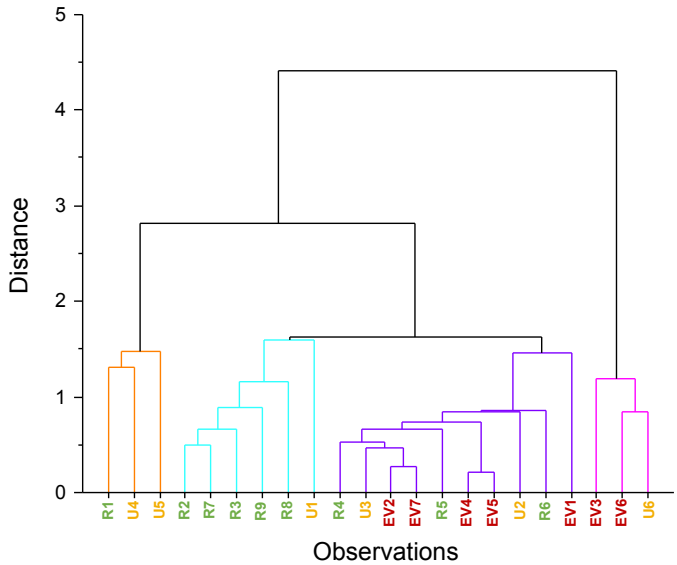

FIG. 2. Dendrogram showing hierarchal cluster analysis of samples using fatty acid profile, sterols, triacylglycerols, and tocopherols as variables. Colors (orange, blue, purple, and pink, from right to left) show the four main groups derived from the analysis. EV stands for extra virgin (red), R for refined (green), and U for unspecified avocado oil (gold).

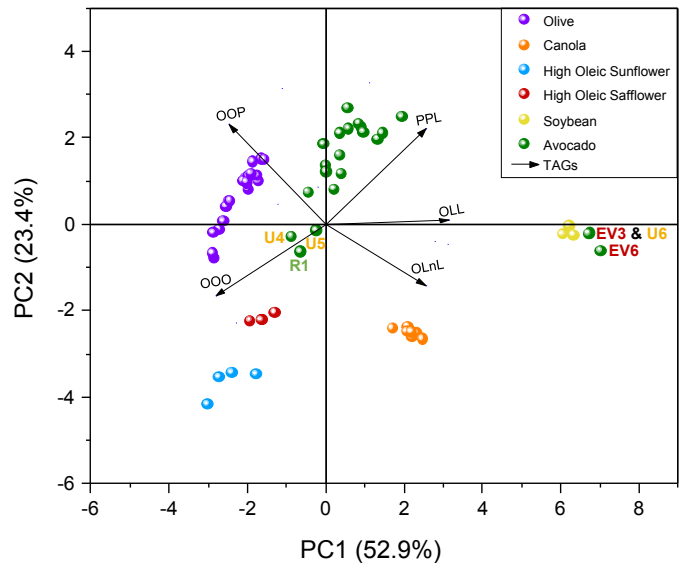

FIG. 3. PCA plot created using the triacylglycerol (TAG) profiles for each oil. Five select TAGs are shown on the plot for reference. The six avocado oils that differed notably from other samples are labeled according to their sample codes: EV stands for extra virgin (red), R for refined (green), and U for unspecified avocado oil (gold). All other avocado oils from this study are labeled as avocado, shown in dark green.

extra virgin and contain oil that has gone through refining. These oils were light in color and had substantially less chlorophyll than the other extra virgin samples (data not shown). They were also lower in price—under $0.75/fl oz, with the other extra virgin oils above $1.25/fl oz. As expected, all the refined oils and all but one unspecified oil had ΔK above 0.01, confirming these oils have been refined.

The purity of the oils was determined using the fatty acid profile (FAP), sterols profile, and triacylglycerol analysis (TAGs). The tocopherol (an antioxidant) content in the oils was also determined. A list of these parameters for each oil is detailed in Green, *et al*., 2020 (https://doi.org/10.1016/j.foodcont.2020.107328). Consistent trends were seen between the purity and tocopherol data, thus hierarchal cluster analysis was used to group the oils based on these parameters. Figure 2 shows that EV3, EV6, and U6 are in a separate branch (in pink), indicating they had many differences from the other oils in this study. Upon examination of their parameters, these oils were significantly outside the range of the CODEX proposed purity standards for avocado oil; however, they were within the ranges for soybean oil. The orange group of oils (R1, U4, and U5) had values for their fatty acids and sterols profiles that were outside the range of 2xSD from the confirmed pure samples in this study (purple). In particular, they had lower palmitoleic acid and higher stearic acid than CODEX proposed standards. They also had less beta-sitosterol than previously seen in avocado oils, as well as higher amounts of several other sterols.

The majority of samples in this study are in the blue and purple branches. The oils in blue are separated due to slightly elevated levels of stearic acid above the proposed standards. The differences seen in the purity data for the samples in orange and blue groups demonstrates the challenges with standard development. Sometimes these purity parameters can change depending on processing conditions of the oil and growing conditions of the fruit. It is essential to better understand how much the impact is so we can determine if the differences seen in the blue and orange samples are due to natural variance or to economically motivated adulteration.

To compare the composition of oils in this study to other edible oils, we took a look at the TAG profiles using a method previously developed in our group (https://doi.org/10.1016/j.foodcont.2019.106773). Figure 3 shows all the avocado oils in this study (in dark green) and several other types of oils, which are grouped according to their TAG profile. We can see that EV3, EV6, and U6 are next to the soybean cluster, further confirming that these samples are soybean oil, not avocado oil. R1, U4, and U5 (the orange group from Figure 2) are removed from the rest of the avocado oils in this study and are located halfway between the other avocado oils and the high-oleic sunflower and safflower oils. Preliminary calculations based on FAP and sterols indicate that the adulteration of avocado oil with 50% high-oleic safflower or sunflower oil leads to values similar to those seen in R1, U4, and U5.

This study demonstrates that there is an urgent need for standards in the avocado oil industry. Two extra virgin avocado oils were adulterated with soybean oil at levels near 100%. The other extra virgin oils were oxidized, and the majority of refined oils and unspecified oils had also oxidized above proposed limits. This indicates that best practices for harvesting,

post-harvesting, processing, and storage of avocado oils need to be developed and followed. Adulteration with soybean oil was also confirmed for an unspecified sample, and adulteration with high-oleic sunflower or safflower oil was highly suspected in three other oils. It is essential to better understand how climate, harvest time, and processing conditions impact the variation seen in avocado oils to develop fair standards that provide confidence in detecting adulteration, while accommodating the natural variances of the global avocado oil industry.

Hilary S. Green is a PhD student in environmental and agricultural chemistry at the University of California Davis. Her work focuses on understanding quality, purity and safety in the edible oils and developing better chemical methods to help address problems in the industry. So far, she has two publications from her PhD work and has presented at American Chemical Society. She can be contacted at hsgreen@udavis.edu.

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Exploring pulse ingredients as egg replacement solutions in food systems

Shuyang (Zoe) Wang, Praiya Asavajaru, Angie Lam, Yvonne Lu, and Mehmet Tulbek

- **A substantial void exists in the plant-based egg replacement market.**
- **Consumers' growing interest in more sustainable plant-based diets and demand for allergen-free products are driving food manufacturers to seek egg-free alternatives.**
- **Pulse ingredients, which contain significantly lower saturated fat and zero cholesterol but more dietary fiber and minerals than eggs, demonstrated comparable functionality with respect to emulsification, structure development, foam stabilization, gelation, and browning.**

Pulses are edible dry seeds (dry peas, lentils, chickpeas, faba beans, and edible beans) within the nitrogen-fixing legume family (*Fabaceae* or *Leguminosae*). They are considered nutritious, as they are typically low in fat but high in protein (21–30%), dietary fiber (~19%), vitamins, and minerals (~3%). Pulses also fit with the top-ranking food claims demanded by consumers, such as non-GMO, high fiber, gluten-free, and allergen-free, making them increasingly go-to ingredients for plant-based dietary alternatives.

Protein and starch are the major functional pulse ingredients. Different milling technologies are used to obtain these two fractions. Protein concentrates (55–60% protein) and starch concentrates (>70% starch) are processed through dry milling in combination with air classification, whereas protein isolate (80–90% protein) and starch isolate (>95% starch) are obtained by wet milling.

Consumers rarely seek out egg as an ingredient in food products, but many look for egg-free labels because of the shift toward plant-based diets as well as concerns about egg allergy, common in approximately 2% children under five years of age [1]. This article delves into the science of pulse product development and explains why pulse ingredients could be a great candidate for egg replacement.

The first step toward replacing eggs is to understand their nutritional composition and functional properties as well as those of potential substitutes. The composition of a typical whole egg is approximately 76% water, 13% protein, 10% fat, 0.7% carbohydrate, and 1% ash, while the composition of pulse ingredients varies depending on the type of processing technology. For example, the protein content ranges from 11–22% in starch concentrates, 50–60% in protein concentrates, and 80–90% in protein isolates, whereas the carbohydrate content ranges from 1–4% in protein isolates, 20–30% in protein concentrates, and 70–80% in starch concentrates (Table 1, page 18).

TABLE 1. Composition of pulse ingredients and egg

Parameters	Pulse ingredients				Liquid egg ingredients [2]		
	Pulse starch concentrate	Pulse flour	Pulse protein concentrate	Pulse protein isolate	Whole egg	Egg white	Egg yolk
Moisture (%)	4–10	5–10	4–10	5–10	76.1	87.7	55.0
Protein (%)	11–22	22–30	50–60	80–90	12.6	10.8	15.5
Fat (%)	1–2	2–3	2–4	5–9	9.5	0.1	26.7
Carbohydrate (%)	70–80	55–68	20–30	1–4	0.7	0.9	1.1
Ash (%)	2–3	2–4	5–7	2–3	1.1	0.4	1.7

Eggs are considered an excellent source of protein with a full protein digestibility-corrected amino acid score of 1 (PDCAAS). On the other hand, even though most cooked pulses are highly digestible, most of them have lower PDCAAS (0.5–0.7) compared to eggs. Nevertheless, the majority of pulses are still qualified as a source of quality protein (PDCAAS >0.5) in both Canada and the United States [3]. Globulins and albumins are the major types of proteins in eggs and pulses, respectively accounting for 12% and 71% of the protein in eggs, and 50–80% and 15–25% of the protein in pulses. Globulins are insoluble in water but soluble in dilute salt solutions, whereas albumins are water soluble and can undergo denaturation more readily when heated. Unlike eggs, which are rich in cholesterol and contain almost no fiber, most pulse ingredients contain zero cholesterol but significantly more dietary fiber and minerals (Table 2). Dietary fiber is beneficial for regular bowel movements and weight control, as well as prevention of diabetes, cardiovascular diseases, and certain types of cancer.

One of the main reasons why pulse ingredients are good substitutes for eggs lies in their major functional component: protein. Even though egg protein has greater quality and digestibility than pulse proteins, the quantity of proteins in pulse ingredients is much higher than that of eggs. For example, even the lowest protein-containing pulse ingredient, starch concentrate (11–22% protein), has an equivalent or higher amount of protein compared to a whole egg (~13%); typical pulse flour, protein concentrate, and isolate respectively contain two-, five-, and seven-fold more protein than eggs (Table 1).

