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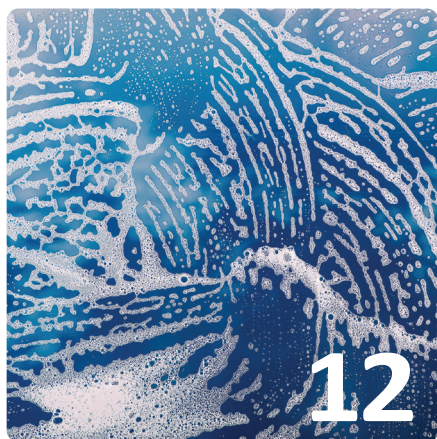


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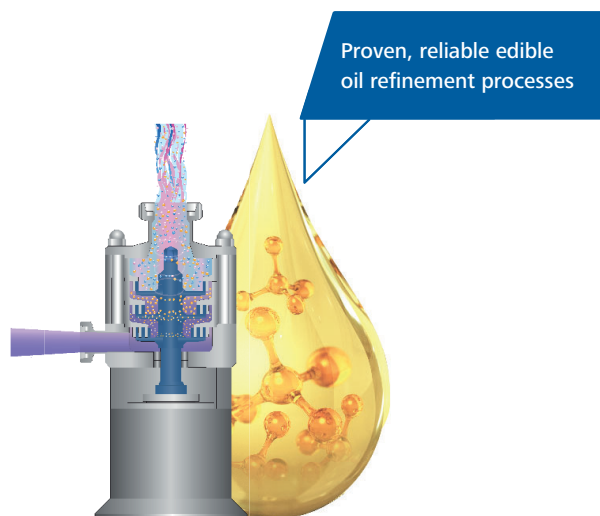
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Gut instincts

Rebecca Guenard

Colonies of bacteria flourish deep in the ocean near hydrothermal vents that expel toxic, super-heated water from the Earth's crust. Other bacteria subsist on rivers of sulfuric acid running through subterranean caves. Given the extreme environments where bacteria dwell, it is not surprising that the human digestive system is another popular home for microorganisms.

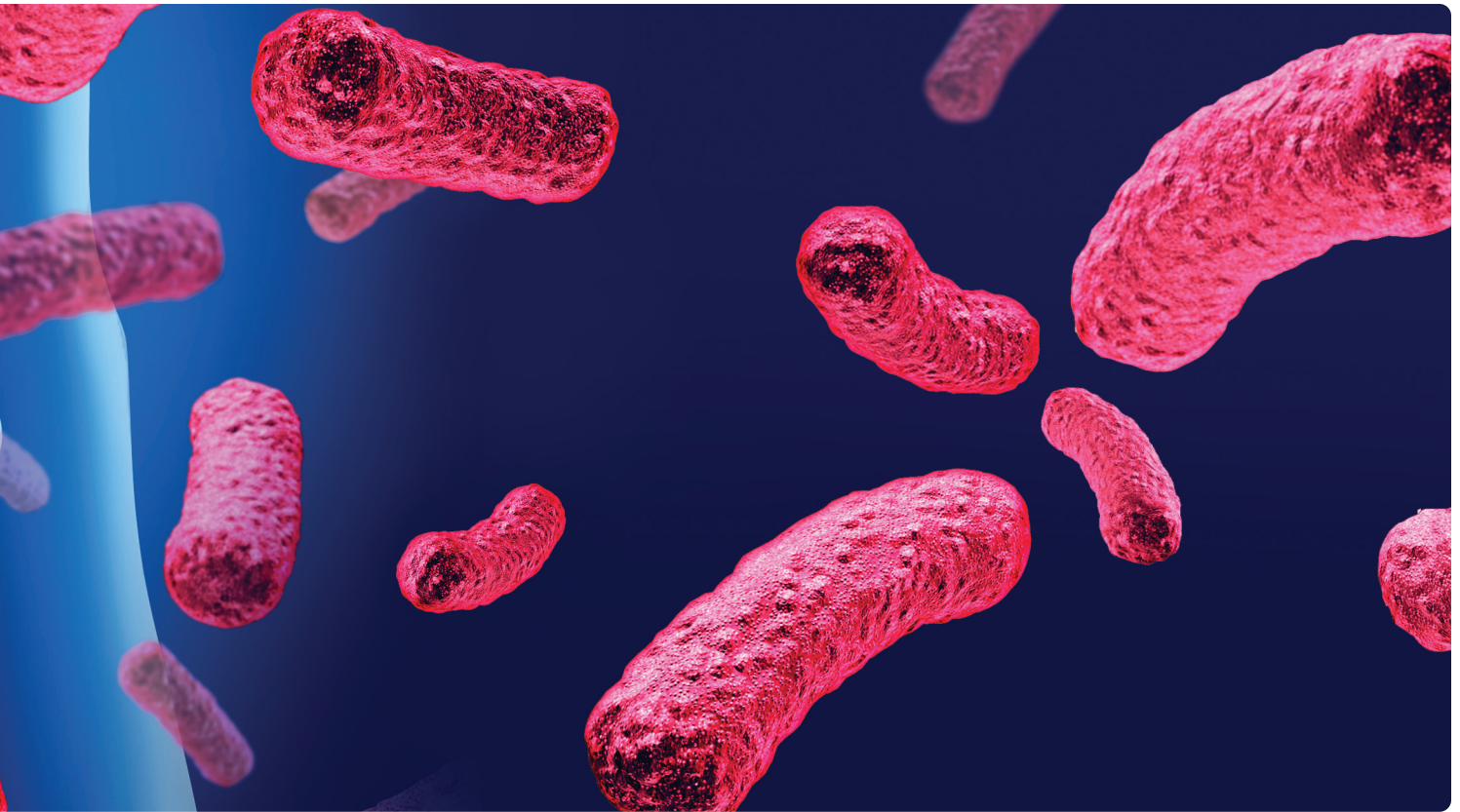
- Mucosal membranes act as a network that provides a connection between the gut and vital organs, like the lungs or the brain, but presumably the entire body.
- Short-chain fatty acids are formed in the colon through the fermentation of soluble fiber, and researchers are interested in finding out more about their role as signaling compounds.
- More research is needed to decisively say that one diet is better for the microbiome over another.

The gut microbiota is an ecosystem comprised of an estimated 1,000 bacterial species along with archaea, fungi, and viruses that reside in the human intestinal tract. For a century now, researchers have studied this community of microorganisms, but it was not until modern sequencing tools enabled them to genetically distinguish bacterial colonies 20 years ago that the mysteries of the microbiome were revealed. Since then, scientists have come to understand that this ecosystem in our gut, just like any ecosystem, is a complex environment with a delicate balance.

Furthermore, all the latest research has redefined a healthy gut to mean more than just comfortable digestion. Scientists now know that human health overall may rely on what colonies populate the intestines.

Gut bacteria have been shown to influence diseases such as colon cancer, but also neurological disorders like multiple sclerosis and even autism spectrum disorder. The microbes determine the effectiveness of drug treatments like statins, which fail to lower cholesterol sufficiently in certain gut bacteria environments. Fatty acid metabolites from bacteria are emerging as a means of immune system modulation which could be a treatment mechanism in the future. But the biggest question researchers want to answer is, how can dietary habits be adjusted to maintain the healthiest possible gut?

Here is a look at the latest information on what scientists have learned about the gut microbiome.



IMMUNE SYSTEM

There is growing evidence that the gut microbiome is a significant factor in maintaining human health and avoiding disease. However, it is becoming clear that the gut does not act alone. There are colonies of microorganisms throughout the body, and researchers are identifying communication links that allow “cross-talk” among them that help the immune system maintain health.

During the recent SARS-CoV-2 outbreak, for example, scientists in Brazil determined that the virus attacks certain gut bacteria with a cascading effect on lung immunity, while at the same time lung inflammation alters the microbiota of the respiratory system, consequently killing bacteria in the gut (<https://doi.org/10.3389/fimmu.2021.635471>). Immunology researchers are in the process of trying to understand what unites these physiological systems that serve such vastly different functions.

One explanation of how the microbiome interacts with the immune system could come from the mucosal membrane, a layer of epithelial cells that line the respiratory and gastrointestinal tracts. In fact, mucus, antimicrobial agents, and immune cells cover all the surfaces of the body that interact with the outside world. (Although, the colon contains the largest, most diverse population of microbes.) Until now, these membranes have been viewed simply as a physical barrier, but scientists have found that pathogens interact directly with mucosal epithelia, triggering cellular changes (<https://doi.org/10.3389/fcimb.2020.602312>).

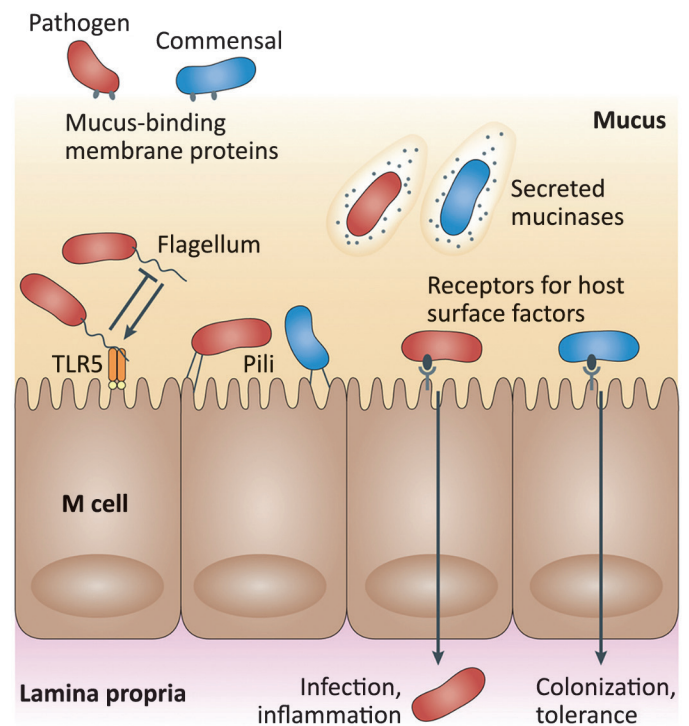


FIG. 1. An image of how the mucosal membrane and the gut microbiota interact with epithelial cells as part of an immune system response. Source: Donaldson, et al., *Nat. Rev. Microbiol.* 14: 20-32, 2016

In response to a pathogen, immune cells can migrate from mucus to threatened tissue through the lymphatic system (Fig. 1, page 7). When this happens, epithelial cells in the mucosal membrane secrete peptides that inhibit specific microbial growth. At the same time, receptors established by genetics orchestrate the colonization of certain bacterial lines. Lymphoid cells also coordinate a response by releasing compounds that regulate bacterial composition in the gut.

The specifics surrounding these observations of the microbiome's role in the mucosal immune system are still being worked out; however, researchers believe that the relationship between epithelial cells and gut bacteria is established in infancy. The adult immune system is activated at birth when the body is first exposed to microbes outside the womb. According to the hygiene hypothesis, exposure to a variety of new cell types early in life is crucial for training the system. Put another way, a sterile early life disorganizes the immune system, increasing the viability of inflammatory pathologies that can lead to allergies or, possibly, irritable bowel disease later in life.

Mucosal cells on infant skin and in the mouth are the gateway to a healthy microbiome that leads to a robust adult immune system capable of fending off pathogens. Scientists are not sure how long the window to feed the microbiome stays open, but they are realizing it is crucial in way they had not previously imagined (<https://www.nature.com/articles/s41385-020-0257-y>).

CENTRAL NERVOUS SYSTEM

"There seems to be something about the social brain, in particular, that makes it sensitive to signals from the microbiome," John Cryan, a biochemist at University College Cork in Ireland, said in a *Nature* article published last year. In the past five years, researchers have determined that gut bacteria are involved in producing bioactive compounds that travel the central nervous system (CNS) up to the brain, affecting thinking and behavior.

Cryan has been studying the association between microbiome composition and autism spectrum disorder. Studies show that children with autism consistently have less *Veillonellaceae*, *Coprococcus*, and *Prevotella* bacteria in their gut. In a study with just five participants, the microbiomes of autistic individuals were transplanted into mice to see how gut bacteria is involved with brain function (<https://doi.org/10.1016/j.cell.2019.05.004>). The microbiomes did not produce as much of two specific amino acid metabolites that increase the activity of a neurotransmitter involved in sensory processing and motor control, key deficits in autism spectrum disorder. The results from this small study, along with other animal experiments, have prompted researchers to consider the microbiome as a possible mechanism for autism treatment.

There is also evidence that interactions between the microbiome and the central nervous system regulate synaptic plasticity and cognition. Certain amino acid receptors interact with the microbiome, and elevated cyanobacteria in the intestines correlate with a build-up of neurotoxins in the brain that hinder their function. Diseases like amy-

otrophic-lateral sclerosis (ALS), Parkinson's dementia, and Alzheimer's are all associated with altered CNS neurochemistry that potentially originates in the gut (<https://doi.org/10.3389/fncel.2013.00153>).

Evidence suggests that the microbiome's connection with the immune system might be the link that influences CNS behavior, but this nascent area of research is still developing. Some experiments have indicated such a possibility, while other experiments signal a different communication mechanism. Researchers have also found that the presence or absence of short-chain fatty acids (SCFA) produced as metabolites of gut bacteria, may act as signaling molecules that modulate cell activity.

SHORT-CHAIN FATTY ACIDS

Soluble fiber in the human diet is fermented in the colon by resident microbes and turned into short-chain fatty acids, such as butyrate, propionate, and acetate. The SCFA are absorbed by the intestines into the bloodstream, where studies show they perform vital roles, like modifying gene expression and regulating cholesterol synthesis.

The common SCFA have been studied for a long time, although researchers still have a lot to learn about them (<https://doi.org/10.1038/d41586-020-00195-1>). They are used by the colon's epithelial cells as a source of energy and act as protein receptors that regulate lipid and glucose metabolism. SCFA are known to activate fatty acid oxidation while inhibiting synthesis and lipolysis to reduce the amount of free fatty acids in the blood (<https://doi.org/10.1194/jlr.R036012>).

While continuing to look for answers to questions about how SCFA fluctuations affect human health, researchers also want to better understand branched short-chain fatty acids. For molecules that are so prevalent in the body, not much is known about how they interact with human tissue or what receptors they trigger.

Branched-chain fatty acids (BCFA) are produced through a different fermentation mechanism than SCFA that involves the fermentation of proteins and amino acids instead of soluble fiber. The breakdown of proteins releases nitrogen, which is essential for bacterial growth. However, degradation of proteins is also accompanied by potentially toxic metabolites, such as amines, phenolic compounds, and volatile sulfur compounds.

The significance of BCFA remains unclear. Published results by a team at Stanford University in Palo Alto, California, led them to believe BCFA might regulate cells that produce a protein in mucous membranes involved in the immune system (<https://doi.org/10.1038/d41586-020-00195-1>). A recent study by a group of German scientists comparing SCFA and BCFA in vegans versus omnivores found that the amount of protein individuals ate did not influence the BCFA concentration in their guts. The researchers hypothesized that gut bacteria may drive SCFA synthesis and maintain stable levels regardless of nutrient levels, switching to proteins as a source when needed (<https://doi.org/10.3390/nu13061808>). Not enough data has been gathered on these SCFA to draw any lasting conclusions.

Continued on page 10

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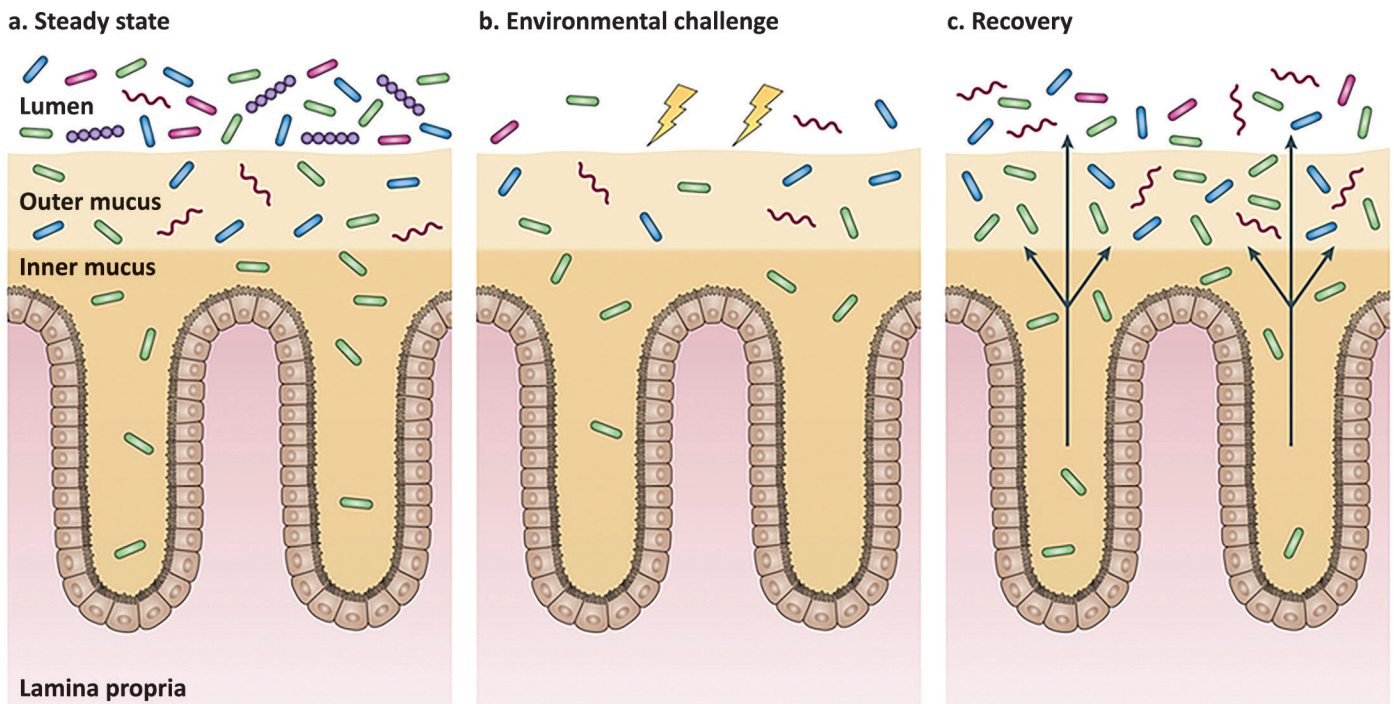


FIG. 2. Researchers imagine that the major population of bacteria in the gut are protected from disruption by dietary changes or antibiotic intake by residing in intestinal niches protected by mucus. Source: Donaldson, *et al.*, *Nat. Rev. Microbiol.* 14: 20-32, 2016

In time, we are more likely to understand the role of these ubiquitous compounds.

EATING FOR A HEALTHY GUT

It is clear that multiple factors, such as your genes, birth, environment, and the medicine you take, influence the composition of bacterial colonies in the gut. Add diet to this complex system, and differentiating between important factors for maintaining a healthy microbiome becomes a significant research challenge. Still, different microbes have different optimal conditions for growth, and dietary choices eventually end up in the gut.

Researchers have conducted experiments on specific food items and found that certain options can impart beneficial effects. As mentioned earlier, microbiota living in the colon possess enzymes that break down dietary fiber through fermentation. Results from studies on fruits, vegetables, beans, and whole grains prove that when we eat these high-fiber foods their fermentation releases short-chain fatty acids into the large intestines. The high concentration of SFCA's lowers the pH, creating an acidic environment where harmful bacteria do not prefer to live.

However, as researchers pointed out in a paper published in May 2021, studies that focus on one food item are incomplete. Dietary components can have counteracting or synergistic effects, and the diet as a whole should be considered when determining what makes a healthy microbiome (<https://doi.org/10.1093/ajcn/nqab077>). The research team from the Department of Food and Nutrition at the University of Helsinki in Helsinki, Finland, conducted a study examining the gut

Information

Associations of healthy food choices with gut microbiota profiles, Koponen, K., K., *et al.*, *Am. J. Clin. Nutr.* 00: 1–12, 2021.

Short- and branched-chain fatty acids as fecal markers for microbiota activity in vegans and omnivores, Trefflich, I., *et al.*, *Nutrients* 13: 1808, 2021.

Microbiota modulation of the gut-lung axis in COVID-19, de Oliveira, G.L.V., *et al.*, *Front. Immunol.* 12: 214, 2021.

Mucosal immunity-mediated modulation of the gut microbiome by oral delivery of probiotics into Peyer's patches, Lin, *et al.*, *Sci. Adv.* 7: eabf0677, 2021.

Mucosal epithelial cells: the initial sentinels and responders controlling and regulating immune responses to viral infections, Yang, J. and H. Yan, *Cell Mol. Immunol.* 18: 1628–1630, 2021.

Imprinting of the immune system by the microbiota early in life, Al Nabhani, Z. and G. Eberl, *Mucosal Immunol.* 13: 183–189, 2020.

