INFORM

International News on Fats, Oils, and Related Materials

MORE THAN A YEAR WITH COVID-19

Where do we go from here?

ALSO INSIDE:

Oleofoams

Alternative base oils

Goodbye to caustic washing

AOCS

Ensuring Food Safety

We deliver technical solutions for producing superior products in terms of either taste, appearance, nutritional value, shelf life and functionality.

We lead the way to innovative solutions for minimizing components that present health concerns to consumers.

The experience of 9,000+ industrial references in the Oils & Fats Field.

desmet ballestra

Science behind Technology

www.desmetballestra.com

SOLUTIONS FOR CHEMICAL PROCESSING EXCELLENCE
Lab, Pilot & Large Scale Production Capabilities

Science and industry have looked to Pope Scientific for over 50 years to fulfill demanding separation, purification and mixing applications. In addition to complete turnkey processing equipment manufacturing, we offer toll processing services (customized contract distillation) for cost effectiveness and feasibility testing to demonstrate process/equipment success. **Our product line-up includes:**

Fractional Distillation Systems

- Batch or Continuous Mode
- Featuring Packed Column Technology for Greatest Purity & Yield

Wiped Film Stills

- Molecular (Short-Path) Still & Evaporator Configurations
- Providing Gentlest Separations of Materials High in BP, MW, Heat Sensitivity and Viscosity

Hybrid Wiped Film/ Fractional Stills

- Combining the Multi-Plate Efficiency of Fractional Columns with the Gentle Reboiling Effect of Wiped Film Evaporators for Optimized Purity and Yield

Process Vessels & Systems

- From 0.1 to 5000 Liters
- Special Designs for Reactors, Fermenters, Mixers, Homogenizers, Dispensers, Hydrogenators and Other Turnkey Systems

Nutsche Filter-Dryers

- Benchtop Model is Logical Step Up from Laboratory Buchner Funnel
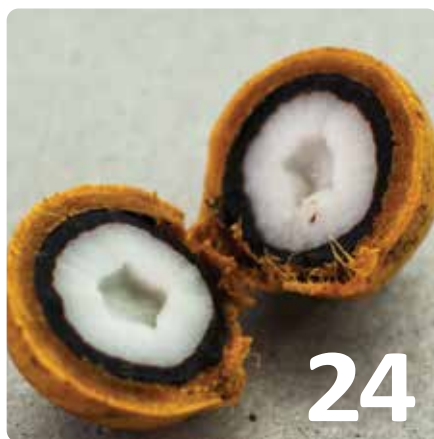- All-in-One Solution for Filtering, Washing, Reslurrying, Mixing & Drying High Purity Solids

INFORM

CONTENTS

DEPARTMENTS

Best In
EDIBLE OIL
FILTRATION

VERTICAL
PRESSURE LEAF FILTER

HORIZONTAL
PRESSURE LEAF FILTER

CANDLE FILTER
BACK WASH TYPE

TUBULAR CENTRIFUGE
FILTER

SINGLE & MULTIBAG
POLISHING FILTER

FILTER
ELEMENTS

Sharplex ®

AOCS MISSION STATEMENT

AOCS advances the science and technology of oils, fats, proteins, surfactants, and related materials, enriching the lives of people everywhere.

EDITOR-IN-CHIEF EMERITUS
James B.M. Rattray

CONTRIBUTING EDITORS
Scott Bloomer   Leslie Kleiner

EDITORIAL ADVISORY COMMITTEE

Julian Barnes	Thu (Nguyen) Landry	Raj Shah
Gijs Calliauw	Gary List	Utkarsh Shah
Etienne Guillocheau	Jill Moser	Ignacio Vieitez
Jerry King	Warren Schmidt	Bryan Yeh

AOCS OFFICERS
PRESIDENT: Doug Bibus, Lipid Technologies LLC, Austin, Minnesota, USA
VICE PRESIDENT: Phil Kerr, SERIO Nutrition Solutions LLC, Dardenne Prairie, Missouri, USA
SECRETARY: Gerard Bailely, Procter & Gamble, Mason, Ohio, USA
TREASURER: Grant Mitchell, Process Plus, LLC, Cincinnati, Ohio, USA
CHIEF EXECUTIVE OFFICER: Patrick Donnelly

AOCS STAFF
MANAGING EDITOR: Kathy Heine
ASSOCIATE EDITOR: Rebecca Guenard
MEMBERSHIP DIRECTOR: Janet Brown

PAGE LAYOUT: Moon Design

The views expressed in contributed and reprinted articles are those of the expert authors and are not official positions of AOCS.

The COVID-19 pandemic, one year later

Rebecca Guenard

When shutdowns began in the spring of 2020, we thought we might have to work from home for a couple of weeks to let the spread of the novel corona virus subside. Avoiding the daily commute was a nice change of pace at first, but then weeks turned into months. As the year dragged on, we realized the myriad things we took for granted in our pre-COVID lives: health, human contact, childcare, collaboration…the list goes on.