In terms of functionality, egg proteins play important roles in various food applications and typically act as emulsifying, structure forming, foaming, gelling, and browning agents. Examples include emulsification and thickening in mayonnaise and sauces, gelation in custards, foam stabilization and struc-

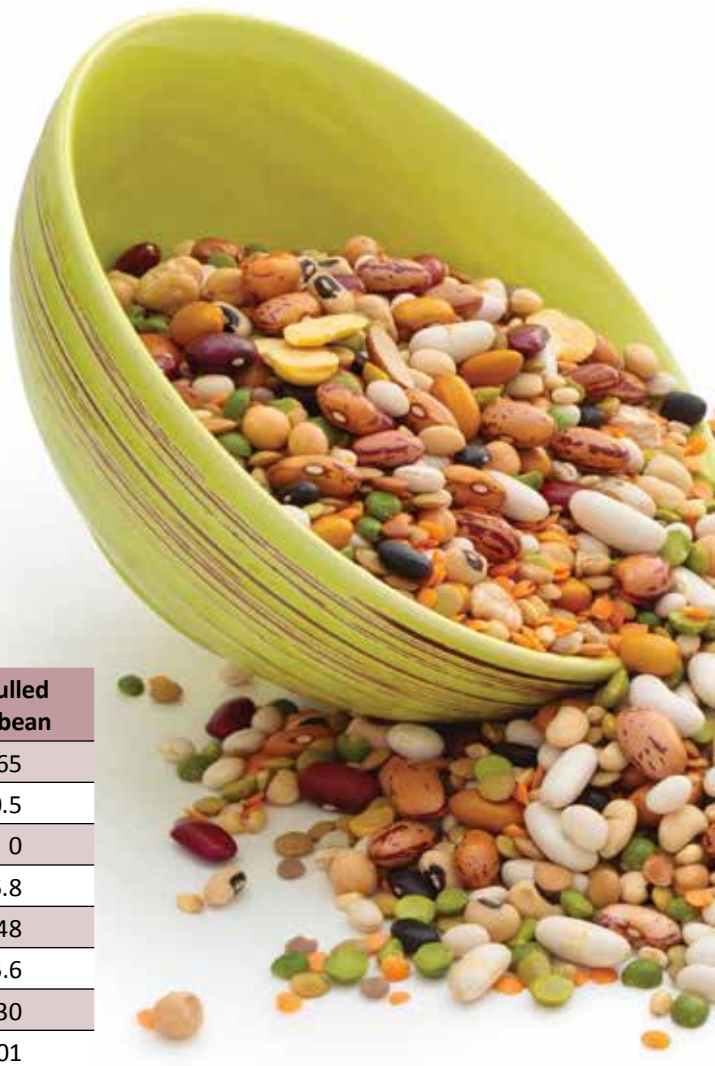

TABLE 2. Example of nutrient comparison between egg and pulses

Per 100g	Egg [2]	Split yellow pea	Dehulled fababean
Calories (kcal)	148	375	365
Saturated fat (g)	3.2	0.4	0.5
Cholesterol (mg)	411	0	0
Total dietary fiber (g)	0	7.4	6.8
Calcium (mg)	48	53	48
Iron (mg)	1.7	5.1	5.6
Potassium (mg)	132	904	1,030
Magnesium (mg)	11	98	101
Zinc (mg)	1.2	3.3	4.2

FIG. 1. Pulse ingredients as egg replacement in mayonnaise, muffin, custard, and egg-wash

ture formation in meringue, nougat, and light cakes, as well as browning and glossing in egg wash. Based on our internal lab trials, many of these functions of eggs are equally achievable with pulse ingredients (Fig. 1).

In the case of emulsification and foam stabilization, both egg and pulse proteins act as emulsifiers—amphiphilic molecules that contain hydrophilic and hydrophobic ends that allow them to bind water to oil or air simultaneously and thus prevent them from separating by reducing surface tension. The results in one phase being suspended as tiny droplets in the other phase, creating a water-in-oil (e.g., margarine) or oil-in-water (e.g., mayonnaise) emulsion. Therefore, the emulsifiers in eggs (e.g., phospholipids, proteins, and lecithin) can be substituted by pulse proteins and further enhanced by pulse starch. Take a wheat-based cake model system as an example (Fig. 2). Pulse proteins will wrap around the hydrophobic air bubbles, oil droplets, and starch granules through hydrophobic interaction in the presence of a gluten network, and thus keep the oil separating from water in the cake batter, forming a mixed colloidal system of emulsion, foam, and suspension. During baking, some of the amylose fraction will leach out.

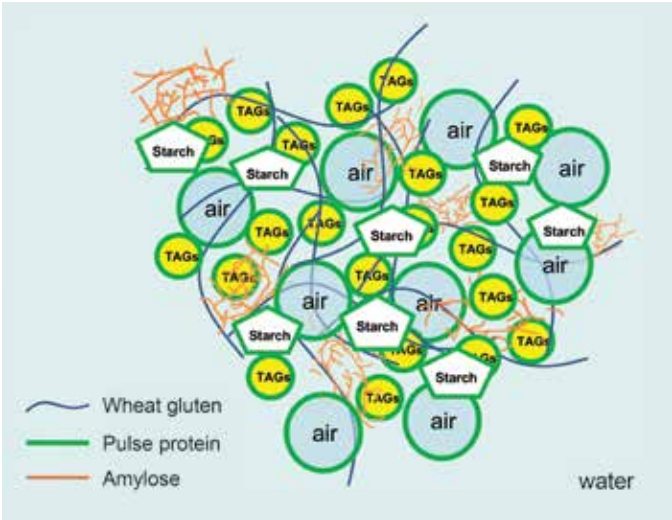

FIG. 2. Demonstration of pulse protein as egg replacement in cake model system

Along with gluten network development and protein interaction, pulse proteins help structure development and improve emulsion stability through physical hindrance; eventually the system is transformed into a solid foam.

Food gels can be made from polysaccharides or proteins. Eggs can form a protein gel upon denaturation by heat, forming a three-dimensional matrix of protein and water through inter-protein hydrogen bonding and immobilization of water within the gel structure. In non-protein pulse ingredients, pulse starches serve as gelling agents as well. Heating starches in water results in a hot solution of amylose that leached out from the swollen starch granules, within which an amylopectin skeleton is left; upon cooling, the matrix will turn into an opaque elastic gel with a minimum starch content of 6%, or a viscoelastic paste with lower starch concentration [4]. Both phenomena were observed during our lab trials in sauce (<6% starch) and custard (>6% starch) formulations.

The browning function of egg arises from the Maillard reaction, a chemical reaction between amino acids and reducing sugars. This reaction not only gives baked goods a golden appearance, but also appealing flavors and smells. Pulse protein concentrates showed outstanding browning capabilities when used as an egg-wash replacement (Fig. 1), which was expected due to its higher levels of protein and sugars compared to eggs.

Free webinar on this topic

The author of this article will be giving a free AOCS webinar on exploring pulse ingredients as egg replacement solutions in food systems at 10:00 AM CDT on November 12, 2020. Register at https://attendee.gotowebinar.com/register/1495063544450950927.

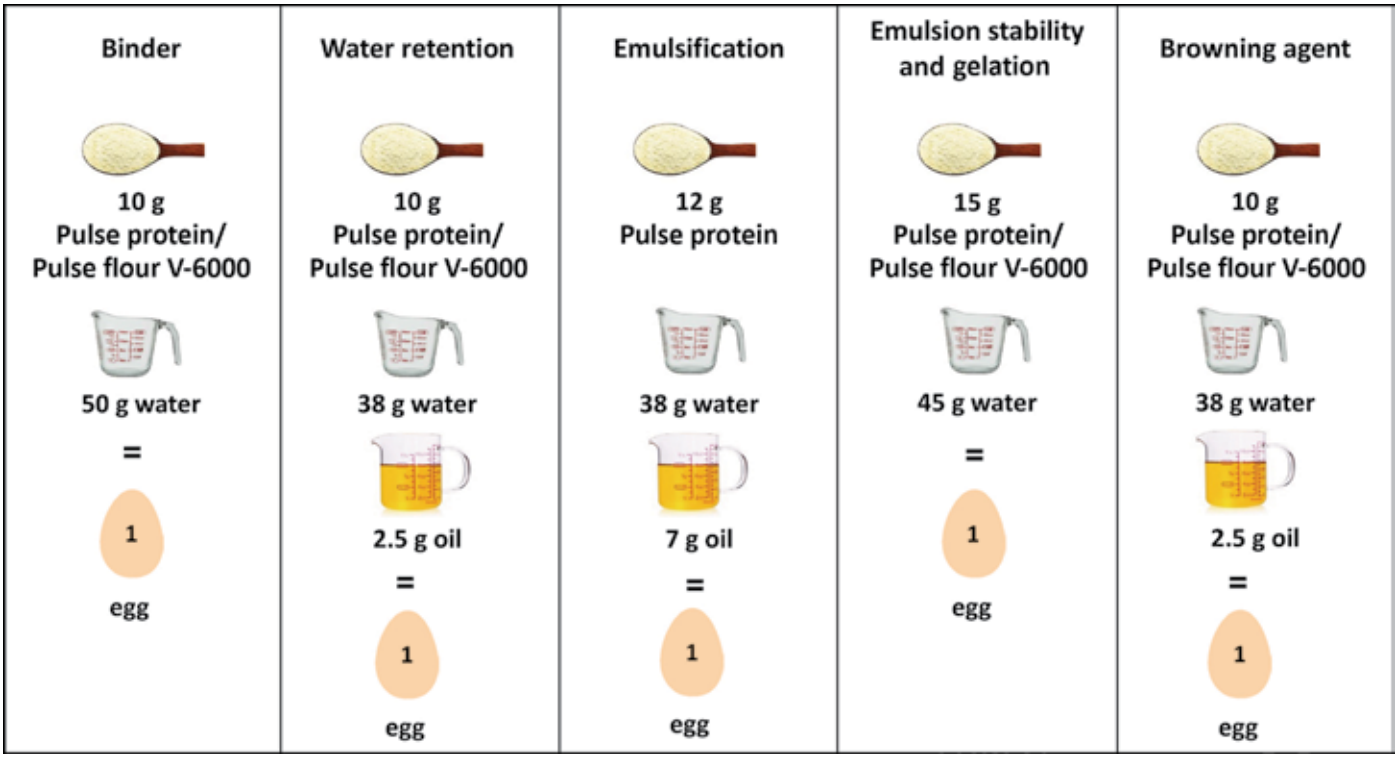

FIG. 3. Pulse ingredients equivalence for one egg

To date, both pulse protein and starch concentrates have demonstrated their potential as egg replacement solutions. Our lab results indicate a simple egg replacement equivalence using pulse ingredients (Fig. 3). As an illustration, replacing one egg in an emulsion system will need 12g of pulse protein, 38g of water, and 7g of oil; for browning and water retention purposes, 10g of either pulse protein or starch concentrate mixed in 38g of water and 2.5g of oil can be equivalent to an egg.

It is worth bearing in mind that although the search for plant-based egg replacement is still in its early stage and awaits much research and development efforts, it is not to be taken lightly since eggs are consumed by over 90% of households and 75% of food service establishments in the United States. Looking forward, plant-based egg replacement, with a projected market share over $1.5 billion by the end of 2026, is believed to follow the example set by plant-based milk that began with virtually nonexistent sales and in just over a decade captured 10% of all fluid milk sales [5].

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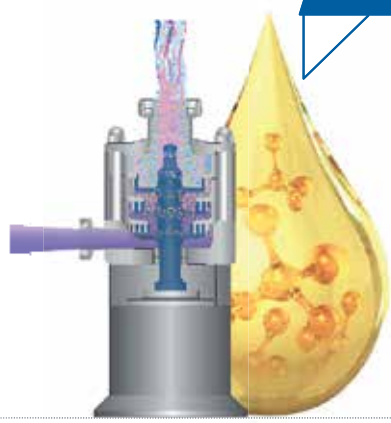

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Rice bran as a potential antidiabetic food material

Hitomi Kumagai, Yusuke Yamaguchi, and Chiaki Sugimoto

The demand for and production of grain are increasing as the world population grows. Rice is one of the world's major grain crops, along with wheat and corn. It is cultivated and consumed as a staple food in many parts of the world, especially in East and Southeast Asia. Rice grains are husked and milled to give endosperm, an edible part of rice, yielding by-products such as rice bran (Fig. 1).