The gut microbiome in neurological disorders, Cryan, C.F., *et al.*, *Lancet Neurol.* 19: 179–194, 2020

The gut microbiome, Brody, H., *Nature* 577: S5, 2020.

microbiomes of nearly 5,000 Finnish people who consumed foods recommended for a healthy diet. Participants reported what they ate each day in their omnivorous diets, and the researchers assigned a score to healthy food items. Some participants also provided stool samples for DNA analysis of the microorganisms.

The study did not produce overwhelming results. There was no dramatic difference in the microbiome of individuals with a higher healthy food score. But the researchers do claim that their results show a modest association between healthy food choices and “compositionally distinct microbiota.”

As in previous studies, the Finnish researchers also found that fiber has the greatest influence on gut health. Individuals with higher healthy food scores contain more fiber-degrading species of bacteria in their gut, along with more bacteria that produce SCFA and the resulting metabolic enzymes. They also found that the guts of individuals who reported eating less red and processed meat contained fewer enzymes associated with negative health effects known to contribute to colorectal cancer.

Many studies indicate the gut microbiome does not stray too far from a base bacteria population when it is in a state of homeostasis (Fig. 2). Exceptions include infancy, old-age, and during antibiotic treatments. These are times when bacterial numbers are low, and scientists believe there could be a benefit from adding a probiotic to the diet. Otherwise, the science is still out on whether adding prebiotics and probiotics to the diet provide any advantage during homeostasis.

In general, confusion about connections between the diet and the microbiome stem from a lack of research on the human body. In such a complex system, scientists have no choice but to build pared-down models to begin to understand how the process works. That means most of the experiments performed to explain the microbiome, including those mentioned here, are conducted on genetically engineered mice or in a petri dish. The few human studies that have been conducted involve very small groups of people, often fewer than 50.

This article barely scratches the surface of all the immunological and bioengineering research that has taken place in the past five years—not to mention the nutritional studies scientists have conducted. It is likely that in the next five years, more consumer products and pharmaceuticals will apply this research knowledge to improve gut health which, as we now know, leads to better overall well-being.

In the coming years, more microbiome experiments will head into the clinic. Research is inching toward treatments aimed at manipulating the mucosal layer as a means of fighting off viruses (<https://doi.org/10.1038/s41423-021-00650-7>). Scientists are also hopeful that they can eventually treat autoimmune disorders, such as lupus, type 1 diabetes, rheumatoid arthritis, and multiple sclerosis, with a regiment of pre- and probiotics (<https://doi.org/10.1038/d41586-020-00197-z>).

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



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Ultra-low dioxane in ethoxy sulphate products

Ilio Sebastiani, Antonio Milicia, and Icilio Adami

Production of both ethoxylated alcohols and their derivatives, ethoxy sulphates, has increased in the last decade, thanks to their growing use as non-ionic (ethoxylated alcohol-based) and anionic/non-ionic (ethoxy sulphate-based) surfactants in all categories of detergent formulations, such as home care, personal care, and industrial and institutional cleaning.

- The progressive reduction of the permitted content of 1,4-dioxane in ethoxy sulphates presents an increased challenge for surfactant producers.
- This article discusses technical solutions that can be put in place based on the chemistry of the 1,4-dioxane molecule and its formation mechanism during surfactant production.
- Desmet Ballestra's "ultra-low dioxane technology" is discussed in detail, with emphasis on the technical features, advantages, and achievable quality.
- Technologies for 1,4-dioxane degradation and disposal into water effluent streams are also illustrated.

The ethoxy sulphates, in particular, find wide applications in personal care formulations, as they provide "high and consistent" quality in terms of both performance (foaming power, mildness, degreasing effectiveness) and purity (absence—or minimization—of by-products) when strictly required.

When reference is made to the reaction by-products of ethoxy sulphates, the focus is on the 1,4-dioxane whose allowed maximum concentration has been progressively reduced (Fig. 1).

The progressive reduction of the permitted content of 1,4-dioxane in ethoxy sulphates presents an increased challenge for surfactant produc-

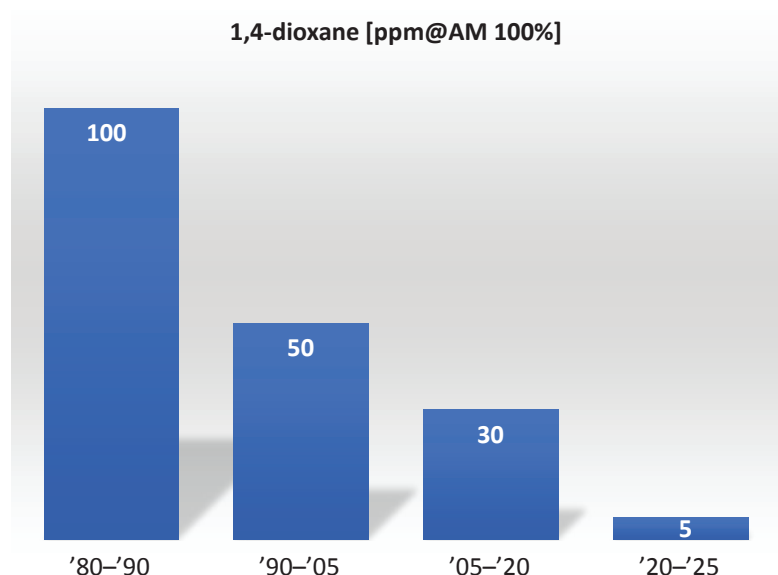
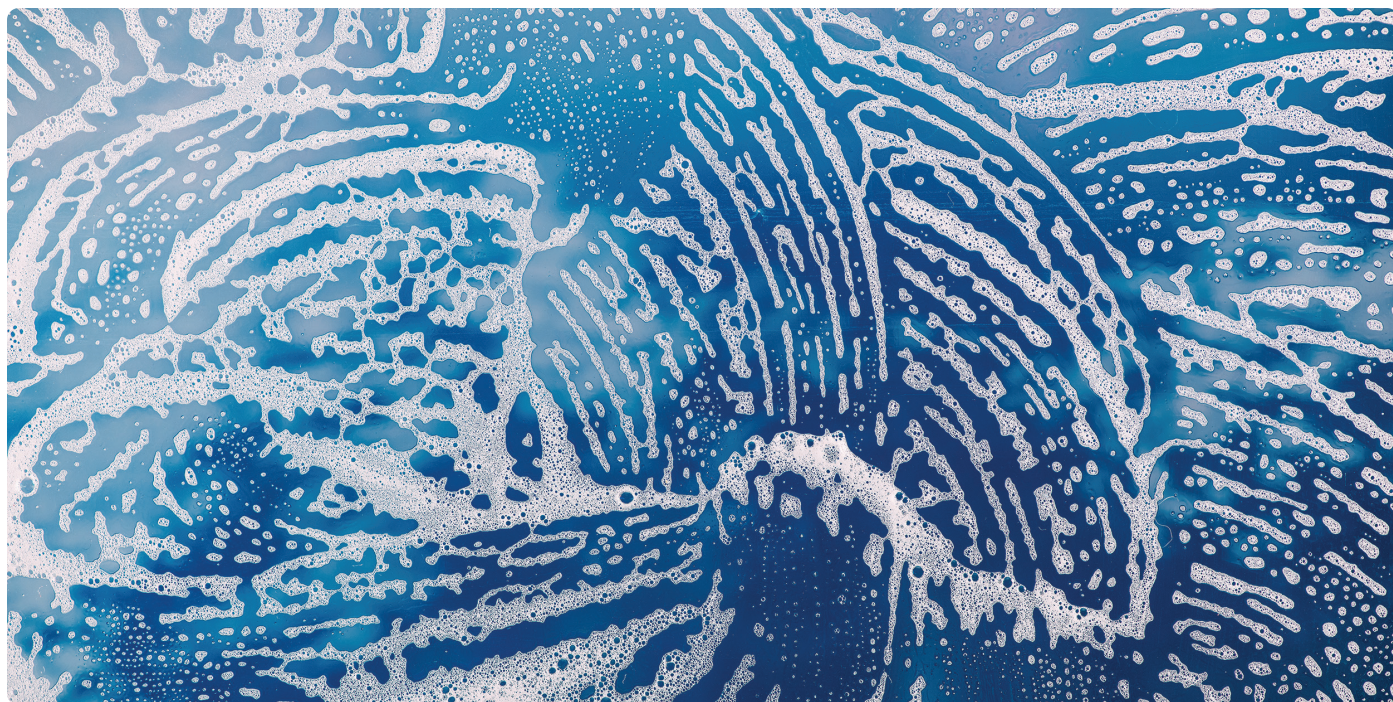


FIG.1. 1,4-dioxane content trend into ethoxy sulphate products (reference SLES 3EO)



ers (see Regulatory Review on page 37), and the technologies involved are continuously developed and upgraded to cope.

1,4-dioxane is an unavoidable by-product in the production of ethoxylated surfactants (Fig. 2).

Particular reference should be made to two important processes, namely:

- Fatty alcohol ethoxylation (which produces ethoxylated alcohols performing as “non-ionic” surfactants); and

- Ethoxylates sulphation (which produces ethoxy sulphated alcohols performing as “anionic” surfactants).

Although used as solvent in several chemical products, in detergent products 1,4-dioxane derives exclusively from the contribution of the above-mentioned processes; therefore, the control and minimization of its concentration in surfactants and detergents necessarily focuses on ethoxylation and sulphation optimization and upgrading processes.

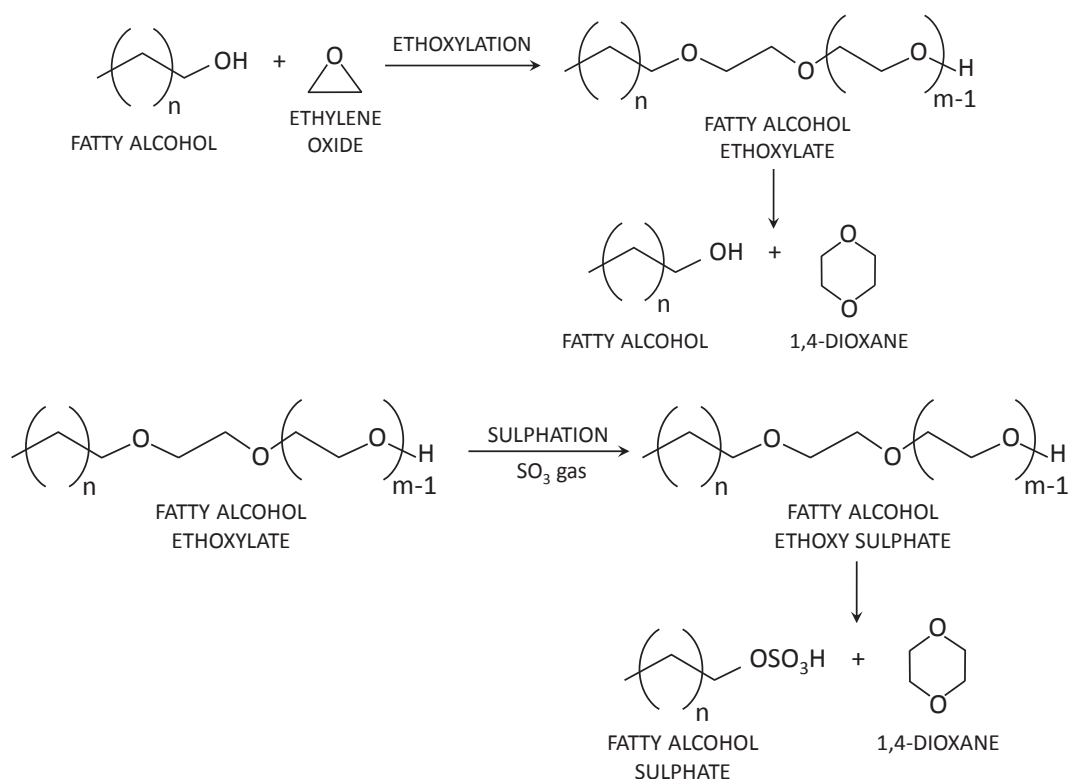


FIG. 2. 1,4-dioxane formation reactions

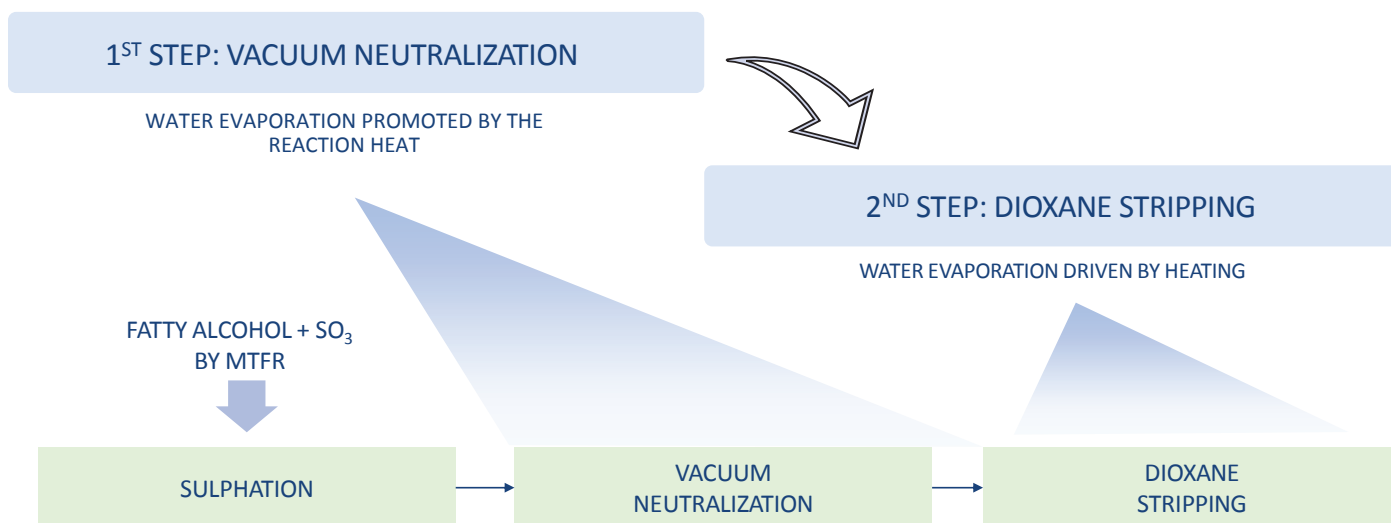


FIG.3. Process block diagram: MTFR sulphation, vacuum neutralization, and dioxane stripping

Due to its chemical structure and characteristics, 1,4-dioxane forms easily when the condensed ethylene oxide (EO) chain is subject to acidic environments, but forms less readily in alkaline environments.

As a consequence of this behavior, the presence of 1,4-dioxane in surfactants containing condensed EO chains is mainly due to the sulphation process.

While the product obtained by ethoxylation may contain a maximum of a few units (ppm) of 1,4-dioxane, the 1,4-dioxane contents of products obtained by a sulphation process may be as much as two orders of magnitude higher.

It is therefore of utmost importance to design process equipment that optimizes the process conditions in a way that minimizes the formation of this by-product whenever SO₃ gas comes into contact with ethoxylates.

The production of ethoxy sulphates is based on the sulphation of the ethoxylates by gaseous SO₃, followed by the neutralization of the obtained sulphonic acid by an alkali.

The production of “ultra-low 1,4-dioxane ethoxy sulphates” requires an additional step to further reduce the content of this by-product which is substantially based on the vacuum boosted evaporation of the mixture water-dioxane.

The state-of-the-art technology developed and industrially implemented by Desmet Ballestra for the production of ethoxy sulphates characterized by ultra-low content of 1,4-dioxane is illustrated in Figure 3, with reference to the following process steps:

1. Sulphation by MTFR (multi tube falling film reactor)
2. Neutralization by “vacuum neutralization” unit
3. 1,4-dioxane stripping by “de-diox” unit

ETHOXYLATES SULPHATION BY MTFR

The sulphation falling film reactor is the tool capable of providing the best and fastest possible contact between the two reactants (gas and liquid) in a way that favors the first order reaction while providing the most accurate control of the chemical and physical modification taking place at the gas-liquid interface.

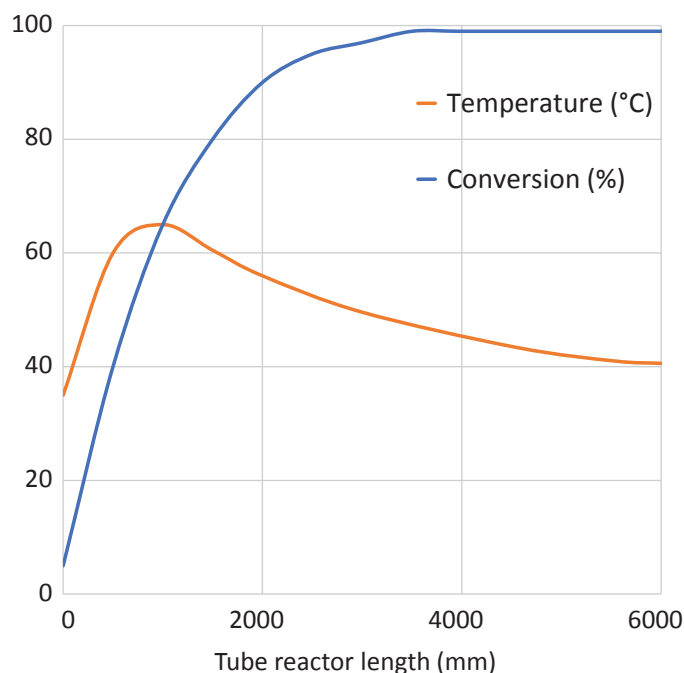


FIG. 4. Conversion and temperature profiles into MTFR

This peculiarity is extremely important, because the reaction rate is gas-phase controlled and the SO₃ concentration will approach the value of zero at the liquid interface, provided the mole ratio of the reactants is kept at ideal levels.

In the falling film reactor, the overall sulphation mass balance is determined by the exponential reduction of the SO₃ absorption rate from top to bottom of the reactor.

Because of this non-linearity in the gas-absorption, the exothermic heat of reaction will follow the same pattern (Fig. 4).

Consequently, a marked peak of temperature will result in correspondence of the top part of the reactor where the liquid film is formed and the gas absorption is maximized.

The ability to control the reaction speed, keeping the ideal ratio at any gas-liquid contact point, depends on the design and construction feature of the reactor.

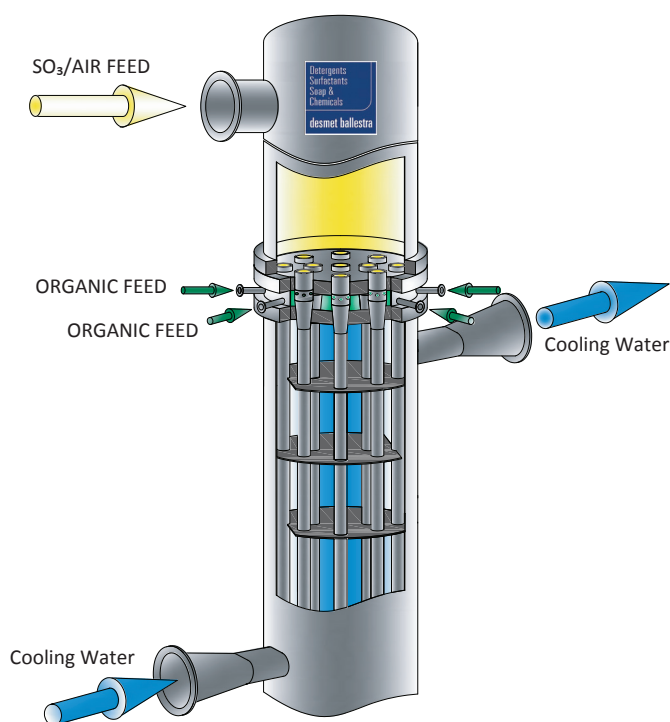


FIG. 5. Schematic design of the top part of Desmet Ballestra MTRF

The MTRF designed and developed by Desmet Ballestra fully complies with the above-mentioned tasks, and its mechanical and operating peculiarities are particularly suitable to guarantee correct operations when ethoxylates are processed. As shown in Figure 5, the SO_3 -gas is fed to the upper part of the reactor and equally distributed into each reaction tube.

The organic feedstock fed on top of the reactor is co-currently contacted with the gas. At the reactor outlet, the liquid is separated from the exhaust gas, and the gas is then sent to a specific treatment to minimize emissions to the atmosphere—at 20 ppm vol. (for SO_x) and 25 mg/m³ for organic mist, respectively.

After being separated from the gas, the stream of sulphonic acid is sent to the neutralization unit (Fig. 6, page 16). The main advantages of this reactor come from its special design, which makes it possible to control the raw material and SO_3 -gas mass flow in a way that maintains the exact ratio between them in each single reaction tube, where the exact mole ratio between gas and liquid reagents is continuously and automatically self-adjusted regardless of the number of tubes in operation, thus eliminating any risk of under or over-sulphonation.