- **Throughout the year, both industry and academia found ways to operate as normally as possible despite much of the workforce being sent home.**

- **Virtual interactions kept us safe during the pandemic. Going forward, they will remain a fixture in our lives.**

- **After decades of preparation, certain areas of health care and manufacturing finally became digitized during the pandemic. As long as networks can be kept secure, digitization provides industries with foresight and agility.**

As challenging as life has been over the past year, living through the COVID-19 crisis has led the world to consider new operational modes. Basic necessities dwindled in the early days of the pandemic, causing the general public to be aware of supply chains like never before. We grew conscious of the resources the world shared and of their often singular origin. Technology forever altered our relationship with the workplace as we reliably began conducting meetings with co-workers over the internet. We found out that, although they are preferred, face- to-face interactions are not a requirement for achieving our work objectives. We learned that the opposite is true for school. Technology cannot replace in-person education and the social benefit students gain from being together in the classroom.

"I look forward to having the students on campus in a regular way," says Gianfranco Mazzanti, associate professor at Dalhousie University in Halifax, Canada. "Seeing each other online is not the same. The human contact is so important."

During lockdowns, Mazzanti and his colleagues acted as their students' avatars in the laboratory so students could complete their assignments virtually. Computer programs put decision-making in the students' hands, even if the laboratory equipment was controlled by an avatar. Given the circumstances, it was a way to provide continuity in his students' education. But nothing beats the in-person educational experience, Mazzanti says.

As vaccines are being administered around the world, life will eventually get back to normal. What will normal look like after all we have experienced this past year? Which of the changes we have adopted to get through the pandemic will stick permanently? No one knows for certain, but business experts have a few predictions. Based on current trends, here are some COVID-prompted changes you can expect to continue.
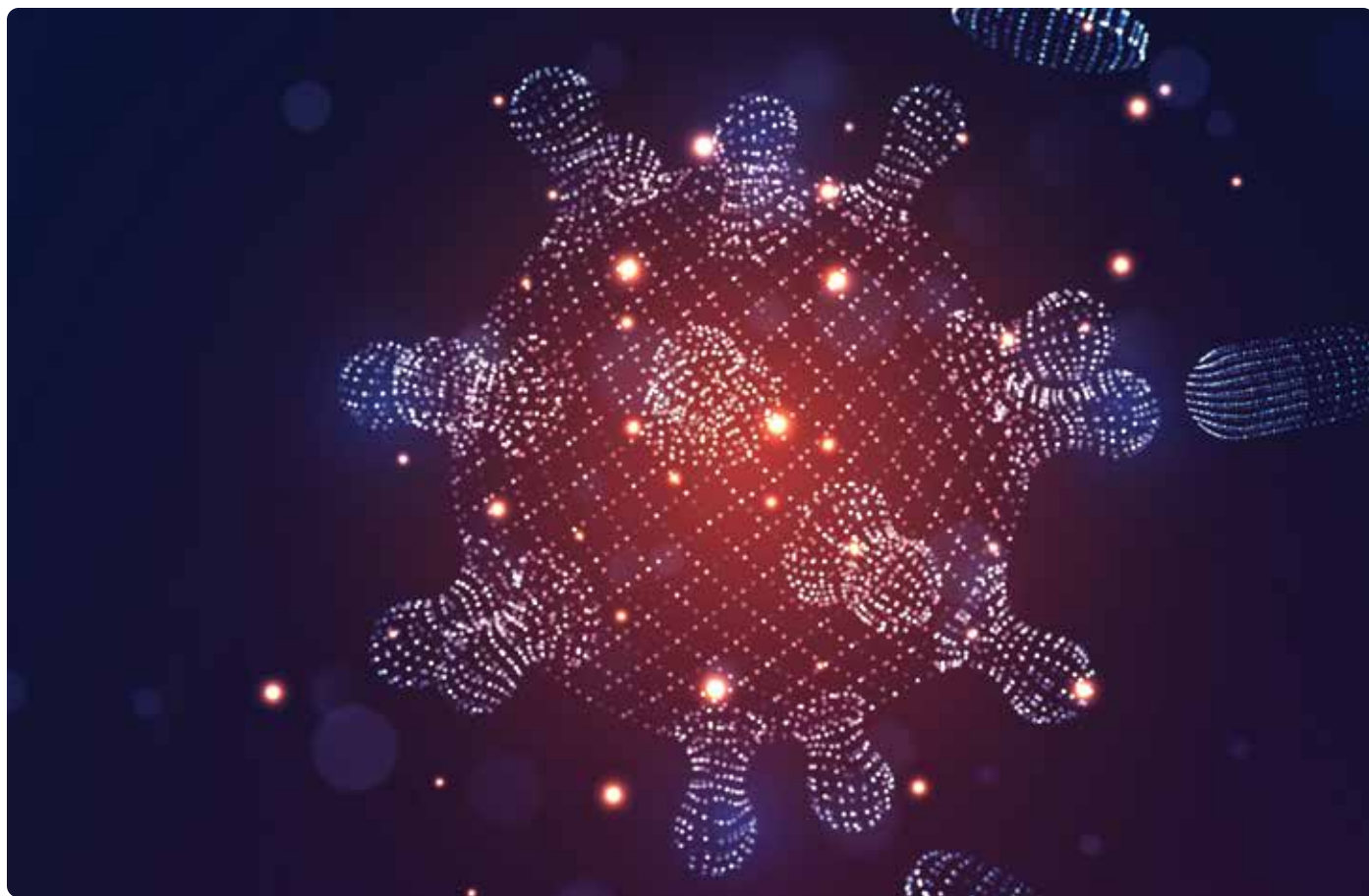
WORK FROM HOME WILL CONTINUE