- **Rice is a major crop and a staple food, especially in Asia.**
- **Husking and milling yield a main edible part as well as by-products such as rice bran, which is mostly used for low-value-added products (about 500,000 tons are discarded annually).**
- **The water-soluble protein in rice endosperm (edible part) adsorbs glucose and prevents its absorption from the small intestine, suppressing the increase in blood glucose level.**
- **The water-soluble fraction of rice bran similarly adsorbs glucose effectively, which could lead to the development of new anti-diabetic material for food additives.**

Rice bran is a valuable food source because it is rich in γ-oryzanol, vitamin B_1, high-quality fatty acids, dietary fiber, and protein (Nagendra, *et al*., 2011, Fig. 2). γ-Oryzanol has been used as a raw material in cosmetics and as an antioxidant in foods because of its various activities, such as sebaceous gland activation, ultra-violet absorption, and antioxidant activity (Taniguchi, *et al*., 2012; Kobayashi, *et al*., 2019; Jung, *et al*., 2017). However, after the oil and functional components, including γ-oryzanol, have been extracted,

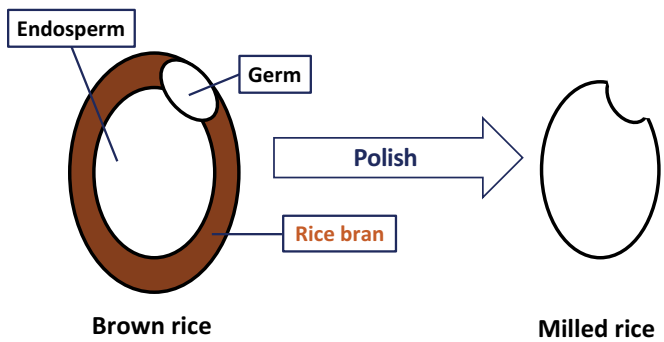

FIG.1. Polishing brown rice

The authors of this article were invited to present this work at the cancelled 2020 AOCS Annual Meeting and Expo.

rice bran is mostly used for low-value-added products like feed, fertilizer, and pickles. About 500,000 tons of rice bran are discarded annually (Nagasaka and Ushio, 2009).

Many studies have been conducted to find valuable uses for rice bran. The physicochemical properties of rice bran proteins, such as foaming, emulsifying, and heat resistance (Chandi and Sogi, 2007; Wang, *et al*., 1999; Tang, *et al*., 2003) as well as their tertiary functions, such as antioxidative properties (Wattanasiritham, *et al*., 2016) and a suppressive effect on blood pressure increase by ACE (angiotensin converting enzyme) inhibition (Uraipong and Zhao, 2016), have been reported in the literature.

SUPPRESSIVE EFFECT ON POSTPRANDIAL BLOOD GLUCOSE ELEVATION

We have already shown that rice endosperm albumin (RA), a water-soluble protein derived from rice endosperm, adsorbs glucose similarly to dietary fibers, such as carboxymethyl cellulose (CMC) and guar gum, and that oral administration of RA

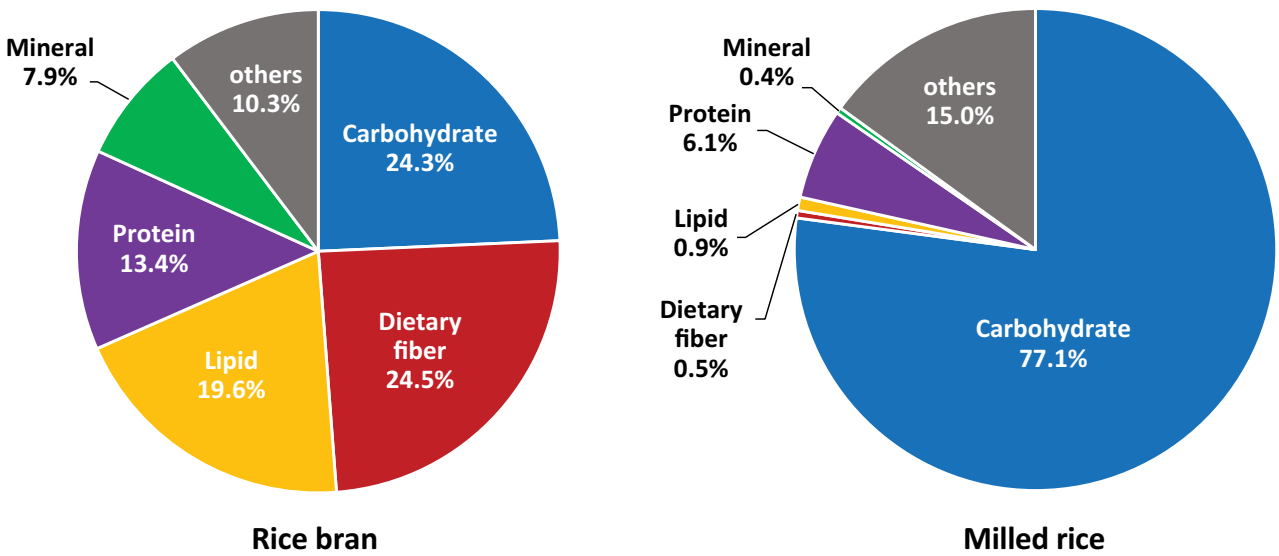

FIG.2. Components of rice bran and milled rice. Source: Food Composition Database (Ministry of Education, Culture, Sports, Science and Technology, Japan)

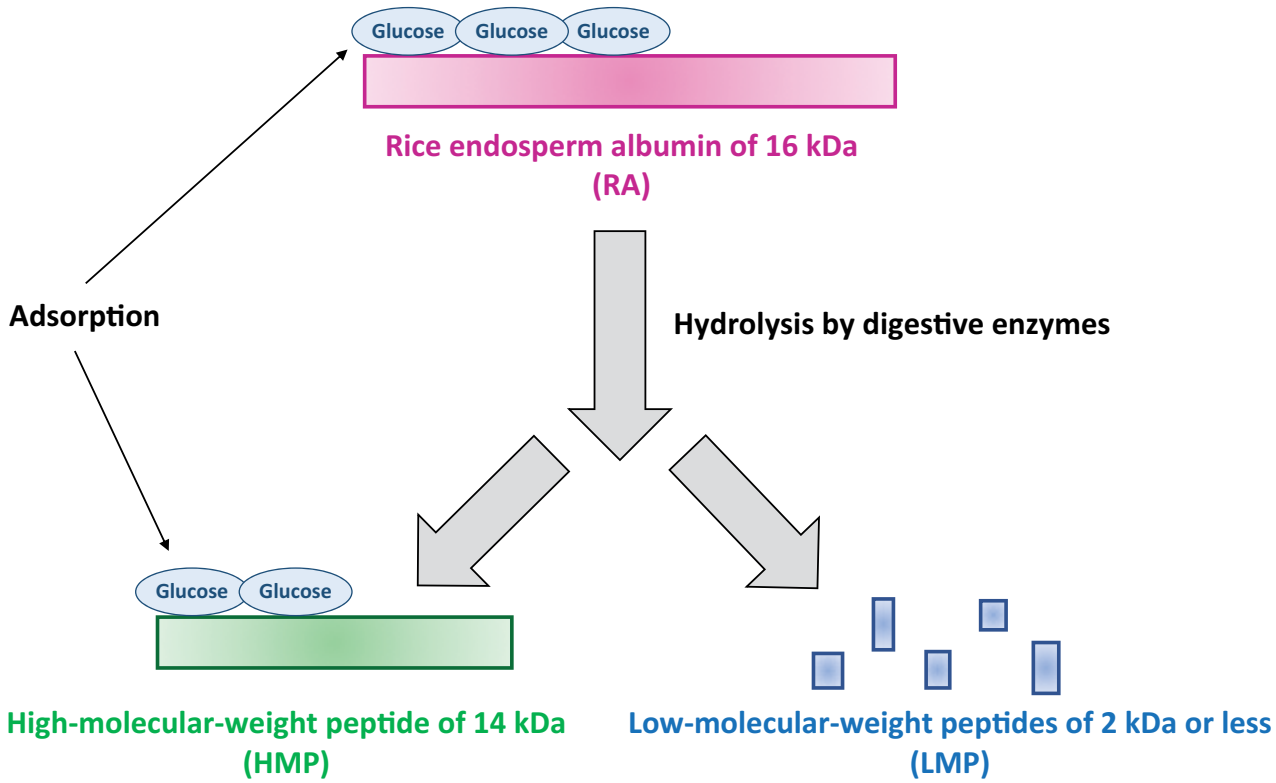

FIG. 3. Hydrolysis of RA by digestive enzymes

suppresses postprandial blood glucose elevation in rats (Ina, *et al*., 2016). RA is a complex of proteins, the major protein being one with a molecular mass of 16 kDa. Although proteins are commonly hydrolyzed by digestive enzymes into peptides and amino acids of 1 kDa or less, the 16 kDa protein in RA is hydrolyzed to a high-molecular-weight peptide of 14 kDa (HMP) and low-molecular-weight peptides (LMP) by digestive enzymes (Fig. 3). This indicates that HMP has a high resistance to digestion by digestive enzymes. As HMP is a large molecule, it adsorbs glucose on its molecule and suppresses postprandial blood glucose elevation in rats (Ina, *et al*., 2020).

If rice bran also contains albumin with glucose-adsorptive ability like RA, it could potentially be used as a functional-food material to control blood glucose concentration, which would increase the value of the rice bran. To find out, we conducted a study that evaluated the glucose adsorbability and digestibility of albumin in rice bran.

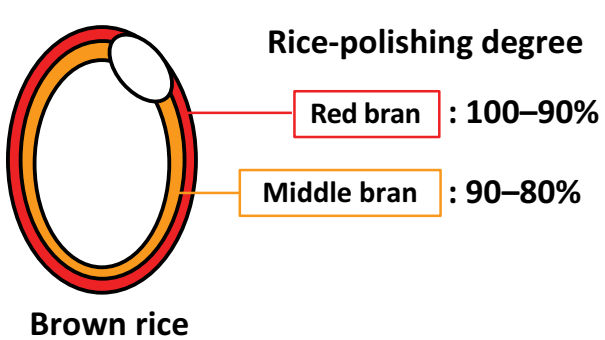

FIG. 4. Types of rice bran

Two types of rice bran were used. Bran obtained by milling brown rice to yield 90% was designated as red bran, while one obtained by milling another 10% to yield 80% was designated as middle bran (Fig. 4). Water-soluble fractions were extracted from red and middle bran flour, and albumin was obtained by ammonium sulfate precipitation.

GLUCOSE ADSORBABILITY OF RED AND MIDDLE BRAN ALBUMINS

The amount of glucose adsorbed on rice bran albumin was determined *in vitro* by measuring the diffusion rate of glucose in the solution of rice bran albumin placed in the upper chamber to the lower chamber (Fig. 5). Low-molecular-weight compounds like glucose can pass through the semipermeable membrane and move to the lower chamber, while high-molecular-weight compounds like albumin cannot pass through the semipermeable membrane. The more the amount of glucose the rice bran albumin adsorbs in the upper chamber, the slower the diffusion rate of glucose through the semipermeable membrane becomes. The diffusion rate of glucose in the presence of rice bran albumin was compared with that of RA, CMC, and guar gum, which are known to adsorb glucose. Glucose diffusion rate was suppressed in the presence of albumin extracted from red and middle bran. The glucose-diffusion rate was suppressed by rice bran albumin, indicating that the protein molecules adsorb glucose. The glucose-diffusion rate in the presence of rice bran albumin was higher than that of guar gum, but lower than that of RA and CMC. From this, it was suggested that red and middle bran albumins have glucose-adsorption ability similar to RA and dietary fibers, such as guar gum and CMC.

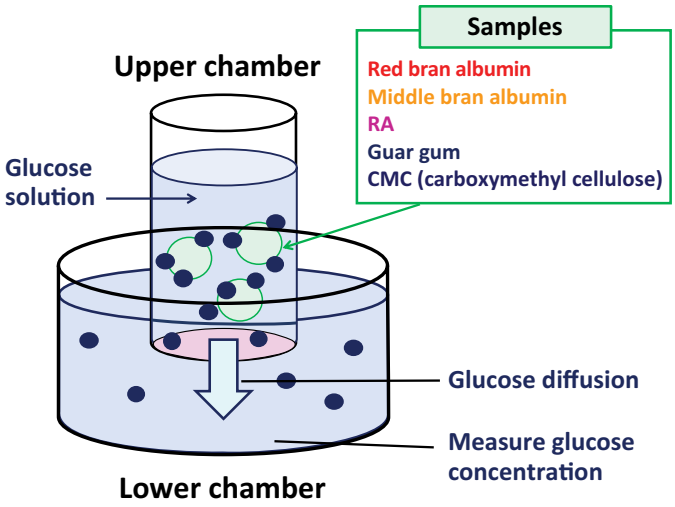

FIG. 5. Evaluation of glucose absorbability onto red-bran and middle-bran albumin

DIGESTIBILITY OF RICE BRAN ALBUMINS

To evaluate the digestive resistance of red and middle bran albumins, albumins were treated by representative digestive enzymes, pepsin and pancreatin. Red and middle bran albumins were treated with pepsin for 2 hours, followed by pancreatin for 6 hours. The digestibility was analyzed by SDS-PAGE (an electrophoresis method that allows protein separation by mass), and the band pattern of red and middle bran albumins was compared with that of RA. Before digestion, a 16-kDa protein existed in red and middle bran albumins, and a 14-kDa peptide remained after digestion. The band of 14 kDa peptide in middle bran albumin after digestion was thicker than that in red bran albumin, indicating that middle bran albumin produces indigestible peptide of 14 kDa more than red bran albumin after enzymatic digestion. Therefore, middle bran albumin is expected to adsorb glucose more than red bran albumin *in vivo* and suppress glucose absorption from the small intestine like RA. Middle bran albumin has potential to be used as a functional-food material to prevent postprandial hyperglycemia.