In fact, an exact and ideal mole ratio between SO_3 and organic feed is essential to obtain the maximum conversion degree with minimum side reactions and color increases with all kinds of feedstocks.

The best theoretical sulphonation yield for a given organic feedstock is achieved by optimizing the SO_3 /organic mole ratio and equal distribution of the reactants in the sulphonation device.

Any SO_3 excess, especially in areas of the reactor where cooling is critical, will cause local charring with consequent poor color and product worsening.

Continuous improvements to the design and construction features of the Desmet Ballestra MTRF make it possible to limit the 1,4-dioxane content down to a maximum of 30–50 ppm in the sulfonic acid leaving the reactor, when processing ethoxylates in the range of 1–3 EO.

VACUUM NEUTRALIZATION UNIT (VACUUM NX)

The modern manufacturing of top-quality surfactants is quite complex and sophisticated: Not only must the process equipment be based on proven technologies, reliable engineering, and construction standards, but also the overall process efficiency must be enhanced by application of state-of-the-art, real time process controls.

Achieving premium quality on a regular basis does not depend only on the choice of the raw materials and type of processing, but also depends on the choice of basic equipment and auxiliary units—as well as control system design, which is also extremely important for the success of the entire operation.

The neutralization of the sulphuric ester coming out of the sulphonation section operates on a single step where, upon providing an intimate contact of the acid substrate and the alkaline neutralizing solution, the heat of reaction is removed by water evaporation, enhanced by vacuum operation, in substantially isothermal conditions (Fig. 6).

The neutralization reactor consists of a special wiped-film, high-shear mixer on the top of which the reagents (i.e., acid, water, and alkali), fed under strictly controlled rates, are thoroughly mixed by a rotor with special impellers.

The reaction completeness is controlled by measuring and adjusting the pH of the product, which is discharged at the reactor bottom, after homogenization in the unit.

A special in-line-sampling device ensures continuous electrode-controlled reading of pH value in real time.

The equipment is automatically kept at the operating pressure by a vacuum pump and condenser system.

The neutral product is extracted from the bottom of the reactor by a volumetric pump and fed to an in-line post blender designed for pH cross check and/or pH fine-tuning and addition of buffer agents and minor components, such as preservatives.

The reaction heat is removed by the evaporation of dilution water, resulting in degassing and consistent stripping of undesired low boiling by-products (i.e., 1,4-dioxane).

The product temperature is accurately controlled by the vacuum maintained in the reactor and by the temperature control in the reactor jacket.

The automatic control of the different process parameters (i.e., flow rate of reagents, vacuum degree, and temperature of thermostating water) also ensures accurate set of the product concentration. The main advantage of this system, if compared to traditional ones of “loop type,” is related to the low residence time, which ensures an accurate pH control and a high

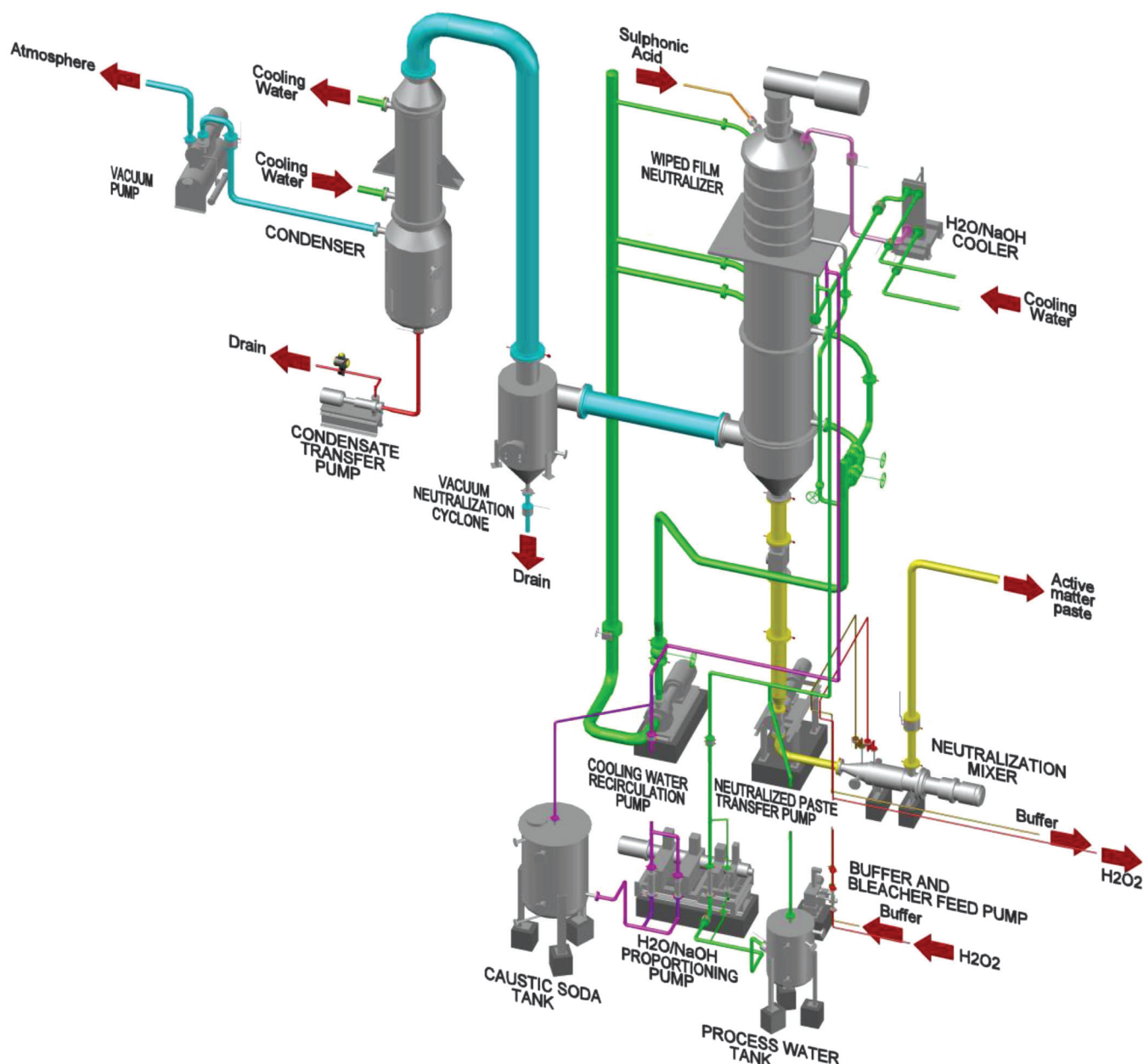


FIG. 6. 3D schematic drawing of Desmet Ballestra Vacuum NX

operation flexibility, especially during start-up, shut-down, and product change-over (thus minimizing or totally eliminating off-spec production).

The specially designed wiped-film neutralizer is therefore characterized by:

- high process flexibility (combined ideal neutralizing, degassing, and stripping);
- short residence time of product in the reactor, with fast response to pH-control;
- overall close correlation between operating parameter and product specs; and
- optimized energy consumption.

Moreover, the mechanical design of the inner parts ensures very efficient mixing that benefits product distribution and consequent decrease of apparent viscosity of the product and increase of heat-transfer rate.

Normally, 1,4-dioxane contents in the range 10–15 ppm (on 100% active matter) are obtained at the vacuum neutralization unit outlet, when neutralizing incoming sulfonic acid streams having a 1,4-dioxane content in the range 30–50 ppm.

The ethoxy sulphates obtained this way are then fed to an additional 1,4 dioxane removal step in the de-diox unit, which is designed to achieve extremely low residual dioxane contents in the ethoxysulphate paste.

DE-DIOX STRIPPING UNIT

The de-diox stripping unit consists in a dioxane/water stripping system, where the evaporation is provided by an external source of thermal energy. The neutralized product coming out of the vacuum neutralization is fed under mass flow control to a stripping chamber featured and fit into a loop scheme (Fig. 7)

The neutralized ethoxy sulphate paste is fed to a high-shear mixer where a controlled amount of dilution water is purposely added to the paste under moderate recirculation.

The paste is then passed through a plates heat exchanger, where it is heated by steam. The recirculation of the operation provides the necessary energy for the subsequent evaporation step, avoiding peaks of temperature that could promote undesired hydrolysis reactions.

The paste is then sprayed, by means of specifically designed nozzles, into a vacuum chamber where the evaporation of the mixture of water and 1,4-dioxane is performed. The evaporation chamber is kept under vacuum by means of the same vacuum pump and condenser of the vacuum neutralization section.

The condensate stream collected in the condenser bottom vessel is finally disposed, while incondensable gases can be safely released to the atmosphere.

The design peculiarities of these process steps result in a "dioxane profile" similar to the one shown in Figure 8.

1,4-DIOXANE REMOVAL FROM WASTEWATER

The last step deals with the 1,4-dioxane chemical treatment or destruction; this step enables chemical degradation of 1,4-dioxane by opening the molecule ring to match local environmental restrictions. A variety of technologies can be adopted for the chemical degradation of 1,4-dioxane, as illustrated in Figure 9, page 18. Among the mentioned technologies, most of the processes are still at an academic and research program level. Only a few, such as advanced oxidation processes, are at a sufficient stage of maturity to guarantee robust and reliable application at an industrial level.

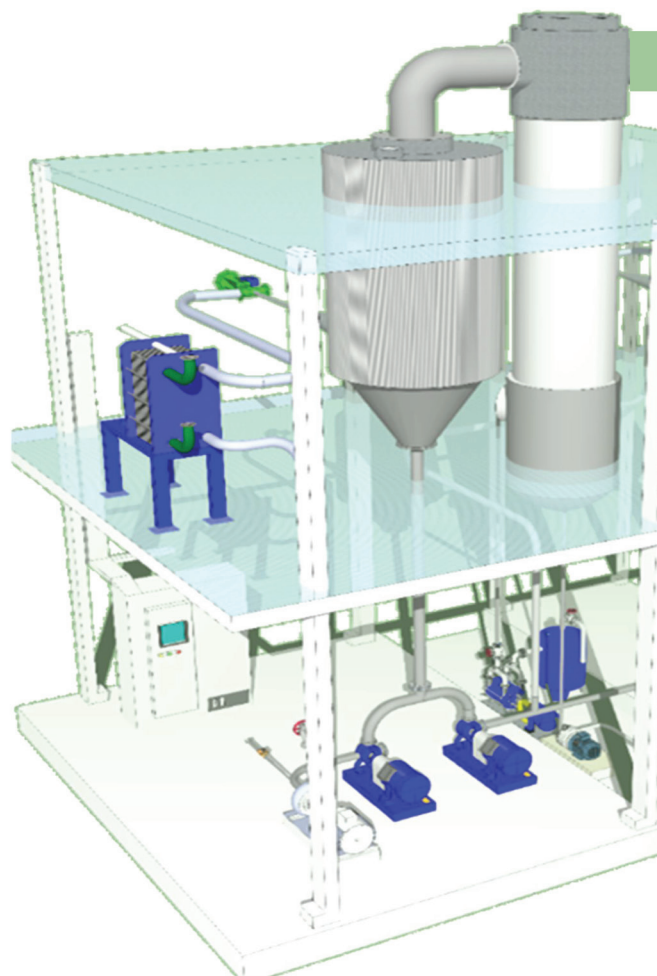


FIG. 7. 3D schematic drawing of Desmet Ballestra de-diox stripping unit

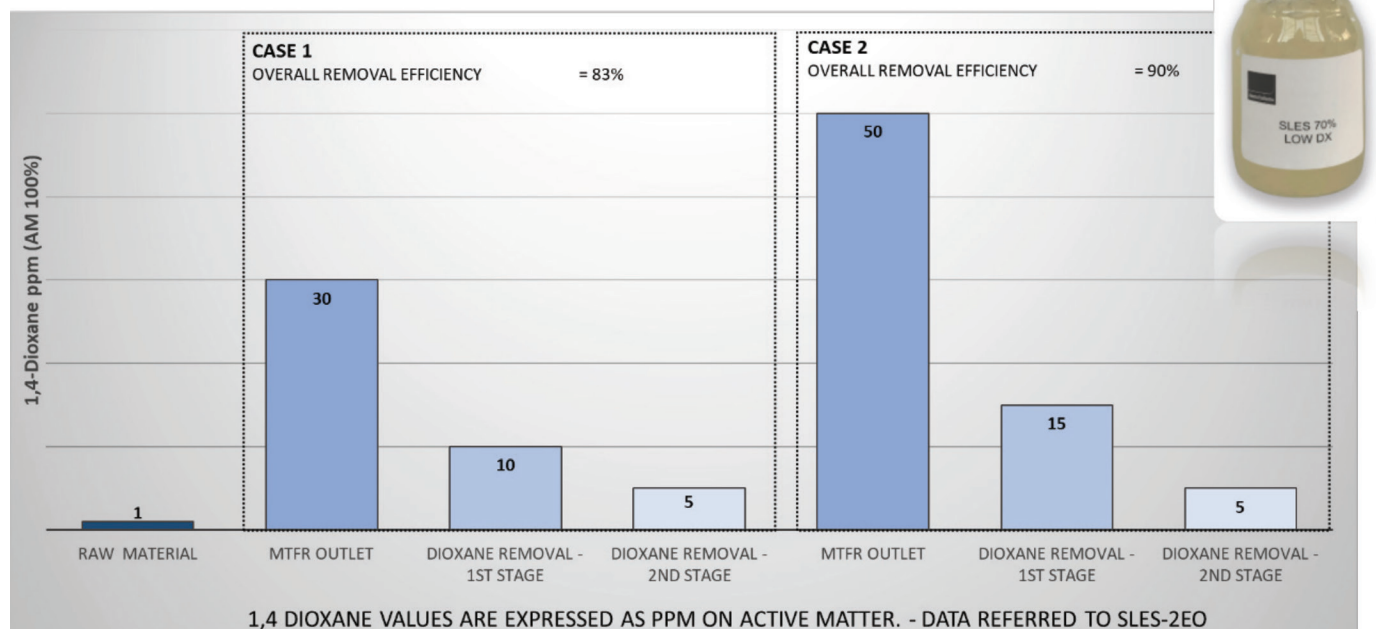


FIG. 8. 1,4-Dioxane content profile along with the processing stage

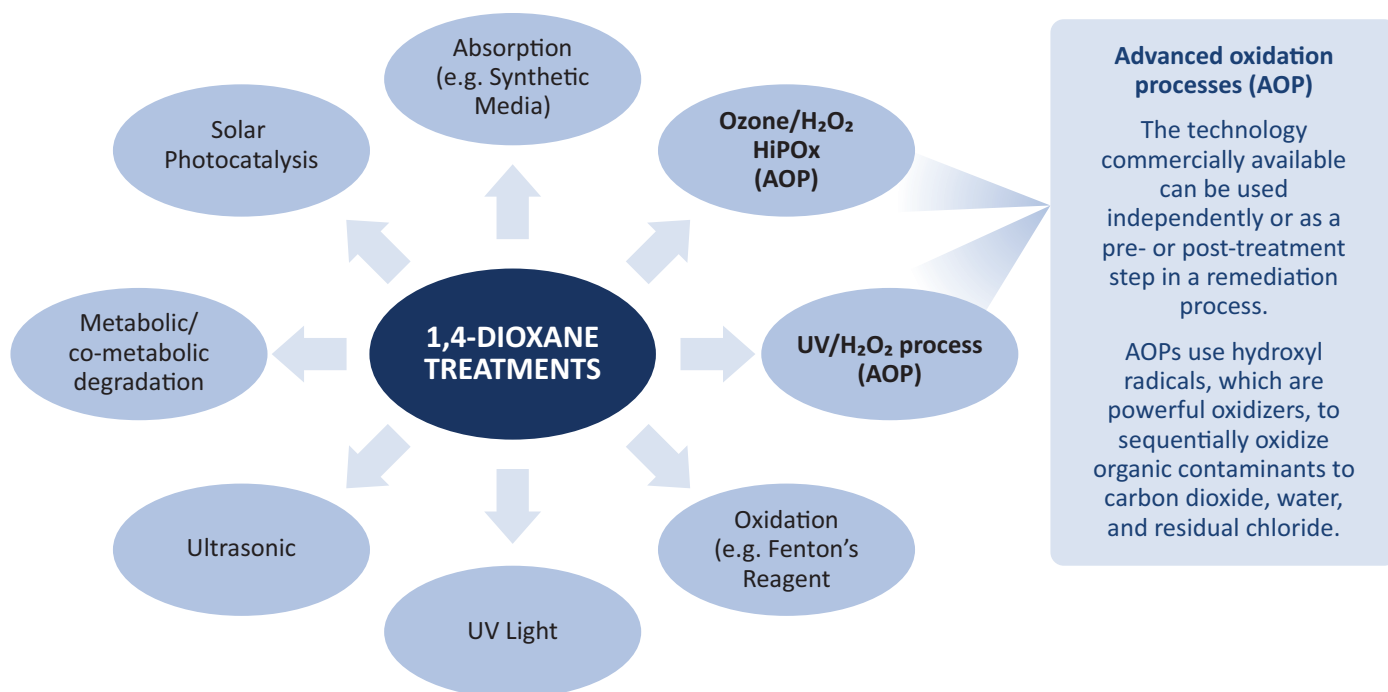
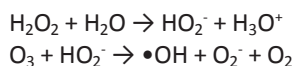


FIG. 9. Technologies for the chemical degradation of 1,4-dioxane

Advanced oxidation processes (such as ozone/H₂O₂ or UV/H₂O₂ or others) combine the synergy of two oxidant systems with the advantage of not generating water sludge, which requires further separated treatments. For example, the water effluent which undergoes an advanced oxidation process based on ozone/H₂O₂ is added with the oxidants in the right proportions and intimately mixed. The combination of ozone and hydrogen peroxide generates the hydroxyl radicals (•OH) according to the following reactions:



The hydroxyl radical acts as the functional oxidant in breaking the cyclic molecule of the 1,4-dioxane. It is worth mentioning that the ozone/H₂O₂ treatment involves the following main process units:

- air oxygen enriching (e.g., pressure swing adsorption, and so on);
- ozone generator;
- oxidant dispersion (e.g., ejectors, diffusers, among others); and
- final ozone thermal removal.

TO SUM IT ALL UP

In the previous paragraphs, we illustrated all the required technologies to ensure the manufacturing of ultra-low 1,4-dioxane ethoxy sulphate products: from the 1,4-dioxane removal by the advanced stripping processes (vacuum and secondary stripping) to the final 1,4-dioxane destruction present in the stripped water.

The technologies by Desmet Ballestra we presented are proven, reliable, and are additionally compliant with the latest water effluent management and safety regulations. The technologies entail several advantages for the surfactant industry, such as:

- reduced need of investments for existing plant retrofitting;
- no disruptive changes in the current supply chain, which could affect raw materials availability and product pricing policy;
- no need for product re-formulation (costly and time consuming); and
- minimized increase of the operating costs, due to the use of heat of neutralization to promote the evaporation of 1,4-dioxane.

In parallel with the technology development, the surfactant industry should be prepared to actively promote clear communication about this issue to consumers and avoid unscientific positions, such as referring to the harmfulness of detergents to humans and aquatic form of life when the cause (1,4 dioxane), once addressed can be technologically reduced to the minimum threshold set by health and environmental protection regulations.

Illo Sebastiani is Head of Research and Development at Desmet Ballestra. He can be contacted at Illo.Sebastiani@ballestra.com. Antonio Milicia is a process engineer in in Research and Development at Desmet and can be reached at Antonio.Milicia@ballestra.com. Icilio Adami is a consultant who works with the Research and Development team at Desmet Ballestra, and can be contacted at Icilio.Adami@ballestra.com.



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Silver ion chromatography for the analysis of trans-fatty acid isomers in humans

Na Wei, Heather C. Kuiper, Enada Archibold, and Hubert W. Vesper

- More than 65 different fatty acids, including positional and geometric fatty acid isomers, have been reported in humans. These isomers have the same molecular mass and very similar chemical structures but can have very different biological functions.
- The large number of cis- and trans- isomers in human blood pose analytical challenges and require detailed investigations to appropriately determine their effects on health and changes in the amount and composition in people.
- Silver ion chromatography, a technology used to separate complex fatty acid mixtures, in combination with an expanded version of Centers for Disease Control and Prevention (CDC) gas chromatography-negative chemical ionization-mass spectrometry (GC-NCI-MS) method for fatty acids, can effectively separate cis- and trans-fatty acid isomers. This approach has potential for determining comprehensive trans-fatty acid profiles in people.

Trans-fatty acids (TFAs) are unsaturated geometric isomers of cis-fatty acids with at least one double bond in the trans- configuration. TFAs are manufactured from partially hydrogenated plant oils and occur naturally in ruminant animals. People are exposed to both sources of TFAs in foods they eat.