According to a survey of key executives across a range of industries in the fall of 2020, there will be an 87% increase over pre-pandemic rates for remote working in the next five years (https://tinyurl.com/2021WFHsurvey). When the pandemic forced workers into isolation, employers discovered that home offices were not the productivity stiflers they were once assumed to be. The survey shows that working from home is now viewed more favorably than before the pandemic. Companies report that with commutes and non-essential meetings eliminated they have actually seen an increase in productivity. In addition, employees are capitalizing on the more flexible schedule work from home provides. The CEOs, small business owners, and human resource (HR) managers who participated in the survey responded that nearly a quarter of their staff will continue working from home through 2025 (Table 1).

The work-from-home option has been made possible in the United States, because technology took a leap into the future during the pandemic. Broadband services expanded across the rural United States through an emergency order initiated by its Federal Communications Commission (https://tinyurl.com/COVIDbroadband). Lawmakers in some rural states have been pushing for the expansion for a long time, but with homebound students and employees needing reliable internet connections the Commission granted providers the authority to expand.

Areas of India and some countries in Asia, however, have reported that internet speeds decreased during the pandemic. With such high demand on high-speed internet, worldwide investment in a network infrastructure of broadband fiber has become a necessity—especially since remote plant access increased during the pandemic, a trend that is likely to continue with a return to normalcy.

TABLE. 1. A look at remote working, now and in the future. Over a quarter of the workers at the 1,000 businesses surveyed will continue to work remotely in 2021. Source: Upwork's Future of Workforce Pulse Report: https://www.upwork.com/research/december-2020-future-workforce-pulse-report

What percentage of your team/department was, is, or will be working remotely at the following points?				
	Before COVID-19	**April 2020**	**Today**	**12 months from now**	**Five years from now**
Fully remote	12.3%	47.7%	41.8%	23.7%	22.9%
Partially remote	8.9%	12.2%	15%	15.2%	14.6%
Not remote	78.8%	40.1%	43.3%	58.2%	62.5%

FIG. 1. An image of augmented reality being used for training purposes. During the pandemic, companies like Honeywell implemented this technology as part of their remote plant operations. A remote team can troubleshoot onsite issues using a wearable device that allows them to see the plant in real time. Source: https://www.honeywell.com/us/en/news/2020/03/3-tips-to-run-operations-remotely

MANUFACTURING OPERATIONS WILL BE DIGITIZED

The pandemic accelerated industry toward its digital future. Avoiding a deadly virus meant avoiding other humans. To maintain commercial production and, in some cases the general function of society, robots were called into action. They cleaned and disinfected common use areas in major cities around the world and whipped up salads in employee cafeterias (https://www.chowbotics.com/). Replacing workers with automated systems has historically been challenged by labor unions, who fight to retain jobs for humans. In a pandemic, the unions could not argue against the increased safety automation provides by limiting the number of humans in a work area (https://tinyurl.com/covidspedautomation).

Just as it made us more accepting of robots, COVID-19 removed any hesitancy companies have had to digitize production. When manufacturing plants shut down and sent their workers home to stop the spread of the virus, they were given an opportunity to start back up in a digital realm they had been contemplating for a decade (Digitizing manufacturing: how companies are using data to improve production, *Inform*, November/December 2019, https://tinyurl.com/y38k7rbo). The healthcare sector, especially, embraced the new way of manufacturing to ensure supplies of critical items, like cotton swabs. Remote plant access became a key technology in 2020, along with the implementation of digital twins and virtual and augmented reality (Fig. 1) (https://tinyurl.com/remoteplantaccess).

The Ontario, Canada water authority, for example, operated remotely last year through employee workstations maintained from home, significantly reducing the number of workers needed on site. System control software inside the plant, provided by GE Digital, provided managers and operators access to real-time data. Employees monitored and controlled plant operations from home using a laptop or mobile device over a secure internet connection (https://tinyurl.com/GEautomationsoftware).

According to the American Chemistry Council, the industry lost 14,000 jobs in 2020 (https://tinyurl.com/chemistry-joblosses). Those cuts were due to a slowdown in production by automakers and airplane manufacturers who stopped buying chemicals used to outfit their machines. Food and personal care industries also shrank, reducing demand on specialty chemicals. These industries are expected to rebound, but how humans will integrate with the computers that performed their jobs during the pandemic remains an unanswered question.