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Can AI learn to detect adulterated oils?

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

Rebecca Guenard

This summer, researchers at University of California, Davis, USA, released an analysis of avocado oils for sale in US grocery stores. They found that over 80% were adulterated, with three of the samples being almost entirely soybean oil (see article on page 12). Similarly, in the olive oil industry, fraudulent producers try to pass off blends as extra virgin or claim they are organic when they are not. In fact, the European Union's directorate-general for Health and Food Safety stated, in a report from 2019, that the fats and oils sector is one of its most active fraud-fighting areas (https://ec.europa.eu/food/safety/food-fraud/aas_en).

Rampant fraud in the edible oils industry strains the livelihood of genuine producers. Fraudulent producers sell their adulterated oils at such a low price that honest producers cannot compete. According to the EU, the economic gain by nefarious producers is 30% of the price difference between a blend and an extra virgin oil. To combat the problem, the EU has implemented the Administrative Assistance and Cooperation System to efficiently share information and react quickly when there is a noncompliance identified in the food chain (https://www.oliveoiltimes.com/). Researchers and oil producers argue that the US system needs to be improved (https://www.oliveoiltimes.com/). Artificial intelligence (AI) and deep learning continue to gain traction as a possible solution.

Analytical techniques like Near-Infrared and Raman spectroscopies have been used to determine food purity for almost a decade. These fast, non-destructive techniques have the added convenience of online installation capabilities in an oil processing plant. However, vibrational spectroscopy distinguishes compounds by their functional groups, and detecting the minute differences in spectra from fatty acids with similar compositions requires expert analysis. Determining the degree of saturation between fatty acids requires evaluating peak ratios in absorption spectra that are often indistinguishable.

Wilmar International, headquartered in Singapore, is Asia's leading agribusiness group. Their business focus includes palm oil cultivation, oilseed crushing, and edible oils refining, among other interests. Untzizu Elejalde, leading the

analytical technology team, says that the company is innovating and growing its R&D to have the resources to make detecting adulteration through artificial intelligence a reality. Her group is teaching computers to recognize changes in IR spectra that can be used to identify adulterated compounds.

"Instead of using partial least squares to analyze the data, we are using deep learning and convolutional neural networks," says Elejalde.

Human vision inspired the development of convolutional neural networks as a means of teaching a computer to read images. Like in the visual cortex, an image is broken up into regions of space and weighted similarly to the way neurons operate. This deep learning approach has been applied to tumor detection and face recognition.

In 2018, Elejalde and her colleagues started gathering infrared spectra on various edible oil mixtures. They found that their algorithm could differentiate between types of oil in an unknown adulterated mixture. These initial findings motivated them to fine tune the algorithm to account for all the variability that might occur in the analysis. They considered how spectroscopic signals are processed and all the ways oils could be adulterated. Then they trained the model to recognize the spectroscopic peaks that arise from known oil mixtures.

In a typical commercial scenario, an expensive oil is adulterated with inexpensive oils and labeled as pure. The Wilmar group chose to build their model based on pure peanut oil. They adulterated the pure oil in 5% increments up to a 50% impurity and used deep learning to train the computer algorithm on the mixtures' spectra. After that, they included different brands of peanut oils that they obtained from their refineries throughout Asia.

"We want to build a system that is robust and generalizable," says Kevin Lim, from the data analytics team, "In case someone tries to adulterate an oil with something that is not considered by our model." The designed algorithm should work for any tertiary system blend, for example if peanut oil can be mixed with sunflower oil and soybean oil, could the algorithm also detect if the oil was adulterated with corn oil instead?

Lim says that deep learning pattern recognition has advanced in recent years because of the way programmers are building neural networks. "AI now has the capability to extract features in a pattern," he says. However, spectra must be handled the right way for AI to pick out the changes that are due to oil mixture composition. For example, Raman spectra have inherent baseline shifts that must be adjusted before features can be automatically extracted by the deep learning machines. Ideally, you want to incorporate data from multiple spectroscopic techniques, which requires that all the spectra be normalized. With large enough labeled examples taken into account, AI can conduct pattern recognition successfully on large sets of spectral data.

"This method is a more precise way to quantify how much corn oil is in camellia seed oil, or sunflower oil in peanut oil, or any other combination of edible oils," says Elejalde. "The experimental results show that the error of prediction value can be as low as 0.2, which outperforms any other prediction methods reported to date."

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"For practical use, this is a work in progress," says Lim. Wilmar has not yet implemented the technology in their factories, though, he says it works as a proof of concept. However, natural products like edible oils present some challenges. Depending on how the oil is refined, its purity varies. The same type of oil can turn out cloudy or clear, yellow or colorless. The spectra will distinguish between these differences, but for the purpose of AI, the model needs to be universal. "Spectroscopic methods are sensitive to some differences in the oils that should be treated as agnostic in the model," says Lim. There are additional variances in product that the model needs to learn before it can be used in a factory setting, he says.

"Besides adulteration, we are also working on other food safety problems," says Elejalde. Another challenge, she says, is that gathering the spectral data needed for deep learning requires considerable manpower and time. It take up to a couple of years before we will be able to use this technology to detect adulterated oils." As they improve on their AI, Wilmar is also considering how best to use their analytical capabilities. "We are looking into developing portable systems for NIR and Raman so they can be used in the field when we are buying an edible oil from a third party," says Elejalde.

For anyone considering using this technology, Lim cautions that AI has turned into a catch-all phrase with many different meanings. Researchers must understand all the components involved in their analysis and be careful of software vendors promising more from the technology than is available. In time, AI has the capability to be an asset to the edible oils industry, but it requires an in-depth understanding of spectroscopy, computer science, and edible oil refining. Without the right team that has experts in each of these areas, resources may be wasted.

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

Fractionated coconut oil and MCT oil production: facts and fiction

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

Saeed M. Ghazani and Alejandro G. Marangoni

We recently discovered that medium-chain triglyceride (MCT) oils with fatty acid compositions that cannot possibly be derived from native coconut oil are being sold as "natural," "organic," or as "organic fractionated coconut oil". While there is nothing wrong with chemically synthesized MCT oil, should manufacturers be allowed to mislead consumers with these deceptive claims? Maybe it's time for some regulation.

Medium-chain triglyceride (MCT) oils have been used for many decades as readily digestible, calorie-dense food ingredients. They provide a quick source of energy for infants and athletes, and for persons with impaired fat digestion. MCT oils are broken down readily by salivary lipase and gastric juices, so pancreatic lipase breakdown is barely required before absorption. Medium-chain fatty acids (MCFAs), including caproic acid (C6:0), caprylic acid (C8:0), and capric acid (C10:0), are small molecules that are preferentially transported through the portal vein to the liver, where they are metabolized as fast as glucose. In recent years, the trend of MCT oil consumption as therapeutic diets, enteral nutrients, and transfusion solutions has dramatically increased. New studies show that MCFAs reduce fasting lipid levels more than unsaturated fatty acids do, and their potential to reduce body weight has been confirmed. MCTs are being used for patients with impaired digestion and patients with fluid restriction, such as acquired immune deficiency syndrome (AIDS), cystic fibrosis, cancer, multiple traumas, burn injury, respiratory distress, and hepatic or renal disease [1]. The main fatty acid composition of commercial MCT oils includes caproic acid (1 to 2%), caprylic acid (35 to 75%), capric acid (25 to 50%), and lauric acid (1 to 2%).

MANUFACTURING MCT OIL

Coconut oil from the coconut palm (*Cocos nucifera L.*) grows within 20° north and south of the equator in the tropical region of Asia (Philippines, Indonesia, and India), Africa (Ghana and Nigeria), and South America (Brazil and Mexico). The chemical composition of fresh coconut kernel includes moisture (50%), oil (34%), ash (2.2%), fiber, (3%), protein (3.5%), and carbohydrate (7.3%) [2].

The primary fatty acids in coconut oil triacylglycerol are either MCFAs (13–17%) or long-chain saturated fatty acids (62.7–69.5%) (Table 1).

TABLE 1. Fatty acid composition of coconut oil

Fatty acid	Amount (Area %)
C6:0	0.5
C8:0	7.4
C10:0	6.3
C12:0	51.1
C14:0	19.3
C16:0	8.0
C18:0	2.7
C18:1	4.6
C18:2	0.6

TABLE 2. Triacylglycerol composition of coconut oil

Triacylglycerol	Amount (Area %)
CpCL	3.5±0.2
CCL	14.0±0.3
CLL	18.8±0.2
LLL	22.0±0.5
LLO	1.2±0.0
LLM	16.9±0.2
LMM	10.0±0.3
LMO	2.6±0.0
LMP	10.3±0.3
other	6.1

Abbreviations: Cp: caprylic; C: capric; L: lauric; M: myristic; P: palmitic; and O: oleic acid

Since lauric acid is the predominant fatty acid in coconut oil (over 50% of total fatty acids), most major triacylglycerols contain lauric acid in their molecular structure, such as LLL, LLM, LMM, CLL, and LMP. Here, "L", "M", "C", and P represent lauric acid, myristic acid, capric acid, and palmitic acid, respectively (Table 2).

MCT OIL PRODUCTION THROUGH DIRECT COCONUT OIL FRACTIONATION, AN UNTRUE DEFINITION

European edible oil companies developed the first large-scale coconut oil fractionation "process" by importing oil from Sri Lanka to Europe in long wooden barrels called "Ceylon Pipes." The barrels were filled with warm coconut oil, which was cooled slowly during transport to European ports. This slow cooling of coconut oil, combined with the gentle agitation from the ships' movement, allowed the coconut oil high-melting TAGs to crystallize out and separate into fractions [3]. In a trial study in our lab, acetone fractionation of coconut oil (acetone: coconut oil 4:1 w/w) was conducted at 10°C for 48 hours. The fatty acid composition of solid- and liquid-fractions after solvent fractionation is shown in Table 3.

Based on these results, although the total amount of MCTs in the liquid fraction was much higher than solid fraction (16.8% and 7.4%, respectively), lauric acid was the primary fatty acid in both fractions (50.8% and 47.4%, respectively). In previous studies, Marikkar, *et al.*, showed that in 97% of solvent–fractionated coconut oil triacylglycerols, lauric acid was always one of the three fatty acids present (Table 4). They reported that no natural triacylglycerol molecule with three medium-chain fatty acids was detected [4].

To study the fatty acid and triacylglycerol composition of commercial MCT oils in the Canadian Market, three samples were selected and analyzed in our lab. The fatty acid and triacylglycerol composition of these commercial MCT oils samples is shown in Table 5. Based on the triacylglycerol profile of sam-

TABLE 3. Fatty acid composition of coconut oil solid and liquid fractions (Area %)

Fatty acid	Liquid fraction	Solid fraction
C6:0	0.7	0.1
C8:0	9.2	3.1
C10:0	6.9	4.2
C12:0	50.8	47.4
C14:0	16.7	27.3
C16:0	7.3	11.3
C18:0	2.6	4.0
C18:1	5.0	2.1
C18:2	0.7	0.3

TABLE 4. Triacylglycerol composition (%) of coconut oil and its fractions (Reference 3)

Triacylglycerol	Coconut oil	High-melting fraction	Low-melting fraction
CCL	15.1	5.0	15.4
CLL	19.5	7.6	19.6
LLL	22.6	14.4	22.7
LLM	16.5	22.3	16.0
LOL	2.8	1.2	3.7
LMM	9.7	21.9	8.6
LLP	0.3	0.2	0.7
LOM	2.3	1.1	2.9
LMP	5.1	15.0	4.2
LOO	0.5	0.1	0.7
LOP	1.4	0.6	1.7

TABLE 5. Fatty acid and triacylglycerol composition (Area %) of commercial MCT oils

Fatty acid	MCT oil A	MCT oil B	MCT oil C
C8:0	56.4	54.5	65.7
C10:0	43.6	45.5	34.3
Triacylglycerol	**MCT oil A**	**MCT oil B**	**MCT oil C**
C8:C8:C8	24.4	25.8	39.4
C8:C8:C10	42.2	42.0	41.1
C8:C10:C10	25.0	23.8	15.0
C10:C10:C10	5.2	4.9	2.0
Other	3.2	3.5	2.5

ples, no lauric acid was detected in the triacylglycerol structure of these MCT oils (Table 5). So, the question arises, how can we have a "fractionated coconut oil" that only contains capric/caproic/caprylic acids? How is this possible since purely C-6 to C-10 containing TAGs do not exist in native coconut oil? How can such product exist or be sold as "organic fractionated coconut oil"? How can these be "natural" or obtained by fractionation? Does the public know these MCT oils are probably synthetic?