Dietary TFA intake is associated with increased risk for coronary heart disease. Worldwide, TFA consumption causes an estimated half million deaths annually, prompting the World Health Organization to call for manufacturers to rid TFAs from the food supply by 2023. Measurement of TFAs in blood tells us about human exposure levels and can be used to assess the effect of policies and regulations to reduce TFAs in people.

The four major TFAs commonly reported in blood include palmitelaidic acid, elaidic acid, vaccenic acid, and linoelaidic acid. Measures of those TFAs from the National Health and Nutrition Examination Survey were 54% lower among adults in 2009–2010 compared with measures from 1999–2000. However, comprehensive information about different TFAs in blood is very limited. Therefore, we have been working to develop new, highly specific and sensitive analytical methods for determining TFA profiles in human blood.

WANT MORE?

The authors of this article presented their work at the 2021 AOCS Annual Meeting & Expo. Their presentation is available at <https://www.eventscribe.net/2021/AOCS/speakers.asp?pfp=BrowsebySpeaker>.

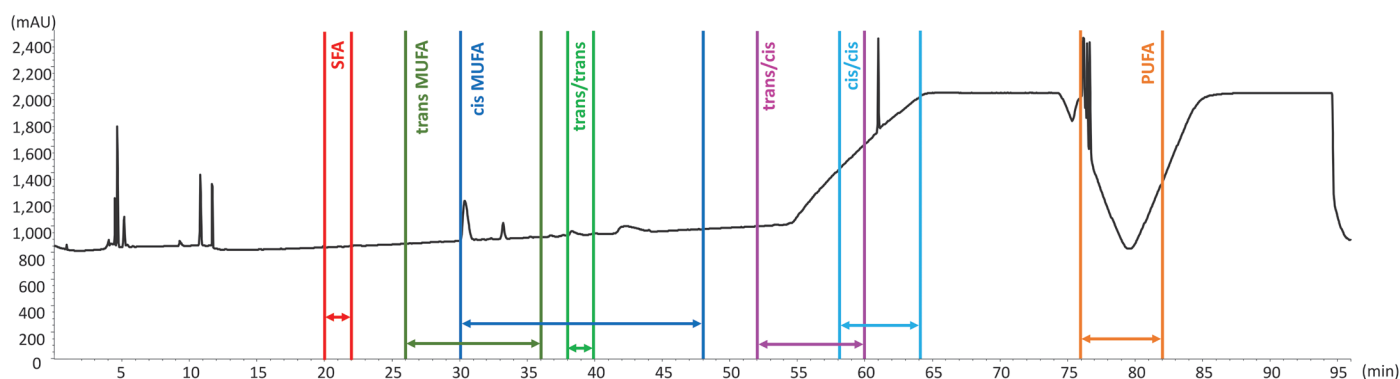


FIG. 1. Silver ion HPLC chromatogram of a human plasma sample separated on two silver LC columns in series and visualized by UV detector at wavelength 210 nm. Fractions are shown by fatty acid type: saturated fatty acids (SFA) (20–22 min, red); trans monounsaturated fatty acids (MUFA) (26–36 min, dark green); cis MUFA (30–48 min, dark blue); trans/trans polyunsaturated fatty acids (PUFA) (38–40 min, light green); trans/cis PUFA (52–60 min, purple); cis/cis PUFA (58–64 min, light blue); and PUFA (76–82 min, orange).

POSITIONAL AND GEOMETRIC FATTY ACID ISOMERS IN BLOOD

More than 65 different fatty acids have been identified in biological samples, with the C18:1 isomers being particularly well characterized. Isomers of C16:1 and C20:1 have been reported in foods and oils but have not been well described in biological samples. The geometric and positional fatty acid isomers have the same mass-to-charge ratio (m/z) and very similar chemical structures, requiring a special analytical approach to ensure appropriate isomer separation for accurate identification and quantitation.

Analytical methods to separate fatty acids typically use a highly polar 100 m column, such as the SP-2560 or CP-Sil 88. These columns give good resolution for many fatty acids but are not able to separate all isomers. In our laboratory, we use a 200 m Select-FAME column for fatty acid analysis. We get better isomer separation with the longer column than with shorter columns, but it is still insufficient to separate all isomers.

SILVER ION CHROMATOGRAPHY

Researchers have used silver ion chromatography in lipid research since 1962, specifically for the separation of cis- and trans- isomers. Since then, silver ion chromatography has proven to be a valuable tool for separating fatty acids by double-bond number and geometry. Various approaches have been used to analyze fatty acid cis- and trans- isomers in oils and dairy products. Those include thin-layer chromatography (TLC), solid phase extraction (SPE), and high-performance liquid chromatography (HPLC) silver ion chromatography approaches, such as AOCS Recommended Practice Ce 1g-96. Despite its utility, silver ion chromatography has seen limited application to biological samples.

We previously developed a silver ion SPE method for assessing cis- and trans- isomer resolution. However, that method had limitations in resolving the wide range of fatty acids in human plasma. We have now developed a method using silver ion high-performance liquid chromatography

(Ag+-HPLC) together with gas chromatography-negative chemical ionization-mass spectrometry (GC-NCI-MS). We expanded the CDC GC-NCI-MS method for the quantitation of fatty acids in human plasma to enable detection of additional fatty acids.

After GC-NCI-MS processing, human plasma samples underwent acidic and alkaline hydrolysis. We extracted the free fatty acids and used Ag+-HPLC to separate them. Silver fractions were collected, derivatized with pentafluorobenzyl-bromide, and analyzed by GC-NCI-MS. Fatty acids were identified by m/z and retention time, in comparison with commercial standards.

We used a Dionex Ultimate 3000 UPLC equipped with a diode-array detector operated at 210 nm to conduct Ag+-HPLC. Two ChromSpher lipid columns were used in series to separate isomers. The solvent system included hexanes, acetone, and acetonitrile. Fractions were collected in a Gilson FC204 fraction collector operated at 5°C. The Ag+-HPLC order of elution for fatty acids was determined using commercial standards and applied to human plasma samples. The elution order starts with the saturated fatty acids, trans- isomers eluting before their cis- counterparts and monounsaturated fatty acids eluting before polyunsaturated fatty acids (Fig. 1).

We confirmed fatty acid HPLC elution order and isomer separation by GC-NCI-MS analysis of each fraction with m/z and retention time comparison with synthetic standards. To optimize Ag+-HPLC method parameters, we used a synthetic standard mixture containing 67 fatty acids, including 18 saturated fatty acids and 49 unsaturated fatty acids. Figure 2 (page 22) shows the separation achieved with this approach, using synthetic material from the C14:1 region to demonstrate the capabilities of Ag+-HPLC combined with GC-NCI-MS. The top chromatogram shows the C14:1 synthetic sample containing C14:1n-5t and C14:1n-5c. The second chromatogram contains Ag+-HPLC fractions collected at 28–36 minutes and contains only the trans- isomer. The third chromatogram contains fractions collected at 36–40 minutes and does not contain C14:1 isomers. The last chromatogram shows fractions collected at 40–44 minutes and containing only the C14:1 cis- isomer.

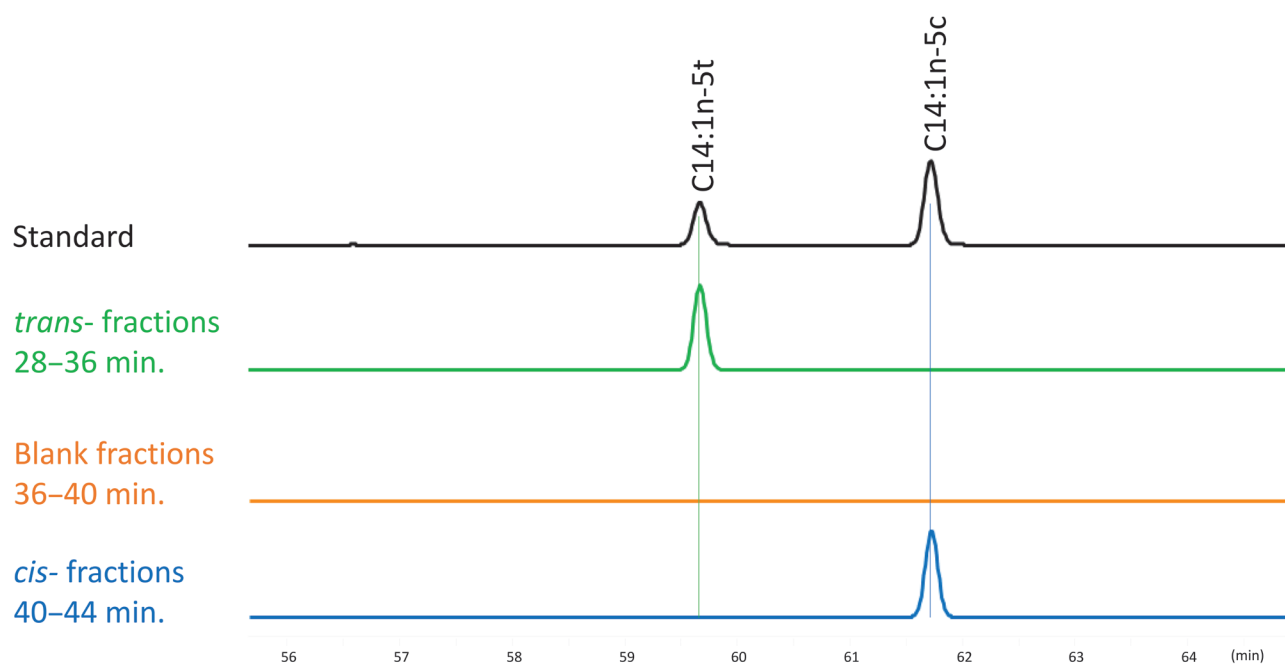


FIG. 2. GC-MS SIM chromatograms of C14:1 isomers in a synthetic standard after fractionation by silver ion chromatography. A synthetic fatty acid standard mix (black) was fractionated by silver ion chromatography. Fractions were analyzed by GC-NCI-MS. Fractions at 28–36 min (green) contain C14:1n-5t, and fractions at 40–44 min (blue) contain C14:1n-5c. HPLC resolution of the trans- and cis-isomers was confirmed by not detecting any fatty acids in two fractions (36–40 min, orange) in between the trans- and cis- fractions.

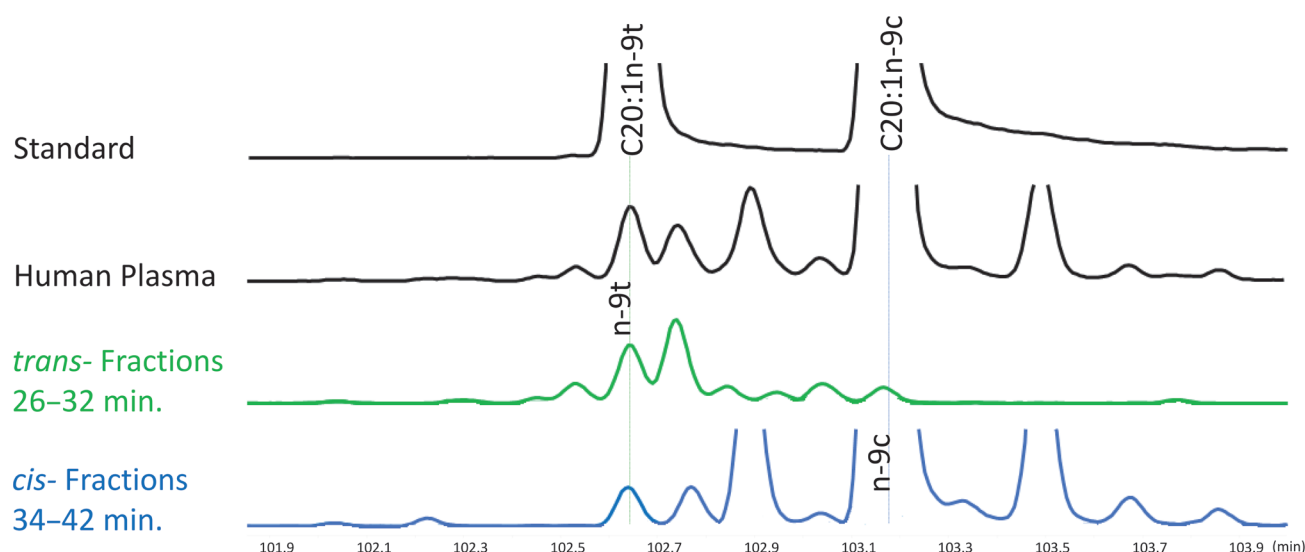


FIG. 3. SIM chromatogram of the C20:1 region (m/z 309.5) from a synthetic fatty acid standard mix (black – top); a human plasma sample (black – bottom); and Ag+-HPLC fractions of human plasma trans-fatty acids (green) and cis-fatty acid (blue) analyzed by GC-NCI-MS. The HPLC resolution of trans- and cis-fatty acid fractions was determined using the synthetic fatty acid standard mix before plasma separation. All labeled peaks were identified by comparison with synthetic material.

Figure 3 illustrates how Ag+-HPLC allows for effective separation of trans- and cis- isomers in human plasma. When combined with GC-NCI-MS, this technique can be used to separate fatty acids that might otherwise coelute. In this figure, we show the C20:1 fatty acid isomers in human plasma, finding at least 12 isomer peaks present for m/z 309.5. Upon separation with Ag+-HPLC, these coeluting isomers are separated into trans- and cis- fractions. The trans- fraction contains nine C20:1 positional isomer peaks and the cis- fraction contains 10 positional isomer peaks. Synthetic standards for the C20:1n-9t

and C20:1n-9c isomers are commercially available and shown here for identity confirmation. Both isomers appear to be present in the plasma sample. However, when we apply Ag+-HPLC to separate the plasma into trans- and cis- fractions, peaks are seen in both fractions at retention time 102.65 minutes (corresponding to the C20:1n-9t standard) and at 103.18 minutes (corresponding to the C20:1n-9c standard). This coelution can only be detected using Ag+-HPLC with GC-NCI-MS.

With the combination of Ag+-HPLC and GC-NCI-MS, we can detect more than 140 fatty acid peaks in human plasma,

including more than 60 trans- positional isomers. In addition to the four trans- and 17 cis- fatty acids currently quantitated with the CDC TFA GC-NCI-MS method, we were able to confirm the identities of 11 trans- and seven cis- fatty acids.

Overall, Ag+-HPLC is an effective tool for separating cis- and trans-fatty acid isomers. Silver fractionation with GC-NCI-MS analysis allows for separation of fatty acid isomers that cannot be fully separated by GC alone, even with 200 m GC columns. Using this approach, initial assessments indicate the presence of at least 140 different fatty acids in the investigated human plasma samples. Further investigations are ongoing to identify these fatty acids, and to further optimize this approach to create TFA profiles in people.

The authors are Centers for Disease Control and Prevention scientists working in the Division of Laboratory Sciences in the Clinical Chemistry Branch. For further information, please contact Heather Kuiper, HKuiper@cdc.gov.

The findings and conclusions in this report are those of the authors and do not necessarily represent the official position of the Centers for Disease Control and Prevention/the Agency for Toxic Substances and Disease Registry. Use of trade names is for identification only and does not imply endorsement by the Centers for Disease Control and Prevention, the Public Health Service and the US Department of Health and Human Services.

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Giants of the past: Robert C. Hastert

(1924–2021)

Michael J. Boyer

Bob Hastert passed away peacefully on June 6, 2021, after a wonderful and rewarding life.

Like many of us in AOCS, Bob never obtained a PhD but enjoyed being referred to as “Dr. Hastert” by AOCS staff. Bob Hastert graduated from the University of Nebraska with a BS degree in chemical engineering and had a successful career in the fats and oils industry, working at the Cleveland, Ohio-based engineering firm Wilson & Co. and then as a top business representative for Harshaw Chemical Co., a diversified industrial chemical producer and major supplier of hydrogenation catalysts used primarily to produce more stable edible oils, shortenings, and the like. He later founded his own consulting business in “retirement”.

Bob’s accomplishments as a “giant” within AOCS are too long to list. He became a member in 1952, and was active in varying capacities until well into the 2000s. His legacy is not in publishing but in bringing hands-on, practical experience in fats and oils, with a focus on hydrogenation, to the industry and Society members.

Bob did contribute as a coauthor on several books related to fats and oils processing, and served as a contributing editor to *Inform*. He was a much sought-after speaker, and could turn hydrogenation into the most interesting subject at any conference with his striking speaking abilities.

Bob also received just about every major service award within AOCS, including the Alton E. Bailey and A.R. Baldwin Distinguished Service awards as well as the Processing Division Distinguished Service Award. He became an AOCS Fellow and Emeritus member later in life. His main interests were in edible applications, processing, and the Processing Division, but Bob would jump into any subject or area of governance that called out for help—and he always did so with eagerness, leadership,



and results. He once told former *Inform* editor George Willhite that he regarded every day as a new challenge—maybe like a game—and did his best to accomplish as much as he could.

Bob Hastert served AOCS in many capacities. He was on the Governing Board, and was the AOCS President in 1987. He offered assistance in fundraising, advertising, and all manner of other areas within AOCS. He helped organize and co-chaired Annual Meetings and other specialty conferences.

But perhaps his biggest accomplishments arose from his ability to size up younger members and then mentor and encourage them into roles he thought would do the most benefit for AOCS and their individual careers. The author is one such person. Bob decided that the Society needed a World Conference Planning Committee while he was president in 1987. World conferences were becoming a big part of AOCS activities, and he saw the value and had the vision to make this a focused area. Bob then sought me out to be the first Chairman of that committee.

Personally, Bob Hastert was one of the most likeable persons and AOCS members I have known. Bob married his high school sweetheart, Ava Bromwich, on September 7, 1941. Bob and Ava had four children, eight grandchildren, and four great-grandchildren. Ava Hastert lost her partner of 73 years on June 6, 2021.

Bob loved the outdoors. This including hiking and back packing with his son. He was active in the Mouth of Platte chapter of Lewis and Clark Trail Heritage Foundation. For many years he organized an annual fishing trip to Canada with “the fellas”. One of my biggest regrets is that I never managed to make time to accompany him after his many invites.

All of us should seek to have a life as full as Bob Hastert’s.

Michael J. Boyer is President of AWT Management Services, which focuses on business strategy and environmental management issues. He can be contacted at mikebawtms@gmail.com.

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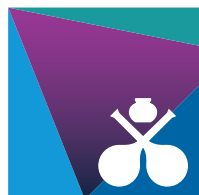
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Physicochemical characterization of licuri oil and ucuuba butter

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LICURI OIL

Licuri (*Syagrus coronata*) is a fruit obtained from a palm tree from the semiarid regions of northeastern Brazil (Fig. 1) (Bauer, *et al.*, 2013) Licuri has a high content of lipids (~ 49%); 11.5% in its almond-like seeds (Queiroga, *et al.*, 2010).

- The Brazilian rainforest is a rich source of exotic fats.
- The physical and chemical properties of such fats are not well-known.
- This article looks at the physicochemical characterization of two fats from the Brazilian Amazon Rainforest: licuri oil and ucuuba butter.



FIG. 1. Licuri fruit. Source: eCycle

Its fatty acid composition (Table 1) shows the presence of medium-chain fatty acids (6 to 12 carbons) that have unique properties with important nutritional and medical applications (caprylic, capric, lauric, and myristic acids) (Trevizam, *et al.*, 2014). This composition, which is comparable to that of coconut oil, contains approximately 83% of short- and medium-chain fatty acids (Bauer, *et al.*, 2013; Queiroga, *et al.*, 2010). Table 2 shows the triacylglycerol composition of licuri oil.

Licuri oil shows a prominent crystallization peak between 1.28°C to -22.09°C (Fig. 2). Although more than 80% of this oil is composed of saturated fatty acids, most are short-chain fatty acids, which have lower melting temperatures.