Another question arose at the end of 2020: How secure are manufacturing operations that are controlled through cloud networks? As of press time, the United States government discovered 30 government agencies, companies, and think tanks, along with organizations in Canada, Mexico, Belgium, Spain, the United Kingdom, Israel, and the United Arab Emirates, that have been compromised by Russian hackers (https://tinyurl.com/russianhackdiscovered). The implications of this infiltration have not yet come to light, but they are likely to give pause to companies considering digitized manufacturing.

Nevertheless, now that these technologies have been installed, few experts believe they will be abandoned. Digitization is here to stay, and in the next year expect oils and fats companies to address questions of workforce integration and network security. As they do, they can look for exam-

ples from the aviation, finance, and tech industries that have already addressed such issues.

SUPPLY-CHAIN ISSUES WILL BE MORE PREDICTABLE

A year after COVID invaded our lives, we are all still dealing with uncertainty. When will we get the vaccine? When will we go back to working onsite? When will we feel comfortable in a crowd of people again? For supply-chain managers, these feelings are particularly acute. However, the frustration they felt in 2020 has led to the adoption of new technology that will help them reduce uncertainty in 2021.

The pandemic originated in the industrial province of Wuhan, China, shutting down factories before the end of February, 2020. By March, when the rest of the world realized the majority of active pharmaceutical ingredients (APIs) and personal protective equipment (PPE) came from this region, the factory closings had already crippled the supply chain (A global supply chain: What could go wrong? *Inform*, June 2020, https://tinyurl.com/y3v4d2on). While some industries struggled, chemical suppliers were able to shift from the production of less necessary polymers and resins to alcohol and disinfectants that were in high demand (https://tinyurl.com/chemicalindustryroundup).

Forecasting supply-chain stressors has always been a challenge, but the pandemic forced mangers to prioritize cen-tralizing data for all stakeholders (Fig. 2) involved in the logistics of producing and moving products (https://tinyurl.com/COVIDsupplychainchanges). Companies are now compiling supply-chain data and populating dashboards to increase supply chain visibility moving forward. For example, demand for skincare and cosmetics dropped during the pandemic; when sales pick-up again, mangers will have end-to-end data telling them where adjustments are needed to revive production.

This, of course, means more digitalization and connections across networks. Companies can build their own software or purchase from a supplier, like infor (https://www.infor.com), that incorporates the particular needs of the chemical industry. Managers previously understood the agility and forecast capabilities a digital supply chain provides, but COVID-19 propelled health-care, personal care, and chemical industries closer to the reality of functioning in that space.

In addition to digitization, industries are rebalancing their supply chain to include more domestic production. The European Commission outlined plans to reduce its dependance on raw materials produced outside the European Union (https://tinyurl.com/EUCrawmaterialsoutline). A similar executive order was signed by the President of the United States (https://tinyurl.com/increasingdomesticsupply). These legal measures are directed toward raw materials for the technology sector, but since COVID, all industries are looking to relocate at least part of their value chain to the Western hemisphere to maintain their agility should a future crisis arise.

FIG. 2. Since COVID-19, companies are rethinking their global supply chain.

FIG. 3. An image of what conferences will look like in the future. Conference organizers will offer a mix of live and virtual attendance and take advantage of the benefit of being online by capturing the conference content digitally.

CONFERENCES WILL HAVE A NEW FORMAT

Now that many of us have spent the past year interacting virtually with colleagues, we recognize that there is a different atmosphere at an in-person gathering than a virtual one. We hear one another better and read social cues better when we stand side-by-side instead of looking at a head and torso through a screen. Experts predict that this irreplaceable quality of in-person interactions means that we will travel to conferences again in the future, but meeting organizers are eager to retain to the benefits of virtual platforms by designing hybrid events that allow those who cannot travel to participate.

A hybrid conference involves more than a livestream from the conference hall. The next time you attend an in-person conference you might once again see the Zoom tiles we all grew accustomed to when working from home. Blending live and virtual audiences will help offsite participants feel they are truly be part of the meeting by allowing them to ask live questions and interact with onsite participants. (Fig. 3).

Of course, the greatest benefit of a hybrid meeting is capturing and storing the digital content for later reference. Technical discussions no longer must be limited to the time allotted for a specific session. In the digital space, subject matter experts can find their community, trade virtual business cards, and build a resource center. A hybrid model expands the capabilities of in-person only conferences, allowing them to live in a digital space long after the conference space has been dismantled. As COVID vaccinations are administered sporadically around the world, business experts predict this new conference format will become popular in 2021, and continue for years to come.

HEALTH CARE WILL BE VIRTUAL

Health and medicine experienced the greatest changes due to COVID. New vaccines were researched and developed in record time. Although this was an impressive feat, other health-care advancements are likely to have a bigger impact on our day-to-day lives.