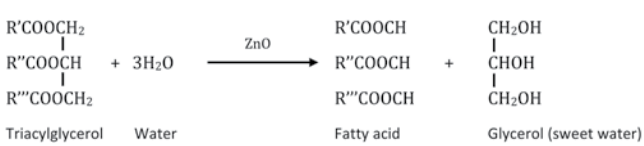
This issue will be further developed below. We will start by reviewing the common methods to synthesize MCT oils in industry.

THE CLASSIC SYNTHETIC METHODS OF MCT OIL PRODUCTION

The earliest methods of producing MCT oils in the literature are reported in the patent literature. However, the two main methods of producing MCT oils are coconut oil splitting or methylation processes. For example, the coconut oil splitting process to obtain MCT oil is discussed in detail in the following four steps.

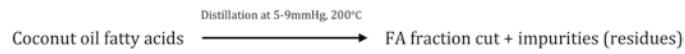
1. Coconut oil splitting

In a batch system, steam is continuously blown at the bottom of the splitting reactor to raise the pressure to above 1000 kPa, and 2-4% catalyst (ZnO) is usually added to accelerate the splitting process.



2. The fatty acid distillation process

During the distillation step, low- and high-molecular-weight impurities (light odor components and residues, respectively) are separated from fatty acids based on their differences in boiling point and vapor pressure.



3. Thermal fractionation

In the thermal fractionation step, after the de-aeration step, fatty acids are heated up to their evaporation temperature under high vacuum. The evaporated fatty acids are separated in packed columns in three cuts as, the first cut (MCFAs) with a yield of 10–15%, the second cut (lauric and myristic acids) that is the main fraction (50–70%), and the third fraction (the rest of coconut fatty acids) with a yield of 15–20%.

4. Glycerolysis of MCFAs

The esterification reaction between separated MCFAs and glycerol is the last step for producing MCT oil. Chemical glycerolysis is a reversible reaction conducted under vacuum above 200°C, and water as a by-product needs to be removed continuously from the medium to obtain a high MCT oil yield.

It is not clear how companies synthesize MCT oils. Stepan uses glycerol from vegetable oil sources and MCTs from coconut and palm kernel oils to make the caprylic/capric triglyceride, NEOBEE®. The company is upfront about the product and does not market it as "natural", "virgin", or misrepresent it as coconut oil. Nisshin Oillio Group Ltd., a large producer of edible oils and fats for processed foods and the winner of

the 2019 AOCS Corporate Achievement Award, has a fine chemicals business that produces a whole line of MCT products. They are also transparent about their products and make no claims that they are from fractionated natural oils. Their MCT oil was recently used in a recent double-blind, randomized, placebo-controlled crossover study investigating the effects of MCT on cognitive ability in patients with mild to moderate Alzheimer's Disease which found positive effects on cognitive ability in mild to moderate patients with *APOE4* $^{-/-}$ (https://www.clinicalnutritionjournal.com/article/S0261-5614(19)33104-8/pdf).

ALTERNATIVE APPROACHES TO PRODUCE MCT OIL

Recently, a metabolic engineering method was developed to produce MCT oil from the oilseed crop *Camelina sativa* [5]. Some biotech companies have developed innovative tactics for producing microbial MCT oils. For instance, a high MCT microalgal oil was produced by Solazyme Inc. in South San Francisco, USA. The amount of caprylic acid and capric acid in this algal MCT oil was 24.9% and 55.8%, respectively, while the rest of fatty acids were lauric acid (1.4%), myristic acid (1.2%), palmitic acid (3.1%), and stearic acid (13.5%). Enzyme technology is an alternative approach to produce MCT oils. The use of lipase to catalyze the hydrolysis and esterification reactions has been studied as an efficient method to produce MCT oils [6].

MORE TRANSPARENCY NEEDED

MCT oils have unique physicochemical and nutritional properties that enhance health and nutrition and are useful in many non-edible applications such as lubricants and cosmetics. With a limited supply of coconut oil, synthetic MCT oils can help meet the growing demand for these healthful oils. Meanwhile, alternative technologies like metabolic engineering and enzyme technology may ultimately turn out to be more sustainable than making these products from coconuts. However, we believe consumers should know that most of these MCT oils are synthetic products, and not just "fractionated" from some natural oils. Claiming that a chemically synthesized MCT oil is "virgin" or "organic", or even "fractionated coconut oil" is not correct or ethical and misleads and possibly defrauds customers.

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*Alejandro G. Marangoni is a Professor and Tier I Canada Research Chair in Food, Health and Aging at the University of Guelph, Ontario, Canada. His work concentrates on the physical properties of soft materials in foods, cosmetics and biolubricants. Marangoni is an AOCS Fellow, a Fellow of the **IFT**, the Royal Society of Chemistry (U.K.) and Editor-in-Chief of Current Opinion and Current Research in Food Science. Marangoni was elected Fellow of the Royal Society of Canada, the Academy of Sciences, in 2018. He can be contacted at amarango@uoguelph.ca.*

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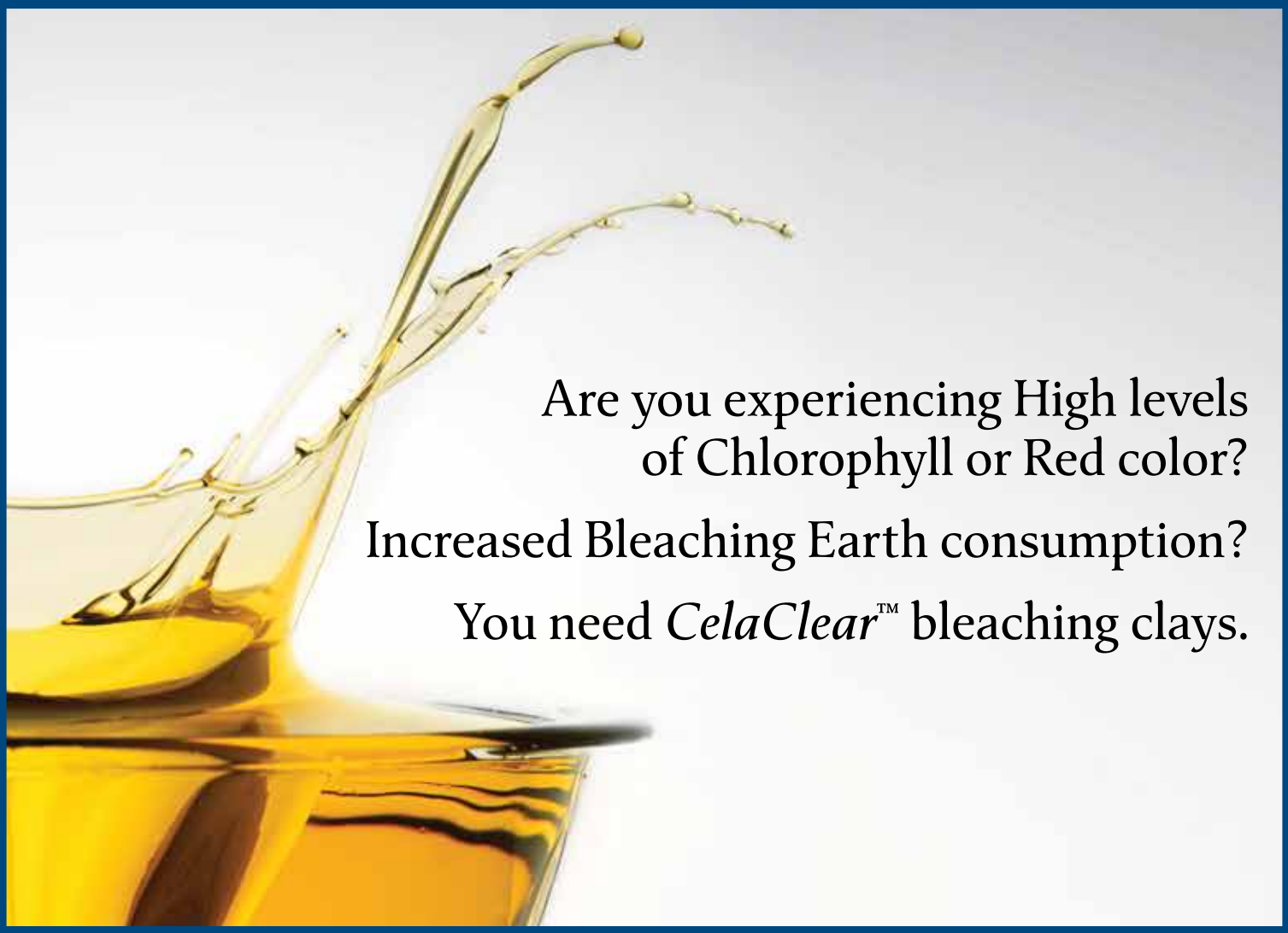

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Direct seeding for sustainable agriculture

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

Leslie Kleiner

I recently read an article in the Argentinean newspaper *Clarín* that described "direct seeding," as a 30-year-old practice used in Argentina to promote sustainable agriculture. Direct seeding is now implemented in various countries [1]. The article by Esteban Fuentes has been modified to fit this column's Q&A format.

Q: What is Aapresid, and what is "direct seeding"?

Aapresid is the acronym for "Asociación Argentina de Productores en Siembra Directa" (Argentinean Association of Producers in Direct Seeding), a non-profit, non-governmental organization with agribusiness members interested in soil conservation. Aapresid is also interested in direct seeding as a type of agriculture that enables a balance between sustainability and production for various market segments, such as food and bioenergy. Direct seeding is the practice of seeding the land directly into the soil, commonly keeping residues from the previous crop, and with minimum or no disturbance of the soil. This practice provides reduced farming costs, while improving physical, chemical, and biological conditions of the soil, ultimately leading to more efficiency in water use and increased productivity [2].

Q: What were some of the highlights regarding direct seeding during the Aapresid online congress held in August?

Agronomists from Europe and LATAM discussed the impact that direct seeding had on specific regions and crops. For example, Manuel Otero, General Director of the Inter-American Institute for Cooperation on Agriculture, explained that due to the toll of the COVID19 pandemic, agriculture has been identified as a strategic sector to reactivate the economy. Agricultural practices like direct seeding play a critical role in combating food insecurity [augmented by the COVID19 pandemic], as there is a need to increase production while minimizing the environmental toll [2]. The use of direct seeding in summer crops in Spain (Aragón region) is an example. In this region, there is a shortage of rain during the summer months, and direct seeding helps bring green areas back. Aragón has two different regions, with rain affecting the sub regions in different ways. The overall objective in these regions is to protect the crops from climate, improve soil conditions by covering it, recirculate soil nutrients, and recover land affected by salinity. [2]

Q: How do different crops adapt to direct seeding?

Marcos Guigou, a producer in Uruguay that has practiced this form of agriculture for the past 30 years, explained that the first step is to recognize which crops are most suitable for adaptation. Wheat and barley adapt well, but he did not have success with sunflower. Changing from one system to the other is complex, and sometimes planting and fertilizing must be integrated. For example, parts of a field could be devoted to growing plants that will provide nitrogen to future crops, and the rest used for more intensive agriculture (five crops within three years) with lower productivity. Overall, this practice has reduced costs associated with farming, while allowing to better treatment of the soils.

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Latin America Update is produced by Leslie Kleiner, a senior research scientist and contributing editor of *Inform*.