TABLE 1. Fatty acid composition of licuri oil

Main fatty acid (%)	Licuri oil
Caprylic acid (C8:0)	11.39
Capric acid (C10:0)	7.06
Lauric acid (C12:0)	43.77
Myristic acid (C14:0)	13.48
Palmitic acid (C16:0)	6.26
Stearic acid (C18:0)	3.57
Oleic acid (C18:1)	10.75
Linoleic acid (C18:2)	3.72
Σ saturated	85.53
Σ unsaturated	14.47

TABLE 2. Triacylglycerol composition of licuri oil

Main triacylglycerol	%
CyCLa	3.10
CyLaLa	9.37
CLaLa	10.13
LaLaLa	15.81
LaLaM	16.30
CyLaO	3.85
LaLaP	8.64
CLaO	3.40
LaLaS	5.70
LaLaO	8.28
LaLaLa	2.91
LaMS	2.60
LaMO	4.97
LaPO	2.60
LaOO	2.34

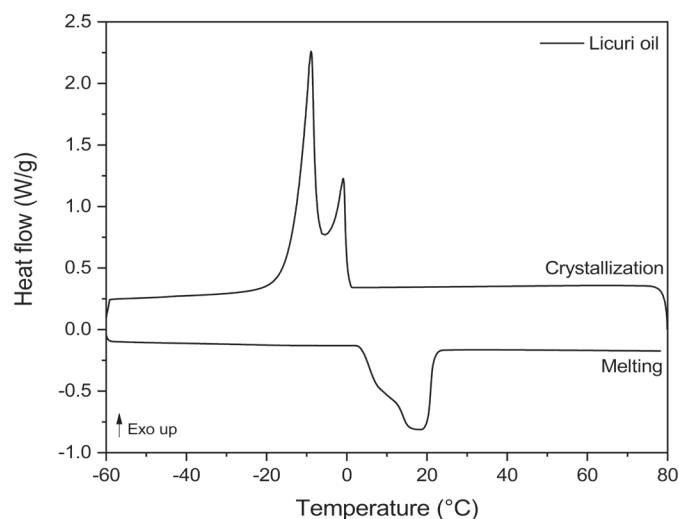
Cy: Caprylic; C: Capric; P: Palmitic; O: Oleic, La: Lauric, L: Linolenic, M: Myristic.

The application of licuri oil is for cooking, as a supplement in the feeding of ruminants (Lima, *et al.*, 2015), and production of soaps (Bauer, *et al.*, 2013). The presence of medium-chain fatty acids provides an excellent skin penetration (Trevizan, *et al.*, 2014).

UCUUBA BUTTER

The ucuuba, *Virola surinamensis* (Rol. Ex Rottb.) Warb., has a fruit with a scarlet-red endocarp (Fig. 3). Ucuuba butter is extracted directly from the fruit, which has between 60 to 70% fat in its seeds (Ramos, *et al.*, 2019). This butter has as its identity standards an iodine index of 33.67 gI₂/100g and a saponification index of 224.26 mgK_{OH}/g. The fatty acid composition of ucuuba butter can be seen in Table 3.

Ucuuba butter has a high content of myristic, lauri, palmitic, and stearic acids. This fat can be classified as a lauric fat,

**FIG. 2. Crystallization and melting curves obtained by differential scanning calorimeter (DSC) as a function of temperature for licuri oil****FIG. 3. Ucuuba fruit.** Source: Biovontade France**TABLE 3. Fatty acid composition of ucuuba butter**

Main fatty acid (%)	Ucuuba butter
Capric acid (C10:0)	0.74
Lauric acid (C12:0)	15.66
Myristic acid (C14:0)	68.17
Pentadecyl acid (C15:0)	0.71
Palmitic acid (C16:0)	5.58
Margaric-oleic acid (C17:1)	1.81
Stearic acid (C18:0)	7.34
Σ saturated	98.19
Σ unsaturated	1.81

due to the high levels of lauric and myristic acids, as well as palm kernel oil. Among the triacylglycerols, nine of them are composed of myristic acid (Table 4, page 28).

Ucuuba butter had a crystallization range between 23.52°C and 0.39°C and melting point from 19.85–47.77°C (Fig. 4, page 28).

TABLE 4. Triacylglycerol composition of ucuuba butter

Main triacylglycerol	%
LaLaLa	1.02
MLaLa	6.47
MLaM	42.95
MMM	39.72
PLaM	1.72
PMM	4.49
SLaM	1.93
SMM	1.71

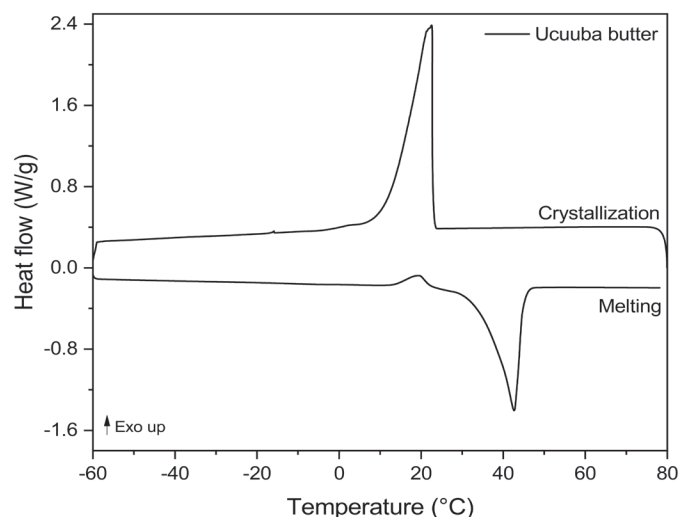
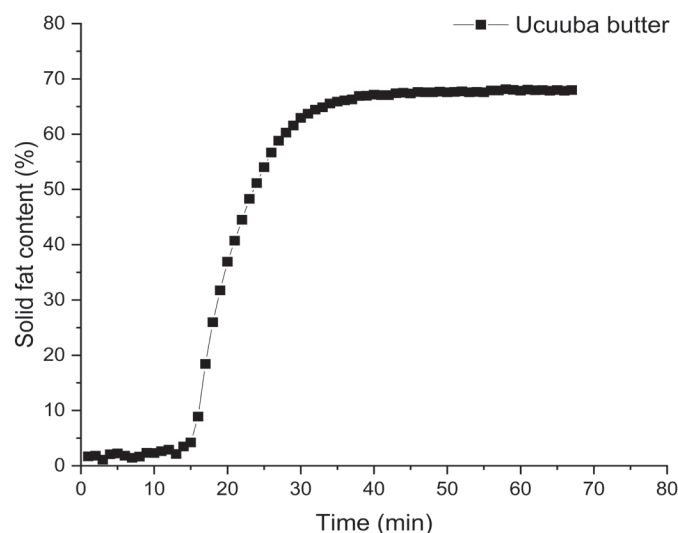
P: Palmitic; S: Stearic; La: Lauric; M: Myristic

Two peaks of crystallization that overlaps at high temperatures are observed. These peaks are the result of the high-saturated fatty acid content.

The crystallization kinetics are shown in Fig. 5. The maximum solid content is approximately 70% after 30 minutes at 25°C. The crystallization induction was initiated at 15 minutes, featuring a quick crystallizing butter. The X-ray diffraction analysis showed a predominance of the β' polymorphic.

Studies have been carried out to develop nanoemulsions and nanoparticles with ucuuba butter, because of its great moisturizing potential (Alves, 2018). It has also been used in the production of soaps and in the substitution of animal tallow (Amazon-oil, 2013).

Thais Jordânia Silva, Larissa Magalhães Grimaldi, Fernanda Luisa Lüdtkke, Kamila Ramponi Rodrigues de Godoi, Mayanny Gomes Silva, Renato Grimaldi, and Ana Paula Badan Ribeiro are with the Laboratory of Oils and Fats, School of Food Engineering, Department of Food Technology, University of Campinas, Campinas, Brazil.

**FIG. 4. Crystallization and melting curves obtained by differential scanning calorimetry as a function of temperature for ucuuba butter****FIG. 5. Crystallization kinetics of ucuuba butter at 25°C**

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Phospholipid-containing films for improved drug release

Jiahao Huang and Shawn Wettig

- Phospholipids from natural sources can delay liquid-liquid phase separation and improve supersaturation for active pharmaceutical ingredients with poor water solubility in aqueous media.
- Researchers have developed oral films containing phospholipids to enhance the dissolution efficiency of hydrophobic pharmaceutical ingredients.
- Phospholipid-based oral films provide an alternative approach for compounding pharmacies to formulate drugs with poor water solubility.

A prerequisite condition for the oral absorption of a drug compound is that the drug can dissolve in the water-based gastrointestinal fluid to form a solution. Therefore, the therapeutic efficiency of an oral pharmaceutical product is highly dependent on the aqueous solubility and dissolution rate of the active pharmaceutical ingredient. Today, approximately 40% of approved drugs and 90% of new drug candidates have poor water solubility and present slow dissolution [1]. Such suboptimal properties hinder the development of both new drug products and generic products. Consequently, extensive pharmaceutical research is aimed at designing dissolution-enhancing dosage forms to facilitate the development of hydrophobic compounds into clinically available forms.

Supersaturated drug delivery is a novel formulation technology that enhances drug dissolution by achieving a physical state in which drug concentration can exceed drug solubility. Supersaturation is enabled by the dissolution of a drug from its amorphous state, which has higher solubility than its crystalline counterpart. The supersaturated state has a high Gibbs free energy favorable for drug diffusion and oral absorption; however, it is also a thermodynamically unstable state and requires stabilizing agents to maintain its duration [2]. Polymeric materials are the most common type of stabilizer for this purpose. Maintenance of the supersaturated state is achieved through delaying liquid-liquid phase separation (LLPS) and inhibiting crystal growth induced by the solution properties of polymers (Fig. 1).

In our study, we found that phospholipids from natural sources showed similar ability to maintain supersaturation of certain drugs with poor water solubility. Prior to this, the pharmaceutical applications of phospholipids were mainly based on their solubilization effects (similar to those of a surfactant) and vesicle forming ability to formulate liposomal drug delivery systems [3]. The ability of phospholipids to maintain supersaturation is expected to provide an alternative pharmaceutical stabilizer for supersaturated drug delivery systems that can be compounded into solid and semi-solid dosage forms.

WANT MORE?

The authors of this article presented their work at the 2021 AOCS Annual Meeting & Expo. Their presentation is available at <https://www.eventscribe.net/2021/AOCS/speakers.asp?pfp=BrowsebySpeaker>.

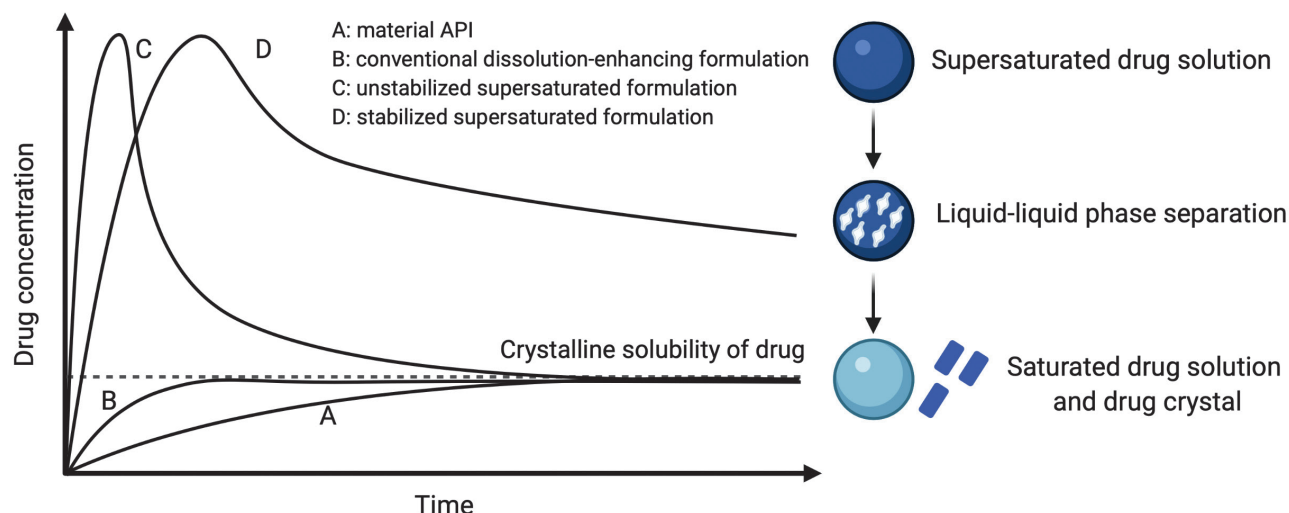


FIG. 1. Illustration of drug supersaturation and crystallization during dissolution (created with BioRender.com)

PHOSPHOLIPID EFFECT ON DRUG LLPS

The onset of LLPS for a drug compound can be determined using a UV double wavelength extinction method upon titrating an organic solution of drug into water. At a non-absorbing wavelength, the drug concentration at which UV extinction significantly increases indicates LLPS onset due to the formation of an immiscible phase (Fig. 2). Correspondingly, the increase in drug absorbance value loses linearity at an absorbing wavelength when LLPS occurs. Indomethacin (IDM) is a classical anti-inflammatory drug that is commonly used as a hydrophobic model drug for novel formulation research. The LLPS onset of IDM in pure water was determined to be 52.8 $\mu\text{g/mL}$. Any increase observed in this value due to the presence of an excipient suggests that the excipient may be useful as a stabilizer for a supersaturated solution of IDM.

LLPS for IDM was observed to be delayed to different degrees in the presence of phospholipids at different concentrations (Table. 1, page 32). With a delayed LLPS up to 171 $\mu\text{g/mL}$, the recrystallization process of a supersaturated IDM solution was expected to be inhibited. The observed effect is comparable with that seen for commercial SDDS polymeric carriers, including sodium alginate and Soluplus®. It is noted that phospholipids may induce early LLPS of other hydrophobic drugs, such as apixaban, which was also examined as part of this study. This effect would likely negatively influence the maintenance of supersaturated solutions for some drugs. Therefore, matching the correct phospholipid with a specific drug is a crucial consideration when using phospholipids as a stabilizing agent for hydrophobic drugs. Based on these observations, we selected IDM for further investigation of phospholipid effect on the drug kinetic-solubility profile.

PHOSPHOLIPID EFFECT ON DRUG CRYSTALLIZATION

The non-sink dissolution condition is commonly used by regulatory agencies to evaluate dissolution performance and crystallization vulnerability of oral drug products in a simulated

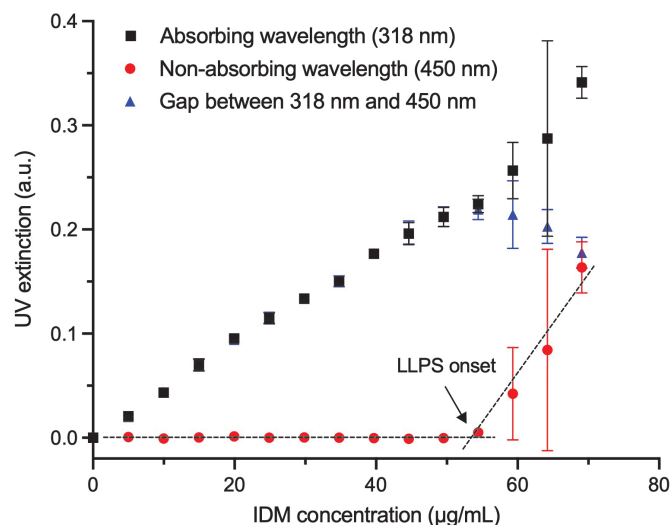


FIG. 2. UV extinction of IDM in pure water at different wavelengths

gastrointestinal environment. As seen in Fig. 3 (page 32), IDM concentration showed a sharp decline until reaching its crystalline solubility (8.1 $\mu\text{g/mL}$) in a water medium free of any excipient, which is a sign of instability. In the presence of phospholipid, the observed rate of decrease in drug concentration was slowed, especially in the early state (0–20 min), maintaining the supersaturation of IDM for longer periods. According to the classical nucleation theory, the concentration-time profile for drug crystallization should show a monotonic decreasing function until reaching drug solubility equilibrium. Therefore, the break point observed in the IDM crystallization rate (at approximately 10 min) indicated the occurrence of a transition state within the whole crystallization process. Within this period, supersaturated drug solution is constantly lowering Gibbs free energy by splitting into two liquid phases, with a minimal buildup of solid phase, so that no obvious reduction in drug concentration was observed. This behavior suggested

TABLE. 1. LLPS values of IDM in the presence of different pharmaceutical excipients

Excipient	Low concentration 320 µg/mL	Mid concentration 800 µg/mL	High concentration 1440 µg/mL
Water	53 ± 1 µg/mL		
Phospholipid	72 ± 5 µg/mL	119 ± 2 µg/mL	171 ± 8 µg/mL
PEG-32 stearate	54 ± 1 µg/mL	88 ± 5 µg/mL	133 ± 2 µg/mL
Polyvinylpyridine	47 ± 1 µg/mL	49 ± 1 µg/mL	49 ± 2 µg/mL
Sodium alginate	143 ± 3 µg/mL	184 ± 2 µg/mL	>200µg/mL
Soluplus®	>200µg/mL	>200µg/mL	>200µg/mL

that phospholipid provided a stabilized drug supersaturation that is essential for oral absorption in the gastrointestinal tract. Also, this effect is comparable with that induced by commonly used polymeric stabilizers.

DISSOLUTION OF PHOSPHOLIPID-CONTAINED FILMS

With validated supersaturation ability, phospholipids are combined with different polymers to formulate solid films for IDM. Their appearances and dissolution behaviors under non-sink condition are shown in Fig. 4.

In the first case, phospholipid was combined with a commercial polymeric drug carrier, polyvinylpyridine (PVP), as a binary matrix for the film base. PVP is well known for its ability to generate a highly supersaturated state followed by a fast loss of free drug concentration, dissolution behavior that is described as the “spring and parachute” effect in pharmaceutical fields. With the addition of phospholipid, the supersaturation profiles of IDM can be adjusted either by increasing the maximum drug concentration or decreasing crystallization rate.

In the second case, phospholipid was combined with an insoluble corn-derived polymer, zein, to form a natural-sourced film carrier for IDM. The supersaturation of IDM-zein-phospholipid film showed a positive correlation with regard to phospholipid content. All films showed a stable first-order dissolution profile without drug concentration decline, and the equilibrium IDM concentration could reach more than 5 times the solubility of IDM after a 6-h dissolution when the film base contains 50% phospholipid.

In the last case, phospholipid was combined with another commercialized pharmaceutical polymer, hydroxypropyl methylcellulose (HPMC). Drug molecules are not freely released from HPMC as they need to diffuse from the swelling structure of HPMC. Consequently, IDM-HPMC showed a typical first-order dissolution. The addition of phospholipid greatly facilitated drug release at the initial stage as seen by the burst release before 50 min. Large amounts of phospholipid generated the “spring and parachute” effect as seen by the fast buildup of supersaturation up to 70 µg/mL followed by drug concentration decay. In this regard, polymer and phospholipid amounts could be adjusted to balance fast dissolution and stabilized supersaturation, to fulfill the specific pharmacokinetic needs of a drug product.

The appearances of IDM films showed different levels of uniformity, depending on the miscibility between polymer,

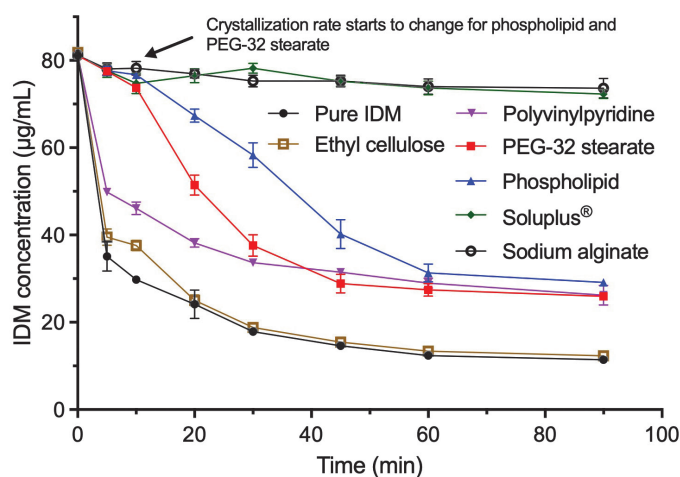


FIG. 3. Crystallization profiles of IDM in the presence of different excipients

phospholipid, and drug. Since phospholipid does not form films by itself, the film forming ability and practicality of film removal from preparation substrate were compromised with increasing phospholipid amount. As such, it is suggested that the phospholipid amount be less than that of the polymer in a formulation to guarantee the suitability of polymer/lipid matrix as film base.

Our investigations demonstrated that phospholipid delayed the onset of LLPS and improved supersaturation for IDM, a drug with poor water solubility. This enabled the formulation of IDM films with improved and tunable dissolution profiles in water. Our ongoing experiments are investigating these behaviors in biorelevant media and using an artificial gastrointestinal tract for more biologically relevant data. The research methodology is being applied to evaluate the usefulness of this phospholipid for the delivery of other hydrophobic drugs. The designed oral films are expected to serve as an alternative approach for pharmacy compounding to formulate solid doses for hydrophobic drugs.