Telemedicine proved its value during the pandemic. In the future, workdays will not be interrupted by a trip to the doctor's office (https://tinyurl.com/telemedheretostay). In March 2020, the United Kingdom's National Health Services experienced nearly a 100% increase over the previous year in downloads of its telehealth app and online prescription requests (https://tinyurl.com/UKoutlookontelehealth). While it was initially seen as a means of reducing potential contact with the virus, it also became an asset for expanding access to health

care in the United States. Telemedicine was so successful during the pandemic (especially for mental health care) that major insurance companies like United Health and Anthem have adapted their coverage to include the technology.

Before COVID, telemedicine was seen as little more than an online conversation, but the pandemic has led (once again) to the incorporation of more technology. At-home devices, such as oximeters and blood pressure cuffs, allow physicians to access certain diagnostics and provide the same level of care as an in-person visit. As it grows in popularity, our mobile devices will assist in making it better. Valuable pools of data are created daily as these devices measure and store our steps, heart rate, and oxygen levels. All that information can be stored in the cloud and monitored for irregularities.

Connecting artificial intelligence with telemedicine could help health-care practitioners predict the onset of an ailment or disease. This type of connected care is already being implemented by The US Veterans Administration, which reports that the financial and human success of the program so far ensures future investment. Likewise, digital health companies across the EU are racing to get more diagnostic telehealth devices to market (https://tinyurl.com/futureofmobilehealth).

The primary lesson of COVID-19 is that the digital world we embraced during the crisis will remain central to our lives in the future. Implementing more technology allowed us to carry on with life as usual throughout 2020. In the next five years, that technology will help us work smarter as we access information that was out of reach before digitization compiled it all in one place. With the addition of artificial intelligence, that information will be used to make future predictions that save time and spare budgets by avoiding pitfalls.

We may have felt like our lives were going nowhere over the past year. In reality, we took a huge leap forward.

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

Enzymatic-catalyzed esterification of free fatty acids to methyl esters

M.A. Agerbaek, R.B.Hansen, P.M.Nielsen, A.Rancke-Madsen, and J.M.Woodley

Enzymatic transesterification of fatty acid oils to fatty acid methyl esters (FAME) is a well-established process involving an enzymatically catalyzed transesterification reaction step with yields greater than 97%. The unreacted residual consists primarily of free fatty acids (FFA), which contribute to the acid value (mg KOH/g biodiesel) and are typically 2–3 wt%. Water is critical to the stability and activity of the enzyme employed in the transesterification reaction. Consequently, reducing the residual FFA further is not possible with the existing process.

- **A typical biodiesel process based on transesterification with enzyme as catalyst leaves 2–3% residual free fatty acids that must be separated after saponification.**

- **Enzyme-catalyzed biodiesel can achieve complete conversion in a two-step enzymatic process: a transesterification step followed by an esterification step.**

- **Two alternative processes for the esterification step are possible and cost efficient with a new liquid formulated lipase; one uses flash drying technology, and the other uses a stirred bubble reactor.**

To reduce FFA concentrations below 0.25 wt% (the specification level), a caustic wash is currently employed. This yields soap, which must be separated. As a result, the current process has a significant yield loss, even though the soap-phase can be recovered and re-processed. Therefore, the industry has sought a solution to bypass the caustic wash. We now know that enzymatic esterification of the residual FFA, using *Candida antarctica* Lipase B (CALB), is possible both with an immobilized as well as a liquid formulated enzyme (Fedosov and Xu, 2011; Nielsen, *et al*., 2016; Wancura, *et al*., 2019), although in our view the liquid formulation is preferable. This new development in the esterification process, combined with a significantly more efficient CALB formulation, provides a cost-efficient enzymatic biodiesel process.

PROCESS DESIGNS FOR FAME POLISHING

When liquid formulated enzyme is added directly to the FAME phase stemming from the transesterification reaction, addition of dry glycerol with the enzyme is used to form a liquid-liquid interface upon which the lipase is known to act. The glycerol also helps by reducing the apparent thermodynamic activity of water, yielding a shift in equilibrium toward FAME. This provides a system capable of reducing FFA to levels beneath 0.5 wt%. The most important parameters of this reaction system are the water, enzyme, and glycerol concentrations. Two possible systems for water evaporation have been examined: 1. a stirred bubble enzyme (SBR) reactor with a methanol reservoir serving as a condenser (*Austic and Burton,* 2012) and 2. flash drying and reactions that yield a shift in equilibrium toward formation of FAME.

FIG. 1. Stirred bubble reactor setup in laboratory. A vacuum pump circulates methanol vapor from condenser tank to the reactor. Water from the reactor is brought to the condenser.