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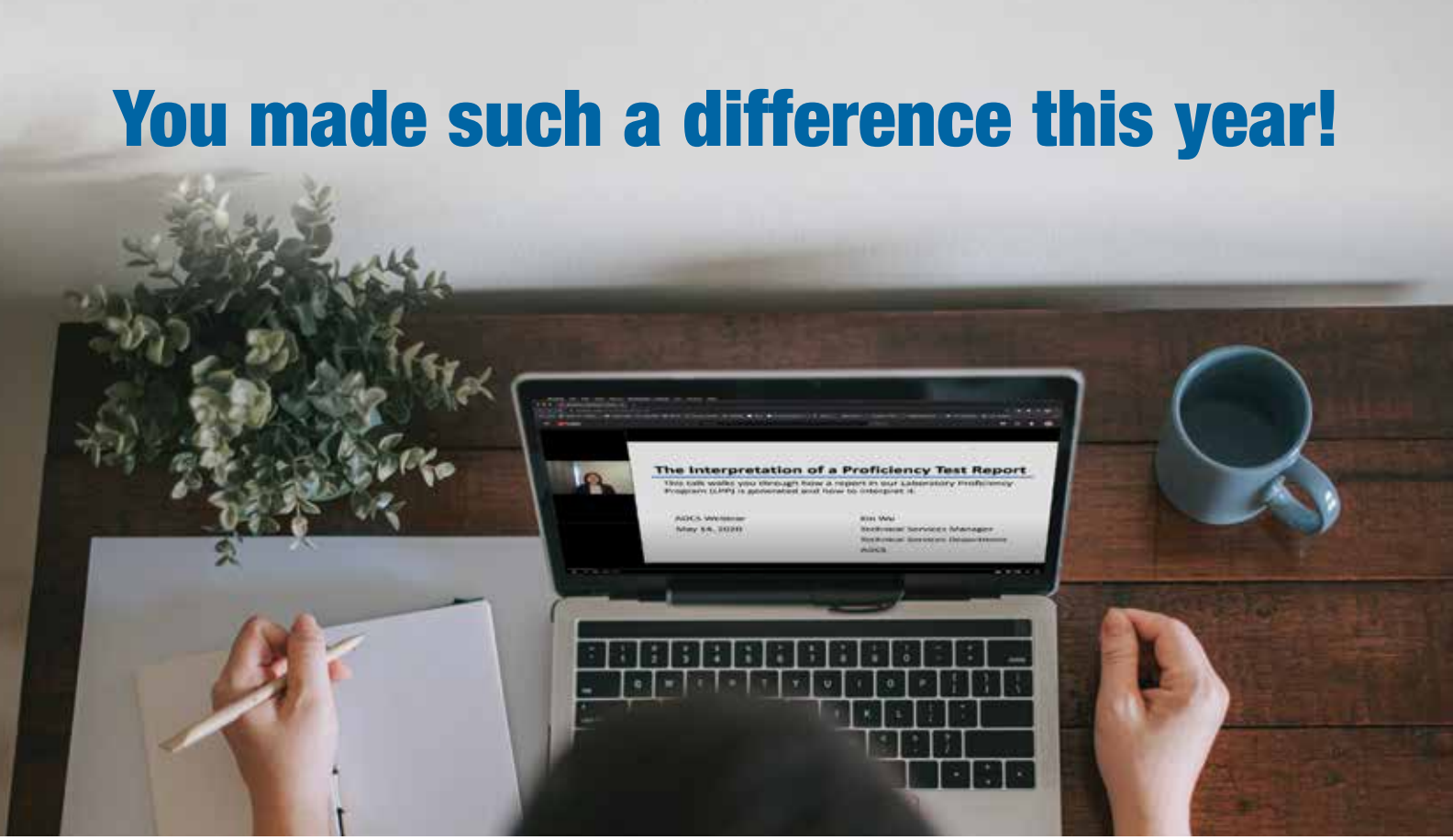

We want to thank our incredible members for all you have done in 2020. While it was not the year any of us imagined, the AOCS community has accomplished so much over the last several months.

From participating in the first-ever online AOCS Annual Meeting & Expo to accessing the latest insights through our exclusive webinar library, you have all continued your professional development during the pandemic. The work you do is an essential contribution to global advancements in science and technology during these changing times.

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Meet Elaine Susan Krul

Member Spotlight is a regular column that features members who play critical roles in AOCS.

PROFESSIONAL

What's a typical day like for you as a consultant?
If I am working on a project under contract, I break it down into parts and set goals each day for completing different sections. I also have several pet scientific research areas I keep up with, with plans on writing articles on topics that have been unaddressed.

My favorite part of my job is…
With the ongoing pandemic, I feel blessed to be able to continue working from home. I enjoy being able to set my own schedule, keep up with and continue to contribute to the areas of science that interest me most, and have fewer but more productive meetings that help move my projects forward.

Flash back to when you were 10 years old. What did you want to be when you grew up?
My father was a civil engineer and loved science. He always was trying to figure out how things worked in nature and shared that excitement with me. From an early age, I knew I wanted to be a scientist.

Why did you decide to do the work you are doing now?
I chose biochemistry as a major in college because I felt it would provide a solid base for any field. While pursuing my career in academia (focusing on the genetics of lipid metabolism) and then in the pharmaceutical industry, I observed that diets significantly impacted outcomes and were often ignored in preclinical and clinical study design. So when I had the chance to pursue nutrition research in the food industry, I felt I had found my research home.

Is there an achievement or contribution that you are most proud of? Why?
I authored a review published in *JAOCS* in 2019 on nitrogen-to-protein conversion factors that summarized the research and the current controversies surrounding how to calculate those factors. It was among the top 10% most-downloaded papers in *JAOCS* in 2018–2019.

Fast facts

Name	Elaine Susan Krul
Joined AOCS	2018
Education	Ph.D. in biochemistry from McGill University (Montreal, Canada)
Job title	Consultant and president
Employer	EKSci, LLC
Current AOCS involvement	Member of the Protein & Co-Products Division; ad hoc search committee member for an AOCS journal; published review in JAOCS on nitrogen-to-protein conversion factors; was requested by AOCS to submit documents and to represent AOCS at Codex Alimentarius on this subject

What event, person, or life experience has had the most influence on the direction of your life?
My dad with his hands-on science activities; my best childhood friend who got me interested in plant-based nutrition, holistic medicine, and meditation (way before it was trendy); and my late husband, who always encouraged and supported me in all my career endeavors.

PERSONAL

How do you relax after a hard day of work?
Kettlebell and yoga—by Zoom, now; playing piano; volunteering at a cancer-patient residence; meditation; and cooking recipes I've modified to be more nutritious or allergen-free.

What is the most impressive thing you know how to do?
I like to make greeting cards for family and friends with personalized poems. I used to make them all by hand but now use software.

What skill would you like to master?
I would like to continue to improve my piano playing skills and learn improvisation.

What are some small things that make your day better?
My miniature Schnauzer and my two adult children.

PATENTS

Method for producing a protein hydrolysate

Lynglev, G.B., *et al.*, Novozymes A/S, US10619177, April 14, 2020

The present invention relates to a method of producing a protein hydrolysate comprising a step of enzymatic protein hydrolysis performed at high temperature.

Animal-based hydrocarbon firearm lubricant

Scott, B.R., Brave Response Shooting, LLC, US10626343, April 21, 2020

A lubricant for firearms includes a base and nanoparticles dispersed throughout the base. The base may include a hydrocarbon or a mixture of hydrocarbons. The base may be in a liquid form or in a semisolid form. Fat from an animal source, such as porcine fat or, more specifically, bacon fat, may be employed as a hydrocarbon of the base. Fat from an animal source may be rendered or otherwise clarified. The nanoparticles may include nanospheres, which may have an average diameter of about 100 nm or less. The lubricant may also include a hydrocarbon from a vegetable source (e.g., a vegetable oil, etc.), a hydrocarbon from a petrochemical source, and/or a synthetic petrochemical lubricant. The lubricant may include a fragrance to impart it with a desired scent (e.g., a bacon scent, etc.). Methods for lubricating and cleaning metallic surfaces of firearms are also disclosed. In such a method, nanoparticles from a lubricant may be introduced into and retained within microscopic crevices in the metallic surfaces.

Compositions and methods for producing elevated and sustained ketosis

D'Agostino, D.P., *et al.*, University of South Florida, US10646462, May 12, 2020

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

Vegetable oil extraction improvement

Kellens, M., *et al.*, Desmet Ballestra Engineering n.v./s.a., US10647077, May 12, 2020

The present invention relates to an apparatus and a process for subjecting oleaginous vegetable material to a continuous prepressing for extracting at least part of the oil contained in said oleaginous vegetable material and producing a cake comprising an at least partially de-oiled oleaginous vegetable material. The apparatus comprises a mechanical prepress for mechanically compressing said oleaginous vegetable material, wherein the prepress is provided with a cake discharge for discharging the cake to a cooler. The cooler comprising means for subjecting the cake to a downward movement, and means for supplying a coolant gas, preferably air, in a counter-current flow to the downward movement of the cake with the purpose of cooling the cake.

Oil-in-water structured emulsion composition for use as a fat substitute

Marangoni, A., *et al.*, Coavel, Inc., US10653162, May 19, 2020

A product in the form of an oil-in-water emulsion is provided. The emulsion includes an oil phase which is an admixture of about 30–60% oil by weight, 0.01–15% wax by weight and a surfactant component, a combination of non-ionic and ionic surfactant in a ratio of at least about 10:1 to 30:1, and an aqueous phase comprising about 30–50% by weight of the emulsion. The product is useful as a fat substitute.

Catalyst and method for biodiesel production from unrefined low-grade oil and crude aqueous alcohols

Yung, K., *et al.*, he Hong Kong Polytechnic University Shenzhen Research Institute, US10654789, May 19, 2020

A catalyst for catalyzing transesterification of esters or esterification of fatty acids; the catalyst is selected from the group consisting of manganese (II) glycerolate, cobalt (II) glycerolate, iron (II) glycerolate, and any combination thereof. A method for transesterification reaction, includes: a) providing a catalyst, wherein the catalyst is selected from the group consisting of manganese (II) glycerolate, cobalt (II) glycerolate, iron (II) glycerolate, and any combination thereof; b) adding the catalyst, one or more alcohols, and a composition comprising one or more esters to a reactor to form a reaction mixture; and c) stirring while heating the reaction mixture for reaction to form transesterification products.

Method for producing a cooked food product

Salsedo, L., La Nicoise, US10660354, May 26, 2020

Method for producing a cooked food product, the method comprising the steps of: selecting a raw material, comprising chick peas, grinding the raw material to obtain a flour, hydrating the flour with water to obtain a flour mixture, preparing an oil mixture comprising at least olive oil and water, mixing the flour mixture and the oil mixture to obtain a batter, cooling and storing the batter to obtain a gel, cutting the gel to obtain individual food elements, and cooking the food elements to obtain a cooked food product, wherein the method is characterized in that, prior to the step of mixing the flour mixture and oil mixture, the oil mixture is heated, wherein the flour mixture is heated by mixing the flour mixture and the oil mixture due to transfer of heat contained in the oil mixture.

Compositions and methods comprising medium-chain triglycerides for treatment of epilepsy

Pan, Y., *et al*, Societe des Produits Nestle SA, US10668041, June 2, 2020

The invention provides compositions and methods for treatment of epilepsy in an animal. In one embodiment, a method for treating epilepsy in a companion animal can comprise administering to the companion animal a food composition comprising a medium-chain triglyceride (MCT), wherein the MCT is present in the food composition in an effective amount for reducing or preventing seizures when the food composition is administered to the companion animal.

Sterol blends as an additive in asphalt binder

Reinke, G.H., *et al.*, A.L.M. Holding Company and Ergon Asphalt & Emulsions, Inc., US10669202, June 2, 2020

Asphalt binder compositions and methods for making such compositions with pure sterol:crude sterol blends. The sterol blends improve various rheological properties.

Process for the selective hydrogenation of vegetable oils using egg-shell type catalysts

Borsotti; G., *et al.*, NOVAMONT S.P.A., US10683465, June 16, 2020

The invention relates to a process for the hydrogenation of vegetable oils that selectively converts polyunsaturated fatty acids into monounsaturated fatty acids, and to the products obtained therefrom. Vegetable oils obtained by the process according to the invention have a particularly high content of monounsaturated fatty acids and are suitable for use as raw materials for the synthesis of chemical intermediates.