Jiahao Huang is a PhD candidate from the Wettig Research Group at University of Waterloo, School of Pharmacy. He received his BSc in Pharmaceutical Sciences from Shenyang Pharmaceutical University, China. He is interested in exploring the fundamental physiochemical properties involved in the development of dissolu-

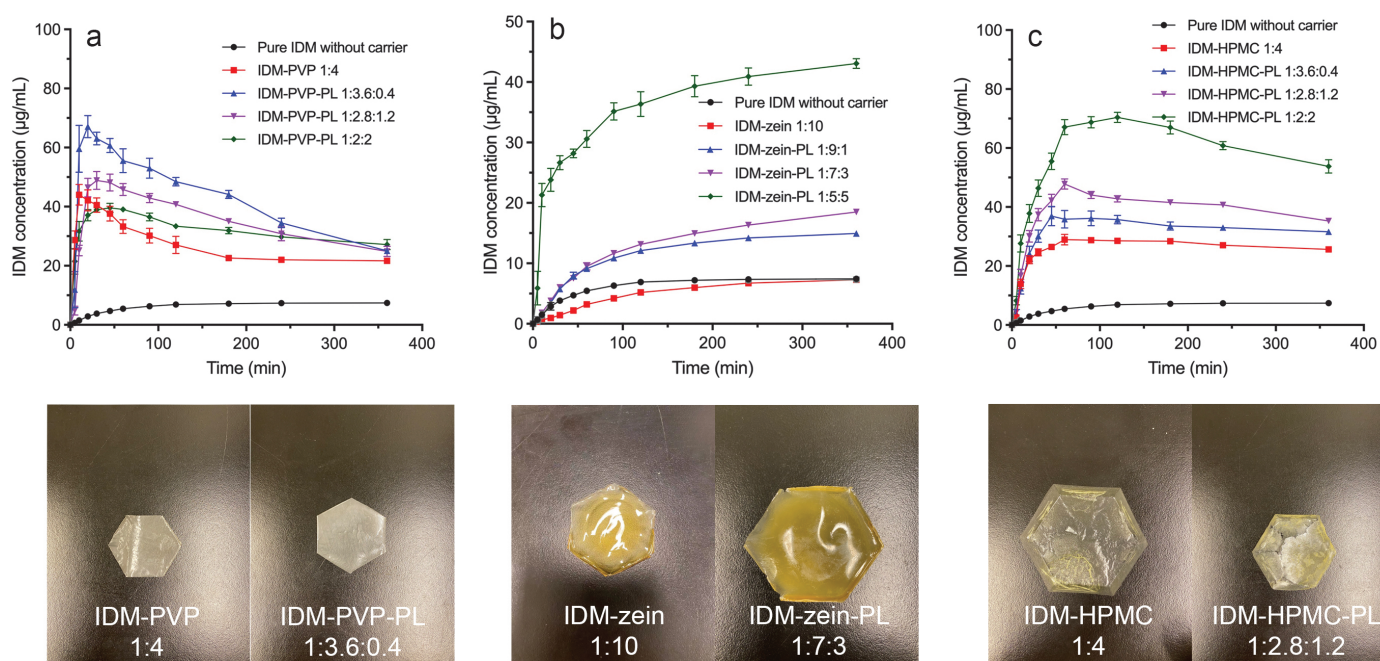


FIG. 4. Dissolution of IDM-PVP-PL films (a), IDM-zein-PL films (b), and IDM-HPMC-PL films (c); film appearances are shown below each dissolution profile.

tion-enhancing dosage forms. He is specifically interested in investigating amorphous solid dispersions using polymeric and lipid materials. These technologies are expected to serve the development of drug, nutraceutical, and food products. He can be contacted at jiahao.huang@uwaterloo.ca.

Shawn Wettig is a professor in the School of Pharmacy and an assistant vice-president for Graduate Studies and Postdoctoral Affairs at the University of Waterloo. He received his BSc in Chemistry and Physics from the University of Lethbridge (Canada) and his PhD in Physical Chemistry from the University of Saskatchewan (Canada). His research interests lie in the general areas of biophysical chemistry and nanotechnology, and at the interface of these two broadly defined areas. This research involves aspects of physical chemistry, solution thermodynamics,

biochemistry, and cell biology applied to the study of self-assembling systems. He has authored over 70 peer reviewed publications on the synthesis, properties, and applications of surfactant and polymer systems in the area of drug delivery. He can be reached at shawn.wettig@uwaterloo.ca.

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Nefarious to noteworthy: Lipid droplets shed their negative image

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

Rebecca Guenard

Since the 1920s, when pathologists observed the presence of lipid droplets in many types of diseased tissue, scientists have wondered if they were harmful to the human body. Last year, a group of scientists at the University of Barcelona, Spain, observed that lipid droplets are pathogen killers that serve a crucial role in the body's immune response (<https://doi.org/10.1126/science.aay8085>). More recently, a team at The Francis Crick Institute in London, UK, discovered that an accumulation of lipid droplets inside the kidney cells could actually protect the renal system from the damaging effects of a high-fat diet. (<https://doi.org/10.1371/journal.pbio.3001230>). The findings prove that lipid droplets serve a far greater purpose in the body than once imaged, opening the door for further investigations and possible treatment applications.

Of all the organelles that make life possible, the lipid droplet (Fig. 1) is relatively simple. Enzymatic esterification of an activated fatty acid or of cholesterol produces triacylglycerides and sterol esters in different ratios, depending on the cell type. Phospholipids surround the fat mixture, enabling an oil-in-water emulsion within the aqueous cytoplasmic environment.

Many living organisms accommodate lipid droplets in their cells, and until recently these inert vesicles were assumed to be simple refueling stations when energy ebbs. Once researchers discovered the droplets have proteins integrated into their membranes and observed a kinship between the droplets and various pathogens, they began considering the unassuming globules differently.

They found that lipid droplets transport fatty acids and proteins within and between cells. The droplets can also act as gatekeepers for the nucleus, inhibiting molecules that control gene expression or generating lipid ligands to activate nuclear receptors. In addition to their interactions with the nucleus and other organelles, the droplets serve a curiously protective role.

"The fact that they correlate with a disease does not mean they are causing that disease. Our work suggests that lipid drop-

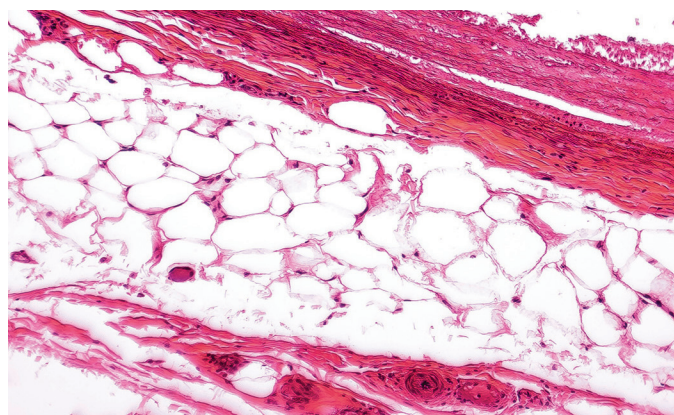


FIG. 1. White adipose tissue, light micrograph, hematoxylin and eosin staining, magnification 100x. Fat cells adipocytes have large lipid droplet which remains unstained.

lets are actually one of the body's ways of fighting back against the disease," says Alex Gould, senior group leader at The Francis Crick Institute in London, UK.

The Gould lab uses the fruit fly species *Drosophila* as a model organism to study lipid droplets since the insects share

several human genes. The researchers knocked out or over-expressed certain genes in the flies to home-in on the important functions of the droplets. They found that in fruit flies, the deceptively simple droplet participates in rather complex metabolic processes. The results, in turn, expose a glimpse of their potential role in the human body.

“Metabolism is so highly conserved across the animal kingdom that you could almost draw the exact same diagram of metabolic pathways in fruit flies as you find in humans,” says Gould.

To understand the involvement of lipid droplets in metabolic ailments like kidney disease, Gould says his group built on recent developments in biochemistry and cell biology that revealed the details of lipid droplet formation. The cellular fuel reserves assemble most commonly within the endoplasmic reticulum of adipose tissue cells. Researchers believe the fats build up within the phospholipid bilayer and then bud off to form a droplet. In doing so, the droplets retain some of the characteristics of the endoplasmic reticulum, often creating an enduring connection with it and other organelles.

Once it detaches from the endoplasmic reticulum, a monolayer of phospholipids envelope a neutral lipid core. A variety of proteins incorporated into the membrane's surface trigger lipid synthesis or hydrolysis according to the cell's needs and steer the droplet to different locations to interact with other organelles within the cell (<https://doi.org/10.1038/s41580-018-0085-z>). Gould discovered that one important job the droplets perform is preventing the build-up of free radicals in the cell.

In earlier experiments, his group revealed that during fruit fly brain development lipid droplets protect polyunsaturated fatty acids (PUFAs), including dietary linoleic acid, during oxidative stress. The PUFAs, essential for healthy growth, do not incorporate into the phospholipid membrane where they are susceptible to damage by peroxidation. Instead, they are buried deep inside lipid droplets, thus limiting the formation of free radicals and reactive oxygen species that could damage developing cells (<https://doi.org/10.1016/j.cell.2015.09.020>).

The Gould group's latest experiments looked at the role of lipid droplet surface proteins in cells of the fruit fly renal system. In mammals, high-fat diet can lead to inflammation and metabolic stress resulting in chronic kidney disease. When the kidneys are in such a state, they commonly contain a smattering of lipid droplets throughout their cells. The renal cells of fruit fly larvae exhibit similar traits on a high-fat diet.

The researchers targeted a particular enzyme in the endoplasmic reticulum known as diacylglycerol o-acyltransferase (DGAT1) that synthesizes triacylglycerides. In their fruit fly model, they switched off the gene responsible for producing the enzyme and saw that the cells suffered more damage. “If you prevent this flow of fats into the lipid droplet, then the whole renal cell does much worse,” says Gould. “This provides evidence that the droplets are protective, not harmful.”

Ramping up the expression of a triacylglyceride digesting enzyme on the droplet surface called adipocyte triglyceride lipase (ATGL) should then help detoxify cells. The researchers performed the experiment on fruit flies fed a high-fat diet and

Information

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observed the restoration of renal function, along with less cell damage. The results point towards a potential mechanism for curing kidney disease.

“Although the new findings are at an early stage, they do suggest that drugs targeting renal lipid droplets may be a useful strategy for treating chronic kidney disease,” says Gould.

Obviously, there is a long way to go before the findings from fruit fly studies could lead to a clinical treatment. Gould says he hopes their work will inspire other researchers to investigate how drugs targeting proteins on the lipid droplet surface can be used to reverse the negative health effects of a high-fat diet on kidneys and other organs. Sequestering lipids in this way allows the cell to safely dispose of excess fats through the mitochondria and avoid an accumulation of free radicals.

A growing body of researchers now recognize peroxidation of phospholipids as a medically significant area worth investigating. Certain cancers and neurodegenerative diseases result from the cascade that begins with the pathological state caused by lipid oxidation (<https://doi.org/10.1016/j.bbrc.2016.10.086>).

In the coming years, research is likely to reveal more mechanisms for the function of lipid droplets and how they might be manipulated to improve human health. Gould credits having an easy to manipulate genetic model like the fruit fly, which allows researchers to quickly target specific cell types and determine the function of their genes. The next step is to see if similar processes occur in mice and then in humans.

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

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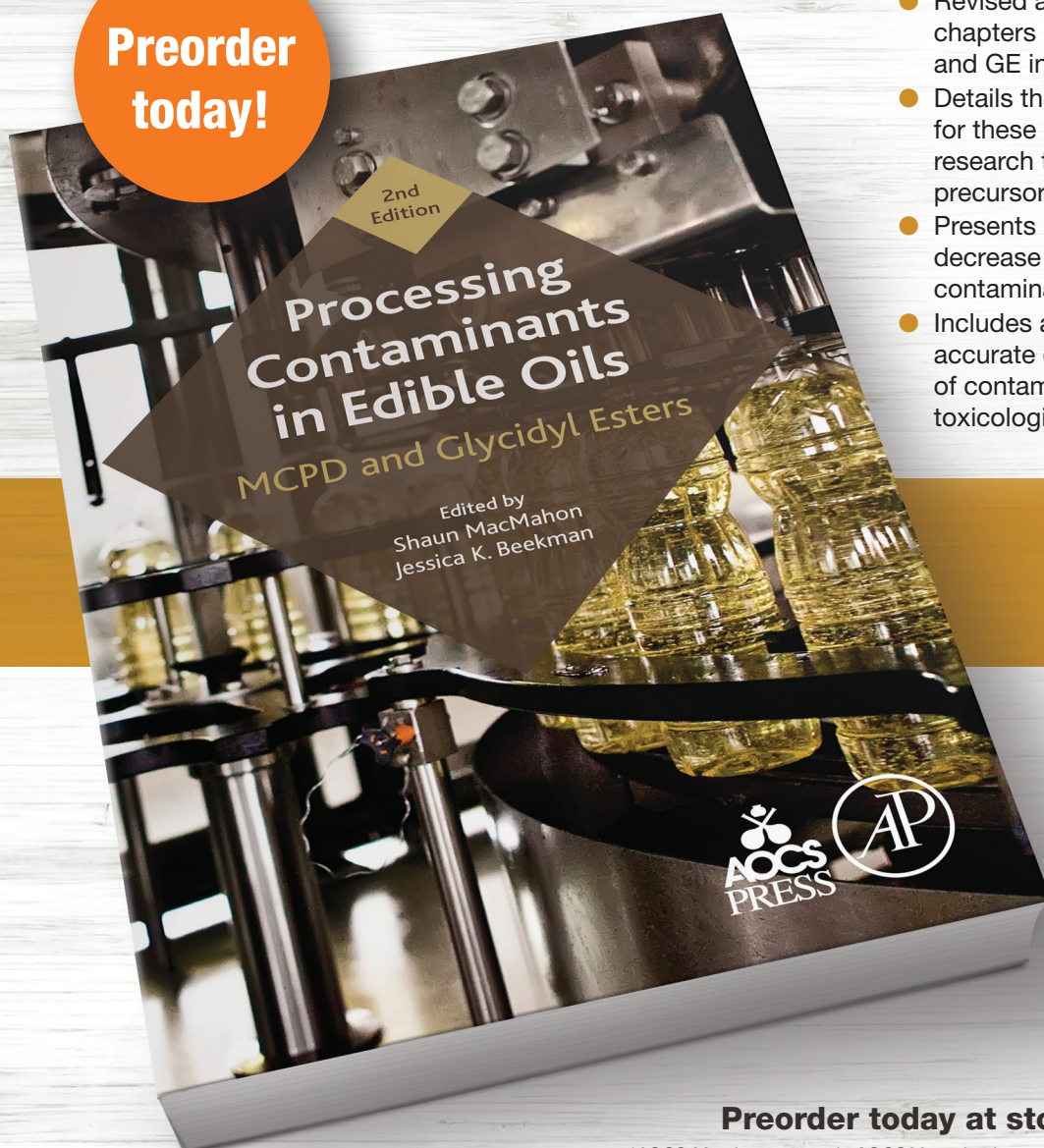
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Updates on increased ethanol exposure and 1,4-dioxane regulations

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

The expanded use of ethanol-containing hand sanitizer during the pandemic has come with a minimal increase in serious health risks, an assessment by the Dutch National Institute for Public Health and the Environment (RIVM) has found.

The Dutch ministries for health (VWS), social affairs and employment (SZW) tasked RIVM with assessing whether people have a greater chance of contracting serious diseases as a result of ethanol exposure during the pandemic.

Ethanol can cause cancer or reduced fertility and affect the development of an unborn child, according to the Health Council of the Netherlands. An impending proposal for the harmonized classification and labelling of ethanol could classify it as carcinogenic, mutagenic, and reprotoxic under the CLP Regulation.

RIVM focused on the risk of breast cancer, colon cancer, and reduced fertility.

It calculated the degree of use of ethanol-containing hand gel at which a person would absorb enough of the chemical through the skin to exceed the legal limit value that Dutch law stipulates for employees: 260mg/m³ per day.

For the risk assessment, RIVM assumed that hand gel contains 83.7% ethanol and that 3 milliliters are applied to the hands at a time. The institute then calculated exposure based on different frequencies of use during one, two or 40 years.

It also considered the amount of ethanol that might be inhaled when it evaporates on the skin.

The total amount of ethanol that people come into contact with over a longer period of time is so low that it does not cause colon cancer or reduced fertility, the institute concluded in its report, *Health risk assessment of ethanol-containing hand sanitizer* (<https://www.rivm.nl/publicaties/beoordeling-van-gezondheidsrisicos-bij-gebruik-van-ethanol-bevattende-handgel>).

The risk of breast cancer increases very slightly. Women who use hand sanitizer 10 times a day for one year have a 143,006 in 1,000,000 chance of developing breast cancer during their lifetime, RIVM found.

In comparison, women who do not use ethanol-containing hand sanitizers have about a 143,000 in 1,000,000 chance of developing breast cancer in their lifetime.

Employees who come into frequent contact with hand gels professionally, such as in healthcare, have a slightly higher risk of breast cancer than consumers, according to the institute.

Using ethanol hand gel 25 times a day for a year, for example, constitutes a 143,015 in 1,000,000 chance of developing breast cancer.

The legal limit value of 260mg/m³ per day is reached when an adult employee uses ethanol-containing hand gel 32 times a day, according to RIVM's research.

NEW YORK, CALIFORNIA ISSUE DRAFT TEST PERFORMANCE CRITERIA FOR 1,4-DIOXANE

Regulators in New York and California have issued draft method performance criteria (MPC) to test for 1,4-dioxane that could inform regulatory actions in both states as they seek to address the chemical's presence in various consumer products.

New York's Department of Environmental Conservation (DEC) is currently working on regulations to implement increasingly stringent limits on the chemical in cosmetic and cleansing products, from 2023.

The "DEC is considering whether to include the draft MPC in forthcoming regulations," the agency said on June 23, 2021. The MPC could "provide guidance on what test methods are acceptable for manufacturers to use to determine whether their products comply with the law".

Limits under the New York law could pose challenges for many cleaning product makers, as 1,4-dioxane can appear as a by-product of the ethoxylation reaction used to make surfactants (see page 13). And many product manufacturers have voiced concerns about their ability to comply with the law.

The allowable amount in cleaning products in New York will be capped at 2 parts per million (ppm) by the start of 2023, and just 1ppm by 2024. Cosmetics will see a permissible level of 10ppm from 2023.

In California, the Department of Toxic Substances Control (DTSC) has been considering a priority product listing for beauty, personal care and hygiene products that contain 1,4-dioxane since early 2019.

On 23 June, in the joint announcement on the draft MPC, the DTSC said it expects to release a product-chemical profile for the substance “later this year”.

A finalized priority designation under the state’s Safer Consumer Products (SCP) program would oblige personal care and hygiene product manufacturers to conduct an alternatives analysis. The DTSC could then proceed with regulations if it determines they are necessary.

The department was clear about its plans to utilize the MPC in its actions. “These criteria will be incorporated into any regulations that DTSC pursues related to 1,4-dioxane as a part of larger guidance on the alternatives analysis threshold (AAT),” it said.

As of press time, comments on the draft MPC were being accepted until July 23. Both the DTSC and New York’s DEC will coordinate efforts to review and incorporate feedback they receive on the draft criteria.

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AOCS MEETING WATCH

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

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

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

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AOCS 

Meet Raj Shah

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



Raj Shah at the Parthenon in Athens, Greece

Fast facts

Name	Raj Shah
Joined AOCS	1995
Education	Ph.D. in chemical engineering from The Pennsylvania State University (State College, Pennsylvania, USA)
Job title	Director
Employer	Koehler Instrument Co.
Current AOCS involvement	Member, <i>Inform</i> Editorial Advisory Committee

PROFESSIONAL

What's a typical day like for you?

In short: exciting and unpredictable. I may interact with customers, put out fires, or solve internal and external issues, and it is always fun and fulfilling. I love the satisfaction of solving a customer's problem; it is difficult to put that feeling into words.

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

I grew up in the suburbs of Mumbai, and my dad was active in the chemical industry. I always wanted to be a chemical engineer. I still remember how excited I was when I was accepted at the Institute of Chemical Technology, which has an acceptance rate of 0.06%.

Why did you decide to do the work you are doing now?

In graduate school, I was part of a research group headed by Professor Elmer Klaus, a world-renowned petroleum engineer and tribologist. We were fortunate to be exposed to industry on an almost daily basis. In fact, I worked on projects for eight different multinational corporations before receiving my degree.

Is there an achievement or contribution you are most proud of? Why?

I have been fortunate enough to work with several mentors. I am currently working with Stony Brook and Hofstra universities, and as a result, Koehler has a number of interns working

at our organization. Watching them grow and learn about our industry gives me great joy. I feel as if I am playing a small but important role in teaching the next generation.

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

I met my wife, Dr. Niloufar Faridi, at Penn State, when we were graduate students. We had our son, Kian, 16 years ago. Kian is a special-needs child who has autism and is nonverbal. Raising him has been a blessing. He has taught me patience and gratitude—both lessons I really needed to learn.

PERSONAL

How do you relax after a hard day of work?