Stirred bubble enzyme reactor (SBR) with a methanol reservoir

The laboratory setup can be seen in Figure 1. Methanol vapor is circulated between the enzyme reactor and the condenser by a vacuum pump, transporting the water formed in the reactor to the condenser. This establishes continuous water evaporation in the reaction vessel and, through temperature control, enables FFA concentrations within specification in a single vessel following the transesterification reaction. Data obtained from operating the SBR can be found in Figure 2. FFA is reduced from 3% to < 0.5% in 425 minutes, while methanol in the reactor approaches 2%. (Hansen, *et al.*, 2020)

Flash drying

A single reaction step has been found sufficient for reduction of FFA beneath 0.5 wt% with this process. Data from testing in laboratory can be seen in Figure 3 (page 14), which demonstrates the reduction in FFA from 3.6% to < 0.5% in one step.
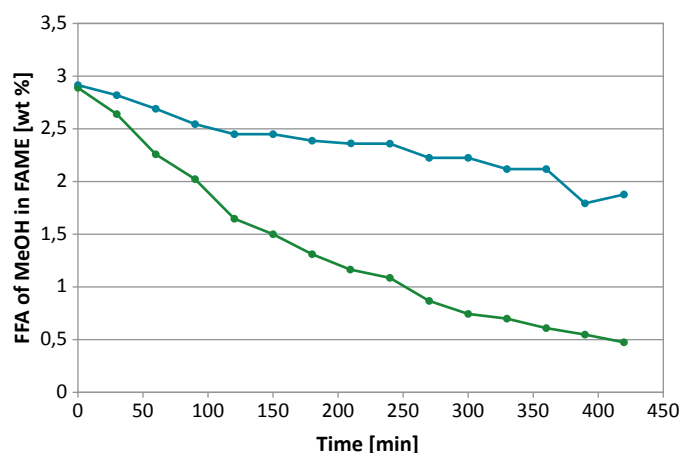
FIG. 2. Simultaneous reaction and water removal using the SBR method. Blue line represents methanol concentration [wt %]; green line represents FFA concentration [wt %] using B100 biodiesel spiked with FFA as feedstock. 4% methanol, 5% glycerol, and 0.455% lipase NS88007 (Liquid CalB lipase). Reactor 40°C, condenser 25°C. (Hansen, *et. al.*, 2020)

IMPLEMENTATION AND OPTIMIZATION

Both processes are designed as additions to the current enzymatic biodiesel process using Eversa Transform. A flowsheet for the process can be seen in Figure 4. There are still optimizations to be done (e.g., temperature, pH, mixing, glycerol and methanol loadings, and enzyme dosage). These are currently being conducted in laboratory- and pilot-plant-scale trials with different qualities of oil feedstock. For example, we are exploring whether these systems could be run at temperatures higher than 40°C. The thermal stability of CALB is highly dependent on the concentration of methanol, and is therefore being examined accordingly.

Process comparison and choice

The SBR process reduces FFA continuously, and control of the temperature difference between the reactor and condenser is important. We believe that some existing equipment used in soap washing could be retrofitted. The SBR method might also be expanded from being a batch process to operate continuously. The productivity of the SBR process is scalable, although energy requirements for mixing and bubbling would increase non-linearly with size. The airflow used in this project (0.8 vvm (volume air/volume reactor/minute) appears realistic when
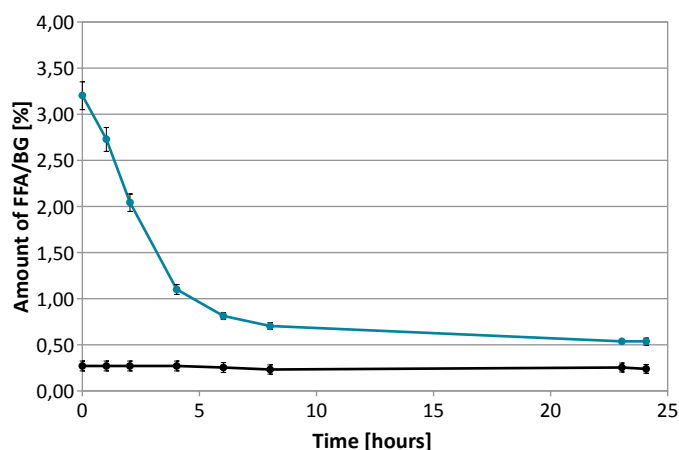
FIG. 3. Reacting a dried FAME with high FFA content. 0.05% NS88007, 10% glycerol, 3% methanol, 30g FAME with 3.2% FFA, 40°C. Blue line: FFA, Black line: Bound glycerin (BG)