Manufacture of lipid-based nanoparticles using a dual asymmetric centrifuge

Massing, U., US10662060, May 26, 2020

The invention relates to a method for producing lipid-based nanoparticles using a dual asymmetrical centrifuge, products produced by means of said method, kits for producing said nanoparticles using a dual asymmetrical centrifuge, and accessories for carrying out the inventive method.

Enzymatic transesterification/esterification processes employing lipases immobilized on hydrophobic resins in the presence of water solutions

Basheer; S., *et al.*, Trans Bio-Diesel Ltd., US10689607, June 23, 2020

Disclosed are an enzymatic batchwise or continuous process for the production of fatty acid alkyl esters for use in the biofuels, food, and detergent industries, and a system therefor. The process utilizes enzymes immobilized on a hydrophobic resin mixed with a fatty acid source and an alcohol or alcohol donor in the presence of an alkaline or mild alkaline aqueous buffer, or in the presence of water or aqueous solution. The production process for fatty acid alkyl esters is carried out by transesterification or esterification simultaneously or sequentially. The biocatalyst activity is maintained with no significant activity losses in multiple uses and also avoids the accumulation of glycerol and water by-products or other hydrophilic compounds on the biocatalyst.

Processes for producing fermentation products

Kang, Z., *et al.*, Novozymes A/S, US10689630, June 23, 2020

The present invention relates to processes for producing fermentation products from starch-containing material, wherein an alpha-amylase and a thermostable hemicellulase is present and/or added during liquefaction. The invention also relates to compositions suitable for use in processes of the invention.

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Editor's choice/ pick of the month

If you read the AOCS Journals column in the September issue, these remaining summaries of articles-of-the month picks from the Editors-in-Chief of *JAOCS*, *Lipids*, and *JSD* are all you need to catch up on work published in the three journals through July 2020. Having trouble finding your September issue? Read it online. Log in as a member and select *INFORM* magazine from the AOCS Member Benefit dropdown on the left side of your accounts page.

JAOCS ARTICLE OF THE MONTH

Fungal oil from cheese whey permeate

Full article at: https://doi.org/10.1002/aocs.12372

In "Scaling up the bioconversion of cheese whey permeate into fungal oil by *mucor circinelloides*," Juliana M. L. N. de Moura Bell and colleagues at the University of California, Davis, describe using an aerated stirred tank bioreactor to convert hydrolyzed cheese whey permeate into fungal oil. After a 120-hour fermentation, they achieved a maximum biomass yield of 10.7 g L-1 and lipid content of 32% dry biomass. "The valorization of agricultural byproducts into food and fuel ingredients on an industrial scale is key to producing valuable commodities in this sector," says Jim Kenar, *JAOCS* Editor-in-Chief.

LIPIDS ARTICLE OF THE MONTH

How moderate alcohol consumption affects blood lipids

Full article at: https://doi.org/10.1002/lipd.12237

Research shows that moderate alcohol intake modifies one's risk of cardiovascular disease. In a *Lipids* article, Henry Pownall and colleagues report their findings on the effects of moderate alcohol consumption on blood lipids in adults with varying blood triacylglycerol concentrations. They show that alcohol consumption modifies cholesterol exchange between chylomicron and HDL particles in a manner consistent with reduced risk of cardiovascular disease. However, the *Lipids* Editor-in-Chief questions if the alcohol consumption was associated with raised plasma non-esterified fatty acid. The full title of the article is, "Dietary alcohol and fat differentially affect plasma cholesteryl ester transfer activity and triglycerides in normo- and hypertriglyceridemic subjects."

JSD ARTICLE OF THE MONTH

Bio-based surfactant derived from cashew nut shells

Full article at: https://doi.org/10.1002/jsde.12384

In "Synthesis, characterization, and evaluation of interfacial properties and antibacterial activities of dicarboxylate anacardic acid derivatives from CNSL of *Anacardium occidentale* L," Sonia Koteich Khatib, Johnny Bullón, and coauthors from the Universidad de Los Andes, Venezuela, describe a simple procedure for preparing a surfactant from cashew nut shells. Anacardic acid extracted from cashew nut shell liquid (CNSL), undergoes carboxymethylation with chloroacetic acid to obtain a benzoic acid and its disodium carboxylate salt. Characterization of its fundamental surface activity properties proved the viability of the dicarboxylated surfactant which performed well against several different gram-positive and -negative bacteria. These findings indicate that cashew nut shells could be a source for natural surfactants.

EXTRACTS & DISTILLATES

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ANA Analytical
BIO Biotechnology
EAT Edible Applications
LOQ Lipid Oxidation and Quality
H&N Health and Nutrition
IOP Industrial Oil Products
PRO Processing
PCP Protein and Co-Products
S&D Surfactants and Detergents

Review Articles

H&N New insights into potential nutritional effects of dietary saponins in protecting against the development of obesity

Syam, P.K., *et al.*, *Food Chem. 318*: 126474, July 2020, https://doi.org/10.1016/j.foodchem.2020.126474.

Excessive energy intake, poor physical exercise, and genetics/epigenetics are instrumental for the development of obesity. Because of rapidly emerging evidences related to off-target effects and toxicity of anti-obesity drugs, there is a need to search for more effective and targeted drugs for treatment of obesity. Substantial studies have found the nutritional effects of dietary saponins (bio-detergents) in terms of decreasing the synthesis of lipids, suppressing adipogenesis, inhibiting intestinal absorption of lipids, and promoting fecal excretion of bile acids and triglycerides. Dietary saponin have been approved as potent pancreatic lipase inhibitors, disaccharidase enzyme inhibitors, antagonistic to *in vitro* lipogenesis and *in vivo* appetite suppressants, antioxidants, immune-regulators, prevent fatty liver formation, protects epithelial vasculature and regulate body weight. Many dietary saponins, such as sibutramine, morgoside, sessiloside, soysaponin B, and diosgenin, have treatment potential against the development of obesity. Excellent scientific achievements have been developed for a better understanding the mechanism of saponins in preventing obesity.

IOP PRO Direct conversion of glyceride-based oil into renewable jet fuels

Lin, C.-H. and W.-C. Wang, *Renew. Sustain. Energ. Rev. 132*: 110109, October 2020, https://doi.org/10.1016/j.rser.2020.110109.

Hydro-conversion of triglyceride to renewable jet fuel (HRJ) plays an important role in drop-in aviation fuels and has drawn the attention of scholars because of its potential to reduce aircraft pollution and mitigate greenhouse gas emissions. A direct one-step conversion of glyceride-based oil into HRJ over NiAg supported on SAPO-11 zeolite was investigated in this paper. Also, the properties of the catalysts were characterized using XRD, TEM, N_2 adsorption-desorption, TG, and Py-FTIR. The NiAg/SAPO-11 catalyst showed good performance in terms of hydro-processing, hydro-cracking, and hydro-isomerization reactions with the assistance of citric acid (CA) and phosphotungstic acid hydrate (HPW). The key to high conversion, high selectivity, and high iso-alkane content depended mostly on the reaction temperature, metal dispersion, acid content and the pore structures of the zeolite. Furthermore, the fuel properties were tested in a GC-MS/FID and flash point tester to ensure that they meet ASTM D7655 specifications. Under the optimal reaction conditions, a conversion of 100%, selectivity of 84%, an I-to-N ratio of 2.1, a yield of 72%, an aromatics content of 7%, and a flash point of 58°C were obtained. The mass, carbon, and energy yield for both one-step and two-step processes were also determined. This study provides a novel technique for producing renewable jet fuel with higher production yield.

IOP Life-cycle energy use and greenhouse gas emissions of palm fatty acid distillate-derived renewable diesel

Xu, H., *et al.*, *Renew. Sustain. Energ. Rev. 134*: 110144, December 2020, https://doi.org/10.1016/j.rser.2020.110144.

This study aims to quantify life-cycle fossil energy use and greenhouse gas (GHG) emissions for palm fatty acid distillate (PFAD)-derived renewable diesel (RD), taking into consideration different feedstock classifications that are applicable to PFAD (residue, byproduct, or coproduct) and incorporating updated data for key processes. Under the three classifications, the PFAD to RD pathway was modeled using the Greenhouse gases, Regulated Emissions, and Energy Use in Technologies (GREET®) model. PFAD-derived RD could reduce fossil energy consumption by 77%–88%, relative to petroleum diesel. GHG emissions are very sensitive to PFAD classification and co-product handling methods. Considering the production of palm oil and PFAD and economic value, we maintain that PFAD should be treated as a by-product in palm oil refineries. With this treatment, PFAD-derived RD could achieve 84% GHG emissions reductions, compared to the emissions of petroleum diesel. We also employed a substitution method to address the substitution of PFAD by other materials in the marketplace. Compared to co-product allocation results, we found substituting PFAD by tallow, soy oil, barley, and canola oil results in lower GHG emissions. Due to high induced land-use change emissions associated with palm farming, if

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PFAD is treated as a coproduct with refined palm oil, PFAD-derived RD may not deliver GHG reductions. A sensitivity analysis identified key parameters such as palm fruit yield, oil extraction efficiency in oil mills, and energy use intensity for RD production affects LCA results significantly; future efforts to improve these parameters could result in further GHG reductions.

Original Articles

ANA PRO H&N Development, validation and application of a LC-MS/MS method for quantification of 15 cannabinoids in food

Christinat, N., *et al.*, *Food Chem. 318*: 126469, July 2020, https://doi.org/10.1016/j.foodchem.2020.126469.

In this study, the occurrence of cannabinoids in hemp-based food products was investigated. For that purpose, a new liquid chromatography tandem mass spectrometry method for the quantification of 15 cannabinoids was developed and validated for multiple matrices. Method performances were good, fulfilling the SANTE/11813/2017 requirements, and allowing for products compliance testing with various national legislations on cannabinoids levels in food products. The limit of quantification of each analyte was 0.15 mg/kg for hemp seed and hemp protein, 0.6 mg/kg for hemp seed oil, and 0.005 mg/kg for raw milk and milk powder. The applicability of the method was further demonstrated by conducting a limited survey on twenty hemp-based food products. The survey revealed that products from the same category can have very different cannabinoids profiles and levels. These results highlighted the importance of cannabinoids testing of food products in view of the current heterogeneous and fast evolving regulatory landscape worldwide.

ANA LOQ H&N Characterization of chemical composition and prebiotic effect of a dietary medicinal plant *Penthorum chinense* Pursh

Yin, J., *et al.*, *Food Chem. 319*: 126568, July 2020, https://doi.org/10.1016/j.foodchem.2020.126568.

Penthorum chinense Pursh is a dietary medicinal plant widely distributed in Asia-Pacific countries. The present study aims to profile the chemical constituents of *P. chinense* and investigate its prebiotic role in modulating gut microbiota. Fifty polyphenolic compounds were rapidly identified using UPLC-HR-MS. Total flavonoid and phenolic contents of *P. chinense* were 46.6% and 61.3% (w/w), respectively. Thirteen individual polyphenols were quantified, which accounted for 33.1% (w/w). *P. chinense* induced structural arrangement of microbial community in mice, showing increased microbiota diversity, elevated Bacteroidetes/*Firmicutes* ratio and enriched gut health-promoting bacteria. After a one-week drug-free wash, most of these changes were recovered, but the abundance of some beneficial bacteria was further increased. The altered composition of gut microbiota enriched several metabolic pathways. Moreover, *P. chinense* increased antioxidant capacity *in vivo*. The results suggest that polyphenol-enriched *P. chinense* modulates gut microbiota and enhances antioxidant capacity in mice toward a beneficial environment for host health.

EAT LOQ Effect of roasting and *in vitro* digestion on phenolic profiles and antioxidant activity of water-soluble extracts from sesame

Chen, Y., *et al.*, *Food Chem. Toxicol. 139*: 111239, May 2020, https://doi.org/10.1016/j.fct.2020.111239.