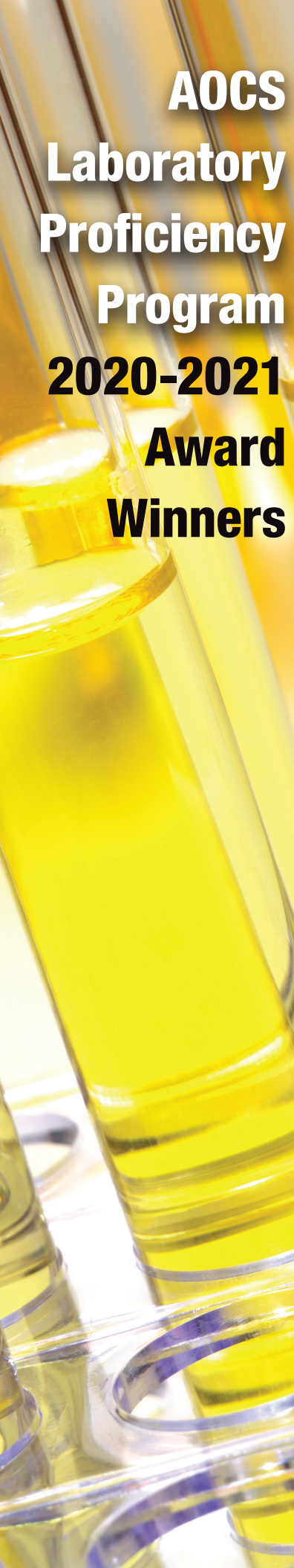
I like to be on the water. When weather permits, Kian and I are on our tandem kayak.

What skill would you like to master?

I would love to play better golf (I make zero effort) and be fluent in Spanish. Both are works in progress. I speak seven languages but learned them all when I was younger. It is much more difficult to learn one at my current age.

What are some small things that make your day better?

Accomplishing what I set out to do at work for the day before I leave the office, interacting with my son, reading a funny one-liner, listening to a soothing piece of music, or tasting a uniquely crafted concoction: All of these bring a smile to my face.



AOCS Laboratory Proficiency Program 2020-2021 Award Winners

Aflatoxin in Corn Meal

First Place

Joe Barney
USDA
Kansas City, MO 64153 USA

Aflatoxin in Corn Meal Test Kit

First Place

Tina Harrell
JLA USA
Edenton, NC 27932 USA

Honorable Mention

Frank Hahn
Hahn Laboratories, Inc.
Columbia, SC 29202 USA
Cindy McCormick & Ashli Brown
Office of the Texas State Chemist
College Station, TX 77843 USA

Aflatoxin in Peanut Paste

First Place

Marisel Corelli
JLA Argentina SA
General Cabrera CD X5809 BAS
Argentina

Honorable Mention

Fabricio Lopresti
SGS Argentina SA
Florencio Varela 1888 Argentina
Juan Regnicoli
SGS Argentina SA
Villa Mercedes San Luis 5730
Argentina
Zhang Peng
JLA China, Inc.
Qingdao Shandong Province 266000
China
Carolina Casagrande Bedani
JLA Brasil Laboratorio de Analise de
Alimentos SA
Marilia SP 17512-120 Brazil

Aflatoxin in Peanut Paste Test Kit

First Place

JLA Dawson Analytical Team
JLA USA
Dawson, GA 39842 USA

Honorable Mention

Judy Thomas
JLA brownfield
Brownfield, TX 79316 USA
JLA Colquitt Analytical Team
JLA
Colquitt, GA 39837 USA
Sylvester Analytical Team
JLA Sylvester
Sylvester, GA 31791 USA
JLA Albany Analytical Team
JLA USA
Albany, GA 31721 USA

Aflatoxin in Pistachio and Almond

First Place

Alina Hernandez
IEH Labs and Consulting Group
Fresno, CA 93727 USA

Honorable Mention

Laura Reina
IEH Kings Chemistry
Lost Hills, CA 93249 USA

Cholesterol

First Place

Imprang Chutiyawawat
Ligand Scientific
Northaburi 1100 Thailand

DDGS from Corn Meal

First Place

Dennis Hogan
SDK laboratories
Hutchinson, KS 67501 USA

Honorable Mention

Anders Thomsen
Eurofins Nutrition Analysis Center
Des Moines, IA 50321 USA

Edible Fat

First Place

James Houghton
AAK USA
Louisville, KY 40208 USA

Honorable Mention

Jerry Buttell
AGP
Hastings, NE 68901 USA
Konni Shipman
AGP
Hastings, NE 68901 USA
Eddie L. Baldwin, Helen Cianciolo,
Derek Gum
Stratas Foods RDI Center
Bartlett, TN 38133 USA
Felicia Melendez
AGP
Hastings, NE 68901 USA
Travis Patterson
AGP
Hastings, NE 38133 USA

Fish Meal

First Place

Pete Cartwright
New Jersey Feed Lab, Inc.
Ewing, NJ 08638 USA

Honorable Mention

Paul Thionville/Andre Thionville/Kris-
topher Williams
Thionville Labs LLC
New Orleans, LA 70123 USA

Gas Chromatography

First Place

Oilseed Lab
Canadian Grain Commission
Winnipeg MB R3C 3G8 Canada

Honorable Mention

Jamie Ayton
NSW Dept. of Primary Industries
Wagga Wagga NSW 2650 Australia
Claudia Guillaume
Modern Olives
Lara VIC 3212 Australia
Zachary Martin
Darling Ingredients
Ankeny, IA 50021 USA
QA Team
Viterro USA LLC
Warden, WA 98857 USA
Sofrida Sofrida
PT Musim Mas
Utera 20371 Indonesia
Heather Compton
Stratas Foods
Quincy, IL 62305 USA
Razmah Ghazali
Malaysian Palm Oil Board AOTD
Kajang Selangor 43000 Malaysia

GOED Nutraceutical Oils

First Place

Edith Von Kries
BASF Personal Care & Nutrition
Illertissen 89257 Germany

Honorable Mention

QC Laboratory
Helene Jehanno, Eric Le Naour
Polaris
La Foret Fouesnant 29940 France
Laboratory
KD Norway AS
Brattvag 6270 Norway
Henriette Meiser-Zessner
KD Pharma Bexbach GmbH
Bexbach 66444 Germany

Marine Oil

First Place

Otelia Robertson
Omega Protein, Inc.
Reedville, VA 22539 USA

Honorable Mention

Lisette Van Schie
TLR International Laboratories
Rotterdam 3077 Netherlands
Matthew Rahn
Omega Protein Inc.
Reedville, VA 22539 USA

Marine Oil Fatty Acid Profile

First Place

Pete Cartwright
New Jersey Feed Lab, Inc.
Ewing, NJ 08638 USA

Honorable Mention

QC Laboratory
Helene Jehanno, Eric Le Naour
Polaris
La Foret Fouesnant 29940 France
Dana Walkenhorst, Steve Clotter,
Dustin Le
Eurofins Central Analytical Labs
New Orleans, LA 70122 USA

NIOP Fats and Oils

First Place

Melanie Greer
Dallas Group of America, Inc.
Jeffersonville, IN 47130 USA

Honorable Mention

George Hicks
Dallas Group of America, Inc.
Jeffersonville, IN 47130 USA

Nutritional Labeling

First Place

Helene Lachance
Trouw Nutrition Canada, Inc
Saint Hyacinthe QC J2R 1S5
Canada

Honorable Mention

Anders Thomsen
Eurofins Nutrition Analysis Center
Des Moines, IA 50321 USA

Oilseed Meal

First Place

Tyler Hack
Amspec
Webster, TX 77589 USA

Honorable Mention

Mumtaz Haider
Amspec
Webster, TX 77589 USA
Trevor Meredith
Solbar Food Technologies
Ashdod 7712102 Israel
Lidieth Solara Carranza
INOLASA
Barranca Puntarenas 6651-1000
Costa Rica
Robert Carr
RMG-PNW
Portland, OR 97218 USA
Jennie Stewart/Brad Beavers
Carolina Analytical Services LLC
Bear Creek, NC 27207 USA

Olive Oil Part A

First Place

Vera Chen
Catania Oils Inc.
Ayer, MA 01432 USA

Honorable Mention

Ryan Drazenovic
Pompeian Inc.
Baltimore, MD 21224 USA
Alex Vargo
Pompeian Inc.
Baltimore, MD USA

Olive Oil Part B

First Place

Vera Chen
Catania Oils, Inc.
Ayer, MA 01432 USA

Honorable Mention

Gwendolyn Truong
Sunset Olive Oil
Montebello, CA 90640 USA

Zhennian Huang
Catania Oils, Inc.
Ayer, MA 01432 USA

Olive Oil Part C

First Place

Vera Chen
Catania Oils, Inc.
Ayer, MA 01432 USA

Honorable Mention

Alex Vargo
Pompeian, Inc.
Baltimore, MD 21224
Ryan Drazenovic
Pompeian, Inc.
Baltimore, MD 21224 USA

Palm Oil

First Place

Tiam Huat Goh
PT Musim Mas
Medan Utera 20371 USA

Honorable Mention

Lin Hendrik
PT Musim Mas Martubung
Medan Sumatera, Utera 20244
Indonesia
Bee Suan Tan
Southern Edible Oils Industries Sdn
Bhd
Kapar Selangor 42200 Malaysia
Kah Soon Ng
PGE0 Edible Oils Sdn Bhd
Pasir Guidang Johor 81700 Ma-
laysia

Peanut

First Place

Tina Harrell
JLA USA
Edenton, NC 27932 USA

Honorable Mention

Fran Fletcher
IEH - Douglas
Douglas, GA 31535 USA

Phosphorus in Oil

First Place

Angie Johnson
Keyleaf
Saskatoon, SK SN 2R4 Canada

Honorable Mention

QA Team
Viterra USA LLC
Warden, WA 98857-0010 USA

Solid Fat Content by NMR

First Place

Eddie L. Baldwin, Helen Cianciolo,
Derek Gum
Stratas Foods RDI Center
Bartlett, TN 38113 USA

Honorable Mention

Ricardo Arevalo Bravo
Grupo Industrial Numar SA
San Jose Costa Rica

Soybean

First Place

Tyler Hack
Amspec
Webster, TX 77598 USA

Honorable Mention

Renato Ramos
Admiral Testing Services, Inc.
Luling, LA 70070 USA
Marvin Boyd, Jr., Luis Robles, Evan
Melancon
Eurofins Central Analytical Labs
New Orleans, LA 70122 USA

Soybean Oil

First Place

Jill Cecil
Owensboro Grain Edible Oils
Owensboro, KY 42302 USA

Honorable Mention

Scott Schuldt
ATC Scientific
North Little Rock, AR 72114 USA

Specialty Oils

First Place

QA Team
Viterra USA LLC
Warden, WA 98857 USA

Honorable Mention

Anders Thomsen
Eurofins Nutrition Analysis Center
Des Moines, IA 50321 USA
Thomas Mawhinney
University of Missouri
Columbia, MO 65211 USA

Tallow and Grease

First Place

Kester Emefiena
Amspec LLC
Webster, TX 77598 USA

Honorable Mention

Zachary Dewilde
Sanimax
Green Bay, WI 54303 USA
Francois Leveille
Sanimax
Montreal QC H1C 1G2 Canada
Tony Mendez
JST Global LLC
Houston, TX 77011 USA

Trace Metals in Oil

First Place

QA Team
Viterra USA LLC
Warden, WA 98857 USA

Honorable Mention

Quality Assurance Team
Richardson Oilseed LLC
Lethbridge AB T1H 9P5 Canada

Jack Riley, William House
Eurofins Central Analytical Labs
New Orleans, LA 70122 USA
Eric Garand
Viterra USA LLC
Ste. Agathe MB R0G 1Y0 Canada

trans Fatty Acid Content

First Place (tie)

Jocelyn Alfieri
Silliker Canada Co.
Markham, ON L3R 5V5 Canada
Anders Thomsen
Eurofins Nutrition Analysis Center
Columbia, MO 65211 USA

Honorable Mention

Heather Compton
Stratas Foods
Quincy, IL 62305 USA
Jill Cecil
Owensboro Grains Edible Oils
Owensboro, KY 42302 USA

Unground Soybean Meal

First Place (tie)

Zhang Xianmei
Bunge (Nanjing) Grains & Oils Co.
Ltd
Nanjing City 210038 China
Ma Yinxia
Bunge Chia Tai (Tianjin) Grain &
Oilseeds Ltd
Tianjin City 300457 China

Honorable Mention

Jun Zhang
Bunge Taixing Grains and Oils Ltd.
Jiangsu 225404 China
Wang Qian
Bunge Rizhao
Shandong 276826 China
Augustin Rodriguez Arguello
Proteinas Naturales SA De CV
Guadalupe NL 67130 Mexico
Jennie Stewart/Brad Beavers
Carolina Analytical Services LLC
Bear Creek, NC 27207 USA
Quality Assurance Lab
Riceland Foods Inc.
Stuttgart, AR 72160 USA
Paul Thionville/Andre Thionville/
Kristopher Williams
Thionville Labs LLC
New Orleans, LA 70123 USA

Vegetable Oil for Color Only

First Place

Hamilton Plant
Bunge Canada
Hamilton, ON L8L 8G7 Canada

PATENTS

Method for manufacturing biofuel

Tachibana, T., Earthrecycle Co., Ltd., US10934567, March 2, 2021

A method for manufacturing a biofuel includes; a step of adding an alkaline agent to a culture fluid in which an algae is cultured and infusing medium quality oil thereto, thereby generating and floating an emulsion; a step of evaporating moisture from the floated emulsion in a vacuum, thereby obtaining a fat and oil component and a solids of the algae and medium quality oil and filtering, thereby separating the fat and oil component of the algae, the medium quality oil and the solids; a step of fractionally distilling the separated fat and oil component of the algae and the separated medium quality oil, thermally decomposing the fractionally-distilled components by adding an alkaline agent thereto, thereby obtaining a pyrolysis oil and a pyrolysis residue, and fractionally distilling the pyrolysis oil into thermally-decomposed gas, light crude oil, medium quality oil and heavy quality oil; and a step of removing an impurity from the fractionally-distilled medium quality oil by means of solvent extraction, thereby obtaining a biodiesel fuel oil.

Process for making flavor and fragrant compounds

Bruhlmann, F., *et al.*, Firmenich SA, US10941419, March 9, 2021

The present invention relates to the production of aromatic hexanols and compositions containing them. In particular provided herein are methods of producing hexenols comprising: a) contacting a hydroperoxide of a polyunsaturated fatty acid with a modified hydroperoxide lyase to form a hexenal; and b) reducing the hexenal to a hexenol in the presence of a hydride donor, a ketoreductase, and a co-factor wherein the contacting and reducing steps are carried out at essentially the same time in the substantial absence of baker's yeast.

Process for the preparation of glycerol carbonate

Coleman, F., *et al.*, The Queen's University of Belfast, US10947211, March 16, 2021

This invention relates to a process for the preparation of glycerol carbonate from the reaction of glycerol and a dialkylcarbonate, for example dimethyl carbonate, or a cyclic alkylene carbonate. More specifically, the invention relates to a process where the synthesis of glycerol carbonate is conducted in the presence of a homogeneous transesterification catalyst and involves the partial reaction of a glycerol reactant stream and a dialkyl carbonate or cyclic alkylene carbonate reactant stream and an intermediate step of alcohol by-product separation before further reaction to improve glycerol conversion and glycerol carbonate selectivity and yield.

Stabilization of cosmetic compositions comprising fish oils and hydroxylated fatty acids and/or its derivatives

Tomashevskaja, M., *et al.*, Conopco, Inc., US10945945, March 16, 2021

Stabilized skin care compositions are described. The compositions comprise a fish oil component that yields a product of oxidation and the component is stabilized with a radical scavenger, a peroxide decomposer and hydroxylated fatty acid and/or a derivative thereof.

Process for removing phospholipids and off-flavors from proteins and resulting protein product

Damodaran, S., *et al.*, Wisconsin Alumni Research Foundation, US10945448, March 16, 2021

Described are methods of removing phospholipids and other off-flavor-causing compounds from edible proteins using a cyclodextrin treatment. The methods include treating soy protein with cyclodextrins such as beta-cyclodextrin to form cyclodextrin-compound complexes and then separating the resulting complexes from the protein. Optionally, prior to treating the protein with cyclodextrin, the protein is sonicated and then treated with a phospholipase, such as phospholipase A.sub.2. Versions of the methods described herein are capable of removing more than 99% of phospholipids from soy protein.

Shampoo composition containing a gel network

Brown, M.A., *et al.*, The Procter & Gamble Company, US10945935, March 16, 2021

A shampoo composition and a method of making a shampoo composition which delivers both good conditioning benefits and good lather performance. The shampoo composition comprises a dispersed gel network phase comprising: from about 2.8 weight % to about 8 wt % of one or more fatty alcohols; at least 0.01% of one or more secondary surfactants, wherein the secondary surfactant comprises sodium laureth-n sulfate wherein n is from about 0 to about 5; and water; and from about 5% to about 50% of a deterative surfactant; from about 0.02% to about 1.50% of a material selected from the group consisting of structurants, suspending agents and mixtures thereof, from about 0.5 to about 1% of a cationic deposition polymer; and at least 20% of an aqueous carrier.

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



Search Engine Optimization (SEO) for your article

More than 50% of traffic to Wiley Online Library, home of AOCS' three journals—*Journal of the American Oil Chemists' Society* (JAOCS), *Lipids*, and *Journal of Surfactants and Detergents* (JSD)—comes directly from Google, Google Scholar, and other search engines. Wiley does everything possible to ensure that all research content is visible and high ranking in the search results of Google and other engines (Fig. 1).

As an author, you can also play a crucial role in optimizing the search results for your article by helping people to find, read, and cite your work.

5 TIPS FOR INCREASING YOUR ARTICLE'S SEARCH ENGINE DISCOVERABILITY

1. Create a search engine friendly title.

- **Include 1–2 keywords related to your topic.**
Place your keywords within the first 65 characters of your title.
- **Keep your title short.**
Consider moving a phrase from your title to the first or second sentence of your abstract.

2. Optimize your abstract.

- **Place essential findings and keywords in the first two sentences of your abstract.**
Only the first two sentences normally display in search engine results.
- **Repeat your keywords 3–6 times.**
Don't forget the purpose of your abstract is to express the key points of your research, clearly, and concisely.

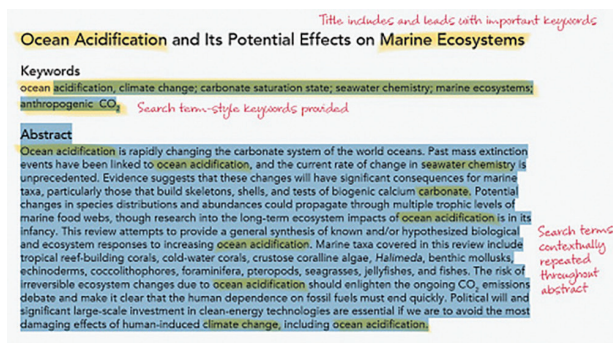


FIG. 2. Example of a well-optimized abstract.

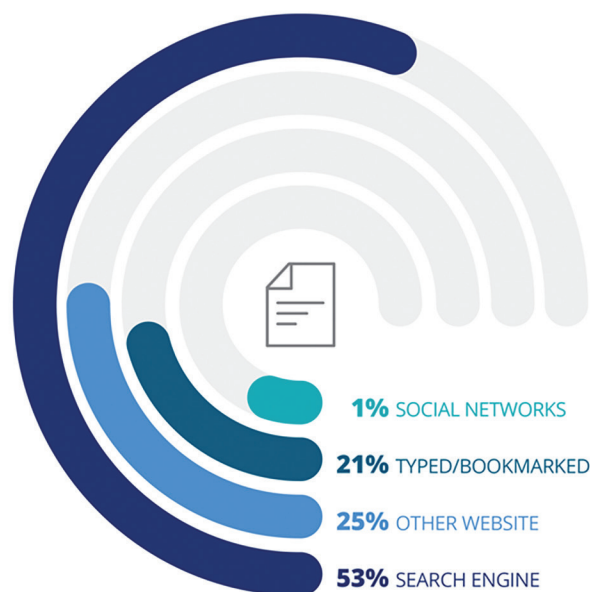


FIG. 1. Search Engine Optimization (SEO) pie chart

3. Use keywords throughout your article.

- **Include keywords in your title (1–2), abstract (2–3), and keyword fields (5–7)**
Keywords may be keyword phrases rather than just single words.
- **Incorporate keywords in your headings, too.**
Headings tip off search engines to the structure and content of your article.
- **Find specific keywords on Google Trends (<https://trends.google.com/trends/?geo=US>) and Google Adwords (<https://ads.google.com/home/#!/>) keyword tools.**
Remember that keywords are important for A&I services as well as SEO.
- **Use keywords consistent with your field.**
If you're unsure, check the words used in your field's major papers.
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Search engines dislike too much keyword repetition, known as keyword stuffing, and may "un-index" your article, making it hard to find online.

4. Be consistent

- **Refer to author names and initials in a consistent manner throughout the paper.**
Remember to be consistent with any previous online publications.