compared to fermentation processes. The process is highly robust in terms of conversion, because the only limitation lies in the obtainable water equilibrium between the enzyme reactor and the methanol condenser. Nevertheless, the SBR process uses newer and less-established technology, which could potentially yield practical operational problems.
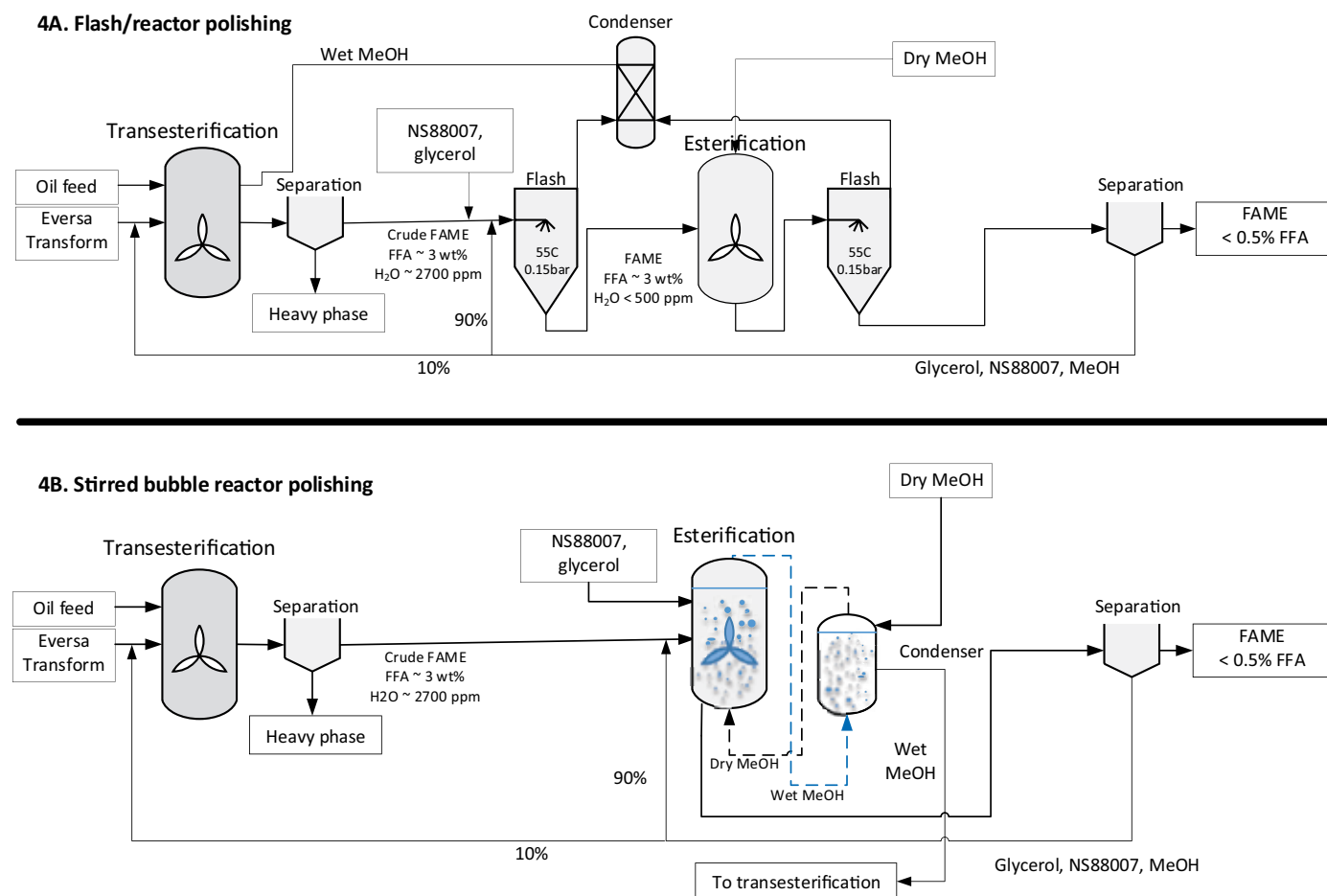
FIG 4. Flow chart of the alternative FFA esterification processes: A) Flash/reactor system, B) Stirred bubble reactor. (Hansen, *et al.*, 2020) Eversa Transform is a lipase for transesterification; NS88007 is lipase for esterification of FFA.

On the other hand, the flash/reactor process uses well-known technology, is scalable, and would fit into any existing Eversa process with minimal difficulty. It can be operated both as batch or continuously, and several degrees of freedom exist for optimizing the proposed process.

IMPACT OF THE CAUSTIC WASH ON PRODUCTIVITY AND PROFITABILITY

Existing plants currently recycle the soap stemming from the caustic wash. When separating the soap from the FAME, a significant portion of the FAME is captured by the soap. When recycling the soap, it is first acidified, yielding a mixture of FFA and FAME, which takes up a significant amount of production capacity. Enzymatic FFA esterification will increase the productivity of the plant and therefore result in a smaller recycle stream or even eliminate it entirely.

ENSURING THE PRODUCT IS WITHIN SPECIFICATION

An important factor is the European specification on FFA concentration of 0.25 wt% (most commonly expressed as 0.5 mg KOH/g FAME). The proposed SBR process has been shown to reach FFA concentrations within specification, as previously described, while alignment with specification has not yet been demonstrated for the flash process. The lowest FFA concentration achieved in the flash process so far was around 0.35 wt%, but this was reached at elevated water concentrations relative to what would be expected industrially. Therefore, it is currently uncertain whether or not a caustic wash will be necessary to ensure FFA concentration within specification when using the flash process. Because the caustic wash is also used for removal of sulphuric compounds and other impurities, such as glycerides, it might be necessary, regardless of the residual FFA concentration. The main goal of the proposed processes therefore is to reduce formation of soap and wastewater production, which will greatly improve the profitability of an enzymatic biodiesel production plant. This has been achieved in both cases and it is now for the engineering companies to determine the most feasible and practical process for commercial use.