The effects of roasting and *in vitro* digestion on total phenolic content (TPC), total flavonoid content (TFC), phenolic profiles, and antioxidant activity of water-soluble extracts from six varieties of sesame were investigated in this study. Our results showed that the major phenolic compounds in raw, roasted, and digested sesame were gallic acid (GA), protocatechuic acid (PA), 4-hydroxybenzoic acid (4 HBA), ferulic acid (FA), and quercetin (Quer). Roasting significantly increased the TPC, pinoresinol diglucoside (PD), sesamol, as well as the content of phenolic compounds (especially GA, PA, 4 HBA and Quer) in sesame, but kept or reduced the TFC, sesamin and sesamolin. After roasting, the antioxidant potency composite index (ACI) of six varieties of sesame was significantly increased by 29.8%–216.6%. Additionally, the ACI of gastric digestion was significantly higher than that of oral and intestinal digestion during the *in vitro* digestion of the roasted sesame, except for the varieties of Ganzhi 9 and Ganzhi 17. This study showed that five phenolic compounds (GA, PA, 4 HBA, p-coumaric acid, Quer) and sesamol of the water-soluble extracts contributed to the antioxidant activities of the digestive products of sesame.

EAT LOQ Study of sesame seeds' antioxidant and emulsifying properties

Abirached, C., *et al.*, J. *Supercrit. Fluid. 166*: 104994, December 2020, https://doi.org/10.1016/j.supflu.2020.104994.

Extraction with supercritical CO_2 as a solvent was evaluated to obtain functional additives from black sesame seeds, and compared to Soxhlet method. From the residues of oil extraction, protein concentrates were obtained by isoelectric precipitation. To evaluate antioxidant activity, the extracts obtained were added to purified sunflower oil. The Induction period (IP) of the oxidation process was determined at 100°C. Fatty acids composition and the content of total phenols and tocopherols were also determined. Using ethanol as a co-solvent in the supercritical extraction increased the yield, the content of total phenols, and the IP. Protein content, protein solubility as a function of pH, thermal behavior, and emulsifying properties were evaluated. No large differences were found in protein content, protein solubility, or emulsion destabilization kinetics of the concentrates, indicating that the oil extraction method did not affect the protein performance.

EAT LOQ Optimization of anthocyanidins conversion using chokeberry pomace rich in polymeric proanthocyanidins and cellular antioxidant activity analysis

Gao, N., *et al.*, *LWT 133*:109889, November 2020, https://doi.org/10.1016/j.lwt.2020.109889.

Chokeberry pomace contains abundant polymeric proanthocyanidins with low antioxidant activity and bioaccessibility. In this study, polymeric proanthocyanidins from chokeberry pomace were converted into anthocyanidins with higher antioxidant activity. Optimal conversion conditions were determined by full factorial design. The composition of anthocyanidin was analyzed by TOF-MS and UPLC. In addition, the antioxidant activity of the conversion products was evaluated by PSC and CAA assays. The results showed that the yield of anthocyanidins was 11.59 mg/g when the pomace was treated with 95% ethanol concentration, 50:1 liquid-solid ratio, 4% hydrochloric acid, and 0.2 mg/mL ammonium ferric citrate concentration at 80°C for 30 min. The newly generated anthocyanidins were identified as cyanidin, which accounted for 51.3% of the total anthocyanidins. The antioxidant activity values of PSC and CAA increased 2.81- and 4.77-fold respectively. The proposed method provides a new strategy for preparation of antioxidants from naturally abundant polymeric ones.

EAT LOQ Enhancement of phenolic antioxidants in industrial apple waste by fermentation with *Aspergillus* spp.

Gulsunoglu, Z., *et al.*, *Biocatal. Agric. Biotechnol. 25*: 101562, May 2020, https://doi.org/10.1016/j.bcab.2020.101562.

Apple peel is a waste from industrial juice processing that contains phenolic antioxidants. This study aimed to evaluate valorization of apple peel by fermentation with four different locally isolated *Aspergillus* spp. Fermentation with *Aspergillus* spp. augmented phenolic content and antioxidant activity of apple peel. *A. niger* ZDM2 and *A. tubingensis* ZDM1 were found to yield the highest level of total phenolic (1440 ± 37 and 1202 ± 88 mg GAE/100 g dm, respectively) and flavonoid contents (382 ± 47 and 495 ± 19 mg CE/100 g dm, respectively). Fermentation of apple peel with these species resulted in 3- and 5-times higher antioxidant activity measured by CUPRAC and DPPH methods compared to those of the unfermented sample, respectively. Quercetin and its glycosides were dominant in unfermented apple peel and their concentrations were reduced by mold fermentation. After fermentation, an isomer of taxifolin was produced in apple peel fermented by *A. aculeatus* ZGM6 and *A. japonicus* ZGM4, whereas isomers of eriodictyol and catechin were identified in apple peel fermented by *A. niger* ZDM2 and *A. tubingensis* ZDM1.

H&N PRO Identification of d-amino acids in tea leaves

Yu Xu, *et al.*, *Food Chem. 317*: 126428, July 2020, https://doi.org/10.1016/j.foodchem.2020.126428.

During manufacturing processes and in the storage period of tea, amino acids may undergo enantiomeric isomerization, converting their l- to d-forms. To examine the hypothesis, a method was developed for the analysis of the enantiomers in tea leaves. After enriched by ion-exchange solid-phase extraction, the enantiomeric pairs were separated by a chiral high-performance liquid chromatography (HPLC) and subsequently detected and identified by using a high-resolution quadrupole time-of-flight mass spectrometry (QTOF MS). Only l-forms of amino acids were found in fresh tea leaves. A total of 11 d-amino acids were found in 19 tea samples, ranging from trace amount to 43 μg/g. The results indicated that the enantioisomerization of amino acids occurred in post-harvest tea leaves and affected by process conditions and storage time.

H&N FRCD: A comprehensive food risk component database with molecular scaffold, chemical diversity, toxicity, and biodegradability analysis

Zhang, D., *et al.*, *Food Chem. 318*: 126470, July 2020, https://doi.org/10.1016/j.foodchem.2020.126470.

This is very helpful database for almost everyone who is interested in safety and toxicology properties of the food components The presence of natural toxins, pesticide residues, and illegal additives in food products has been associated with a range of potential health hazards. However, no systematic database exists that comprehensively includes and integrates all research information on these compounds, and valuable information remains scattered across numerous databases and extensive literature reports. Thus, using natural language processing technology, we curated 12,018 food risk components from 152,737 literature reports, 12 authoritative databases, and numerous related regulatory documents. Data on molecular structures, physicochemical properties, chemical taxonomy, absorption, distribution, metabolism, excretion, toxicity properties, and physiological targets within the human body were integrated to afford the comprehensive food risk component database (FRCD, http://www.rxnfinder.org/frcd/). We also analyzed the molecular scaffold and chemical diversity, in addition to evaluating the toxicity and biodegradability of the food risk components. The FRCD could be considered a highly promising tool for future food safety studies.

H&N Laxative metabolites from the leaves of *Moringa oleifera*

Jiang, M.-Y., *et al.*, J. *Agric. Food Chem. 68*: 7850–7860, 2020, https://doi.org/10.1021/acs.jafc.0c01564.

Three new flavonoids, quercetin-3-O-6-[methyl-(S)-3-hydroxy-3-methylglutaroyl(1→6]-β-d-glucopyranoside (1), kaempferol-3-O-[methyl-(S)-3-hydroxy-3-methylglutaroyl(1→6)]-β-d-glucopyranoside (2), and quercetin-3-O-6-[(E)-4-

methoxy-5-methylhexa-2,4-dienoatyl(1→6)]-β-d-glucopyranoside (3), and two new alkaloids, 5-dehydroxymethyl-pyrrolemarumine 4″-O-α-l-rhamnopyranoside (4) and N1-methyl-N2-((4-O-α-l-rhamnopyranoside)benzyl) oxalamide (5), together with 45 known compounds (6–50) were isolated from the leaves of *Moringa oleifera* Lam. Among those compounds, 1-octacosanol (50), a straight-chain 28-carbon alcohol, exhibited good activity against diphenoxylate-induced constipation in mice, which is obtained as a laxative constituent from the plant for the first time. To have an accurate understanding of the content of compound 50, a quantification with gas chromatography–tandem mass spectrometry (GC–MS/MS) was carried out. The anti-inflammatory and α-glucosidase inhibitory activity of some compounds also was assessed.

H&N LOQ EAT Physicochemical, radical scavenging activity, and sensory properties of a soft cheese fortified with *Arbutus unedo* L. extract

Masmoudia,, M., *Food Biosci*. 35: 100579, June 2020, https://doi.org/10.1016/j.fbio.2020.100579.

Arbutus unedo L. fruit is known for its high phenolic content, dietary fibers, and antioxidant capacity. This study evaluated the effect of adding *A. unedo* fruit extract obtained using a water decoction on the physicochemical, DPPH-scavenging activity, textural, and sensory properties of a soft "Sardaigne" cheese. Total phenolic content recovered using aqueous extraction was 23.4 mg GAE/g extract. The characterization of the extract using LC-MS showed that quinic acid and catechin were the main phenolic compounds. The scavenging activity of the fruit extract was evaluated using the DPPH radical scavenging assay. The IC50 value was ~0.31 mg/ml. The cheese, fortified with *A. unedo* extract, showed an increase in crude protein and firmness. Incorporation of the extract at 0.3 g/l increased the cheese yield, improved its DPPH scavenging activity after 5 days of storage, and did not alter its color and sensory properties. These results suggested that addition of *A. unedo* extract to soft cheese at 0.3 g/l might be a functional ingredient with potential health benefits and good properties.

IOP PRO Biodiesel production from microalgae by direct transesterification using green solvents

de Jesus, S.S., *et al.*, *Renew. Energ. 160*: 1283–1294, November 2020, https://doi.org/10.1016/j.renene.2020.07.056.

This study evaluated the production of biodiesel from the microalgae *Chlorella pyrenoidosa* by direct transesterification using 2-methyltetrahydrofuran or cyclopentyl methyl ether co-solvents as an alternative to chloroform. Acid-catalyzed transesterification using hydrochloric acid was developed for biodiesel production. Biodiesel was purified by column chromatography using one of the two green solvents as eluents. The highly ecological biodiesel quality was compared to the biodiesel produced using chloroform as co-solvent. A 33 factorial design was conducted to optimize the production of fatty acid methyl esters, being the independent variables investigated: solvent and catalyst volume, solvent and catalyst ratio (methanol:co-solvent:hydrochloric acid), and temperature, with a reaction time of 150 min. Biodiesel production was mainly influenced by temperature increase. The achieved yields using cyclopentyl methyl ether, 2-methyltetrahydrofuran, and chloroform were 71–92%, 67–91%, and 61–76%, respectively. The fatty acid methyl esters profile of biodiesel produced with 2-methyltetrahydrofuran and cyclopentyl methyl ether consisted of approximately 45% polyunsaturated components. The biodiesel obtained was within the ASTM D6751 and EN 14214 standards. The replacement of fossil solvents used in direct transesterification of biomass with a green solvent resulted in a further ecological biodiesel production process toward a circular bioeconomy.

PRO BIO H&N *De novo* high-titer production of delta-tocotrienol in recombinant *Saccharomyces cerevisiae*

Sun, H., *et al.*, *J. Agric. Food Chem. 68*, 29: 7710–7717, 2020, https://doi.org/10.1021/acs.jafc.0c00294.

Delta-tocotrienol as a vitamin E isomer has received much attention because of its diverse biomedical applications. Microbial biosynthesis of delta-tocotrienol is a promising strategy for its economic and environmental advantages. Here, we accomplished complete biosynthesis of delta-tocotrienol in *Saccharomyces cerevisiae* from glucose. We first constructed and incorporated a heterologous pathway into the genome of *S. cerevisiae* by incorporating the genes hpd (from *Pseudomonas putida* KT2440), hpt (from *Synechocystis* sp. PCC 6803), and vte1 (from *Arabidopsis thaliana*) for the biosynthesis of delta-tocotrienol. We further enhanced the biosynthesis of the precursor geranylgeranyl diphosphate by overexpressing the thmg1 and ggppssa (from *Sulfolobus acidocaldarius*) genes, leading to a production titer of delta-tocotrienol of 1.39 ± 0.01 mg/L. Finally, we optimized the fermentation medium using the response surface methodology, enabling a high-titer production of delta-tocotrienol (3.56 ± 0.25 mg/L), ~2.6-fold of that of the initial culture medium. Fed-batch fermentation in a 2 L fermenter was further used to enhance the production titer of delta-tocotrienol (4.10 ± 0.10 mg/L). To the best of our knowledge, this is the first report on the de novo biosynthesis of delta-tocotrienol in *S. cerevisiae*, and the highest titer obtained for microbial production of delta-tocotrienol.

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