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H&N Health and Nutrition	IOP Industrial Oil Products
PRO Processing	PCP Protein and Co-Products
S&D Surfactants and Detergents	

Review Articles

EAT **LOQ** Encapsulation of bioactive compounds from fruit and vegetable by-products for food application—a review

Marcillo-Parra, V., *et al.*, *Trends Food Sci. Technol.*, In Press, July 7, 2021, <https://doi.org/10.1016/j.tifs.2021.07.009>.

Encapsulation protects bioactive compounds against the adverse conditions inherent in food processing. This review describes the creation of encapsulated bioactive compounds (EBC) using microencapsulation techniques and their applications and identifies previous studies involving the extraction, characterization, encapsulation, and application of bioactive compounds from fruit and vegetable products (FVBP). Spray-drying, freeze-drying, and coacervation were the technologies most used to produce EBC from FVBP with high encapsulation efficiency, excellent functional properties, and improved stability and solubility of bioactive compounds. EBC from FVBP have been added to food for a variety of purposes, including enrichment, fortification, coloring, and improved stability against oxidation and microbial proliferation.

BIO **IOP** **PRO** Biodiesel from oil produced in vegetative tissues of biomass—a review

Singh, R., *et al.*, *Bioresour. Technol.* 326: 124772, 2021, <https://doi.org/10.1016/j.biortech.2021.124772>.

Biodiesel is a green, renewable alternative to petroleum-derived diesel. However, using vegetable oil for biodiesel produc-

tion significantly challenges food security. Progress in metabolic engineering, understanding of lipid biosynthesis, and storage have enabled engineering of vegetative tissues of plants such as sugarcane, sorghum, and tobacco for lipid production. Such sources could be cultivated on land resources, which are currently not suitable for row crops. Besides achieving significant lipid accumulation, it is imperative to maintain the fatty acid and lipid profile ideal for biodiesel production and engine performance. In this study, genetic modifications used to induce lipid accumulation in transgenic crops and proposed strategies for efficient recovery of oil from them have been presented. This paper highlights that lipids sourced from vegetative biomass in their native form would pose significant challenges in biodiesel production. Therefore, different strategies have been presented for improving feedstock quality to achieve high-quality biodiesel production.

H&N The neuroprotective effects of polyphenols, their role in innate immunity, and the interplay with the microbiota

Annunziata, G., *et al.*, *Neurosci. Biobehav. Rev.* 128: 437–453, 2021, <https://doi.org/10.1016/j.neubiorev.2021.07.004>.

Neurodegenerative disorders, particularly in the elderly population, represent one of the most pressing social and health-care problems in the world. Besides the well-established role of both oxidative stress and inflammation, alterations of the immune response have been found to be closely linked to the development of neurodegenerative diseases. Interestingly, various scientific evidence reported that an altered gut microbiota composition may contribute to the development of neuroinflammatory disorders. This led to the proposed concept of the gut-brain-immune axis. In this scenario, polyphenols play a pivotal role due to their ability to exert neuroprotective, immunomodulatory, and microbiota-remodeling activities. This review summarizes the available literature to provide scientific evidence regarding these neuroprotective and immunomodulatory effects and the interaction with gut microbiota of polyphenols and the main signaling pathways involved that can explain their potential therapeutic application in neurodegenerative diseases.

IOP **PRO** Oilseeds to biodiesel and renewable jet fuel: an overview of feedstock production, logistics, and conversion

Khanal, A. and A. Shaw, *Biofuel. Bioprod. Bior.* 15: 913–930, 2021, <https://doi.org/10.1002/bbb.2198>.

Diesel and jet fuel contribute to ~22–27% and ~8–13% of the total energy used in the US transportation sector, producing ~25% and ~9% of the total greenhouse gas (GHG) emissions from this sector, respectively. Biobased alternatives, such as biodiesel and renewable jet fuel (RJF) produced from oilseeds, have lower GHG emissions than their petroleum counterparts, are renew-

able in nature, and support energy security. Thus, the objective of this review was to analyze the information on different oilseed types and characteristics along with their production, harvest, and post-harvest operations; oilseeds conversion to biodiesel and RJF along with their properties and uses; and their cost and environmental status in the United States. More than 80% of the feedstock currently used for biodiesel production in the United States consists of edible oilseeds, including soybean, corn, and canola. Carinata and pennycress are inedible oilseeds that are promising feedstocks for biodiesel and RJF production. The biodiesel and RJF produced from oilseeds have similar density, calorific value, and cetane number while having lower acid value and sulfur content compared to their petroleum counterparts. At the current state of technology, the retail prices for biodiesel (B20) are 1.0–1.4 times, and RJF are 3–4 times higher than their petroleum counterparts. However, GHG emissions of oilseed-based biodiesel and RJF are 37–92% and 32–121% lower than their petroleum counterparts, respectively. The economic competitiveness of oilseed-based bio-fuels production and use could be improved by enhancing oilseed traits and optimizing field operations, which would further reduce the emissions from the transportation sector.

PRO EAT IOP Fruit seeds and their oils as promising sources of value-added lipids from agro-industrial byproducts: oil content, lipid composition, lipid analysis, biological activity, and potential biotechnological applications

Alves, E., *et al.*, *Crit. Rev. Food Sci. Nutr.* 61: 1305–1339, 2021, <https://doi.org/10.1080/10408398.2020.1757617>.

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

LOQ H&N Extraction of phenolic compounds and terpenes from *Cannabis sativa* L. by-products: from conventional to intensified processes

Emilie Isidore, *et al.*, *Antioxidants* 10: 942, 2021, <https://doi.org/10.3390/antiox10060942>.

Cannabis sativa L. is a controversial crop due to its high-tetrahydrocannabinol-content varieties; however, the hemp varieties are gaining increased interest. This paper describes (i) the main categories of phenolic compounds (flavonoids, stilbenoids, and lignans) and terpenes (monoterpenes and sesquiterpenes) from *C. sativa* by-products and their biological activities and (ii) the main extraction techniques for their recovery. It not only describes common techniques, such as conventional solvent extraction and hydrodistillation, but also intensification and emerging techniques, such as ultrasound-assisted extraction and supercritical CO₂ extraction. The effect of operating conditions on the yield and composition of these categories of phenolic compounds and terpenes was discussed. A thorough investigation of innovative extraction techniques is indeed crucial for the extraction of phenolic compounds and terpenes from cannabis toward a sustainable industrial valorization of the whole plant.

Original Articles

ANA IOP Ester oils prepared from fully renewable resources and their lubricant base oil properties

Hu, C., *et al.*, *ACS Omega* 6 25: 16343–16355, 2021, <https://doi.org/10.1021/acsomega.1c00808>.

The work reports on the physicochemical and tribological properties of gallate ester oils prepared from fully renewable resources, such as gallic acid and fatty acids. The ester structures were identified by proton nuclear magnetic resonance spectroscopy (¹H NMR), carbon nuclear magnetic resonance spectroscopy (¹³C NMR) and high-resolution mass spectra (HRMS) data. The density at 20°C (*d*₂₀), kinematic viscosity (KV), viscosity index (VI), pour point (PP), flash point (FP), thermal and oxidative stabilities, friction-reducing, and antiwear properties of gallate ester oils were evaluated. The tribological properties of gallate ester oils as lubricants for steel, copper, and aluminum tribo-pairs can be compared with those of the commercially available lubricating oil tris(2-ethylhexyl) trimellitate (Phe-3C₁₈), but their viscosity-temperature characteristics, thermal and oxidative stabilities are better than those of Phe-3C₁₈. More importantly, they have much higher biodegradabilities than Phe-3C₁₈. The study of the lubrication mechanism shows that the physical and/or chemical adsorption film formed by gallate ester molecules between friction pairs is the key factor for them to obtain friction-reducing and antiwear properties.

BIO IOP PRO Techno-economic feasibility analysis of engineered energycane-based biorefinery co-producing biodiesel and ethanol

Kumar, D., *et al.*, *GCB Bioenergy*, Early View July 2021, Open Access, <https://doi.org/10.1111/gcbb.12871>.

High feedstock cost and low oil yields per unit of land from temperate oilseed crops limit the growth of commercial-scale biodiesel production. Recently, highly productive crops, such as sugarcane and energycane, have been engineered to accumulate triacylglycerides (TAGs) that allow the production of far more industrial vegetable oil than previously possible. A proof-of-concept suggests that biodiesel production from engineered energycane will be possible. However, before making efforts for scale-up, it is critical to understand the commercial feasibility and economic competitiveness of this process. This study performs techno-economic analysis of a unique biorefinery processing energy cane to co-produce biodiesel and ethanol. Comprehensive process simulation models were developed for two scenarios: (i) biodiesel from TAGs and ethanol from fermentation of sugars in juice and (ii) biodiesel from TAGs and ethanol from fermentation of sugars in juice and hydrolysis of carbohydrates in bagasse. Based on the target levels, the analysis was performed for energy cane containing 0%, 5%, and 7.7% TAGs (d.b.). The biodiesel from engineered energy cane was found economically viable and competitive to soybean bio-

diesel. Although the capital investment is higher compared to the soybean biodiesel plant, the biodiesel production costs (\$0.66–\$0.9/L) were lower than soybean biodiesel (\$0.91/L). Biorefinery-scenario-1 processing energycane containing 7.7% TAG produces biodiesel with profitability (IRR 7.84) slightly lower than soybean biodiesel (IRR 8.3) but yields five times of biodiesel per unit land and is self-sustainable for energy requirements. The surplus electricity can displace fossil electricity and provide environmental benefits. Monte Carlo simulation indicated that biorefinery is profitable with a 29%–65% probability ($NPV > 0$) which is largely controlled by feedstock composition and biodiesel market price. It is important to note that energy cane can be grown on the marginal rainfed lands in S.E. USA, where soybean would not be viable. Biodiesel from engineered energy cane would therefore be complementary to soy diesel in the United States.

EAT ANA Rheo-NMR to investigate fat crystallization under shear

Rebry, F., *et al.*, *Curr. Res. Nutr. Food Sci.* 4: 414–420, 2021, <https://doi.org/10.1016/j.crfs.2021.05.004>.

It is well known that shear has an effect on fat crystallization. Whereas rheo-NMR has been used to study the impact of shear on the crystallization kinetics in the past, these methods mostly used a simple Teflon mixing shaft inside a sophisticated NMR instrument to apply shear to the sample. However, this method did not enable the determination of rheological parameters. In this work, a



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custom made low-field rheo-NMR device was evaluated, consisting of a commercial rheometer combined with a low-field permanent magnet to enable simultaneous rheological and NMR measurements. Two fats, i.e. partially hardened sunflower oil (PHSO) and soft palm mid fraction (sPMF), were submitted to several rheo-NMR experiments. The results of these experiments clearly indicated that these fats crystallized differently. First, PHSO crystallized faster than sPMF. Moreover, the latter seemed to crystallize in two steps. Initially a weak structure was formed when a low amount of solids was present, but this structure was replaced by a stronger network once more crystals were present. Both fats were studied under stagnant conditions, but also when submitted to low shear rates (1 s⁻¹ and 5 s⁻¹). It was shown that the amount of solids necessary to obtain a viscosity of 10 Pa s was higher when the shear rate was higher. The strength of the formed crystal network at a given percentage of solids was also weaker as the shear rate during crystallization increased. Whereas these experiments were done non-isothermally, it was shown that rheo-NMR can also perfectly be used for isothermal measurements.

EAT S&D The presence of crystalline sugar limits the influence of emulsifiers on cocoa butter crystallization

Simoes, S., *et al.*, *Food Chem.* 346: 128848, 2021, <https://doi.org/10.1016/j.foodchem.2020.128848>.

The effects of 2 wt% emulsifier and crystalline sugar on the isothermal solidification and polymorphic behavior of cocoa butter were evaluated. The emulsifiers investigated were commercial soy lecithin, polyglycerol polyricinoleate (PGPR), citric acid esters of mono- and diacylglycerols (CITREM), and ammonium phosphatides (AP). All emulsifiers accelerated cocoa butter nucleation and growth from the melt, with PGPR showing the smallest enhancement. Lecithin and AP minimally affected the polymorphic form IV-to-V transition contrary to PGPR and CITREM, which both promoted the formation of form V crystals. The presence of sugar alone accelerated cocoa butter solidification while limiting the ability of the emulsifiers to do so. Sugar alone, and in the presence of emulsifier, hindered the polymorphic form IV-to-V transition. This study shows that the effects of emulsifiers on the isothermal crystallization of cocoa butter can be muted in the presence of crystalline sugar, suggesting a complex interplay dependent on emulsifier molecular structure.

EAT LOQ H&N Bee pollen powder as a functional ingredient in frankfurters

Novaković, S., *et al.*, *Meat Sci.* 182: 108621, 2021, <https://doi.org/10.1016/j.meatsci.2021.108621>.

The objective of this study was to determine whether the addition of different pollen powder concentrations (0.0, 0.5, 1.0 and 1.5 g/100 g) to frankfurters had an influence on antioxidant potential and oxidative changes during storage, without detrimental effect on the quality of sausages. After cold storage of frankfurters, signif-

icant ($P < 0.05$) reductions of psychrotrophic bacteria populations were achieved with higher amounts of pollen (1.0 and 1.5 g/100 g). Good antioxidant properties and maintained TBARS values were accomplished by incorporating pollen into the frankfurters. In terms of quality parameters, statistically significant changes were obtained regarding the color, but sensory characteristics of the products were not disturbed. Also, the incorporation of pollen did not cause changes in terms of texture profile analyses of frankfurters. It can be concluded that the natural component, bee pollen powder, can be used as an antioxidant in frankfurter formulations, but further research is needed to estimate whether it can be an adequate replacement for synthetic antioxidants.

H&N PRO EAT IOP The unique crystallization behavior of buffalo milk fat

Pratama, Y., *et al.*, *Cryst. Growth Des.* 21: 2113–2127, 2021, <https://doi.org/10.1021/acs.cgd.0c01543>.

A full understanding of milk fat crystallization is important for the structural development of dairy products such as butter, ice cream, and cheese. The influence of triacylglycerol (TAG) composition on the dynamic of milk fat crystallization and the nanostructure of the formed crystals was investigated using two chemically different types of milk fat, namely buffalo and cow milk fats (BMF; CMF). The TAG composition was determined using liquid chromatography and mass spectrophotometry (LCMS), whereas differential scanning calorimetry (DSC), small- and wide-angle X-ray scattering (SAXS and WAXS) and polarized light microscopy (PLM) were used to characterize the crystallization behavior of the two milk fats. A total of 37 TAG species were identified in both BMF and CMF, but in different proportions. In particular, BMF was found to have a higher amount of low-molecular-weight TAGs in comparison to CMF. This difference in chemical composition explains the different kinetics of polymorphic transformation in the two samples. Specifically, it clarifies the delay in the nucleation of the β' -polymorph in BMF in comparison to CMF. BMF also showed a higher nucleation rate due to its higher proportion of saturated TAGs and higher melting range. Finally, this work presents a novel interpretation of the mechanism of formation of the beta-polymorph (53 angstrom), which has recently become the subject of a vivid debate in milk fat crystallization studies.

IOP PRO Biorefinery methods for extraction of oil and protein from rubber seed

Yang, M., *et al.*, *Bioresour. Bioprocess.* 8: 1–11, 2021, <https://doi.org/10.1186/s40643-021-00386-2>.

Rubber seeds are a by-product of rubber production and are rich in oil and protein. Upgrading of rubber seeds to produce proteins, oils and feedstock can generate additional revenue for rubber production and reduce waste. The present study investigates the effects of different pre-treatments and extraction methods to determine the optimal methods to produce oil and protein from rubber

seed kernels. Mechanical expulsion using a screw press and solvent extraction using n-hexane were employed for oil separation. The highest oil recovery efficiency of 95.12% was obtained using rubber seed meal that was pre-dried at 105°C. The sequential water-alkaline treatment was ideal for achieving high protein recovery while reducing the protein denaturation that can result from high operating temperatures and organic solvent contact. Over 90% of the total protein from rubber seed kernels could be recovered. Separating oil from kernels using hexane followed by protein extraction from the meals by enzymatic treatment provides a suitable method for comprehensive utilization of rubber seeds.

PRO IOP Techno-economic analysis of hydroprocessed renewable jet fuel production from pennycress oilseed

Mousavi-Avval, S.H. and A. Shah, *Renew. Sustain. Energy Rev.* 149: 111340, October 2021, <https://doi.org/10.1016/j.rser.2021.111340>.

Commercial production of jet fuel from biobased feedstocks is still encumbering, mainly due to their high production cost and competition with food resources. Pennycress oilseed is a potential jet fuel feedstock which can be supplied at a lower price compared to similar oilseeds, such as soybean and canola. The objective of this study was to assess the technical feasibility and costs of hydroprocessed renewable jet fuel (HRJ) production from pennycress. The production capacity was considered to be 18.9 million L/yr (5 million gal/yr). The analysis considered pennycress grain handling and conditioning, oil extraction, and conversion to HRJ and byproducts, i.e., LPG, naphtha and green diesel, through hydroprocessing technology; as well as pennycress meal processing as boiler fuel and wastewater treatment. Total investment for establishing the HRJ biorefinery at the selected capacity was estimated to be 90.8 million USD. Minimum selling price (MSP) of HRJ was estimated to be 1.2 USD/L, which was comparable to the MSP of HRJ from similar oilseeds, including soybean and canola. It could also be further reduced by supplying pennycress grain at a lower price, increasing the oil content and increasing the production capacity of the biorefinery. The outcomes of this research would help establish the performance targets needed to reach the economic viability of HRJ production from pennycress at the commercial scale.

PRO IOP Biorefining for olive waste management and efficient bioenergy production

Najafi, E., *et al.*, *Energy Convers. Manag.* 244: 114467, 2021, <https://doi.org/10.1016/j.enconman.2021.114467>.

The potential of olive wastes for development of a multi-product biorefinery was investigated. Different parts of olive wastes, i.e.,

stone, pomace, leaves, and wood, were subjected to liquid hot water, organosolv, and acid-catalyzed organosolv (ACO) pretreatments prior to bioconversion through three different scenarios. The first scenario, i.e., anaerobic digestion of substrates for biogas production, yielded 219.3 m³ biomethane per square hectometer (1 hm²) of olive trees, equated to 247.4 L gasoline. The highest methane production of 103.3 m³ was attributed to liquid hot-water-pretreated wood, and ACO increased methane yield for leaf and stone samples by 200 and 33%, respectively. The second scenario, i.e., fermentation of wastes for bioethanol production, resulted in 295.9 L bioethanol per 1 hm² of olive trees, equivalent to 196.1 L gasoline. Organosolv pretreated wood with 82.9% production yield and 152.5 L bioethanol constitutes this plan's dominant part. The ACO pretreatment improved fermentation yield for pomace and stone samples by 49% and 53%, respectively. The third scenario, included the utilization of olive wastes in bioethanol production, anaerobic digestion of fermentation residues, and lignin separation, resulted in 295.9 L bioethanol, 137.2 m³ biomethane, and 347.1 kg lignin, equated with 521.6 L gasoline. Furthermore, the remaining oil content in pomace and stone samples was 17% and 20%, respectively, which could be used for biodiesel production. Overall, olive wastes processing through an integrated biorefinery plant with multiple products significantly improved the energy recovery of the whole plant.

PRO EAT IOP The enhancement of rice bran oil quality through a novel moderate biorefining process

Li, D., *et al.*, *LWT* 15: 112118, 2021, <https://doi.org/10.1016/j.lwt.2021.112118>.

Edible grade rice bran oil (RBO) rich in γ -oryzanol and phytosterol but without glycidyl esters (GEs) and monochloropropane diol esters (MCPDEs) was obtained from rice bran by moderate biorefining. High-acid RBO (acid value of 51.22 mg KOH/g) was firstly extracted from rice bran and was then moderately biorefined by combining improved enzymatic degumming, dewaxing, enzymatic deacidification, bleaching, and low-temperature purification (120°C). Through monitoring the refining process, results showed that the improved enzymatic degumming by combining partial glyceride lipase SMG1-F278N and phospholipase A1 enabled the phosphorus content to quickly decrease to 4.56 mg/kg after optimization by response surface methodology. After biorefining, the final RBO with the acid and peroxide value of 0.10 mg KOH/g and 2.05 mmol/kg reached the grade-one standard of edible oil. Additionally, the final RBO contained 20.66 mg/kg γ -oryzanol and 20.63 mg/kg phytosterol with the retention rate of 71.67% and 59.2% but with undetectable GEs and MCPDEs. The environmental friendliness of the novel moderate biorefining process and superior product quality of the final product point toward a promising viable process for RBO refining in the further.

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A photograph of a man with dark hair and a beard, wearing a grey sweater, leaning over a young child with blonde curly hair. The child is wearing a blue and white patterned cardigan over a white shirt. They are in a kitchen, looking down at a wooden surface where they are baking. A rolling pin, a stick of butter, and a white bowl are visible on the counter. The background is softly blurred, showing kitchen shelves.

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