REUSABILITY

Most of the enzyme activity was found recyclable through isolation of the glycerol phase. This lowers enzyme costs, addition of water through (wet) enzyme addition, and reduces the costs related to addition of dry glycerol. Reuse is thus a key part of the proposed processes since the economics can benefit greatly. Likewise, in the flash process, reuse of the relatively dry glycerol phase will positively impact the obtainable conversion, because the water concentration at steady-state operation will be lower due to reduced addition of new enzyme product.

COMPARISON BETWEEN CHEMICAL AND ENZYMATIC PRODUCTIVITY

One disadvantage with enzymatic catalysis has been the price of the catalyst compared to the chemical catalyst. Even though enzymatic processing typically requires much larger volumes to obtain adequate productivity, enzyme price has decreased lately. It is also possible to reuse enzyme, and the overall cost contribution from enzyme is such that the processes can now be cost competitive.

TOWARD MORE SUSTAINABLE BIODIESEL PRODUCTION

Both proposed systems for converting residual FFA in crude biodiesel are complete, economically viable, and sustainable processes capable of significantly reducing waste generation and the need for caustic washing, which is the typical method for reducing FFA in enzymatic biodiesel operations today. Both lower soap recovery requirements, which will increase the productivity and yield of existing (and future) biodiesel plants. Hopefully, this will result in a positive shift toward more sustainable biodiesel production.

M.A. Agerbaek and R.B. Hansen are chemical engineering students, and J.M. Woodley is a professor in the Department of Chemical and Biochemical Engineering at the Technical University of Denmark, Lyngby, Denmark.

A. Rancke-Madsen is a science manager, and corresponding author Per Munk Nielsen is a senior science manager for Application Research Oils & Fats, Novozymes, Denmark. Nielsen can be contacted at pmn@novozymes.com.

References

Austic, A., Burton,,R., and Fan,,X. (2012). Fatty acid esterification process, US Patent 9,422,584.

Nielsen, P.M., A. Rancke-Madsen, H.C. Holm, and R. Burton, Production of biodiesel using liquid lipase formulations, *J. Am. Oil Chem. Soc. 93*: 905–910, 2016.

Fedosov, S.N. and X. Xu, Enzymatic synthesis of biodiesel from fatty acids. Kinetics of the reaction measured by fluorescent response of Nile Red, *Biochem. Eng. J. 56*: 172–183, 2011.

Wancura, J.H.C., *et al.*, Improving the soluble lipase–catalyzed biodiesel production through a two-step hydroesterification reaction system, *Appl. Microbiol. Biotechnol. 103*: 7805–7817, 2019.

Hansen,R.B., M.A. Agerbaek, P.M. Nielsen, A. Rancke-Madsen,A, and J.M. Woodley, Esterification using a liquid lipase to remove residual free fatty acids in biodiesel, Process *Biochem. 97*: 213–221, 2020.

Alternative base oils:
a perspective

Raj Shah, Mathias Woydt, and Hillary Wong

Engine oil is a product that has existed for a very long time and dates as far back as 1866, when the first branded engine oil emerged [1]. The technology of automobiles is always evolving; therefore, innovation of engine oils must keep up with the evolution of automobiles. Creating new formulas and compositions of engine oil is vital to allow the automobile to perform at its best with minimal wear and maximum protection to prolong engine life.

- **Alternative base oils can improve fuel economy and performance, as they can reduce the material footprint and leave the engine oil cleaner for much longer compared to traditional engine oil.**

- **This increases vehicle mileage and allows the vehicle to reduce CO$_2$ emissions without having to replace the engine.**

- **In the near future, it can be expected that more lubricant formulas will incorporate alternative base oils.**

When creating new engine oil formulas, it is also essential to consider formulations that lower viscosity and improve friction properties. When viscosity is reduced and friction properties at boundary and mixed lubrication regimes are improved, the fuel economy, performance, and life of an engine can be improved. One notable aspect of engine oil formulas is the base oil, as base oil is a fundamental ingredient of all engine oils. Base oil makes up about 75% to 80% of an engine oil, which is the majority of the product [2]. Figure 1 shows the percentages of the ingredients used to make engine oil.

Base oil is typically produced by refining crude oil [2]. Base oil is categorized by the American Petroleum Institute (API) into five different groups, I–V. These groups distinguish the types of base oils by the way they are processed. Early traditional base oils are categorized in Group I and are produced by solvent-refining technology [4]. Group II and III are also refined from crude oil, except Group II has better antioxidation properties and is processed by hydrocracking (a two-step process that uses high pressure, heat, a catalyst, and hydrogen [5]), while group III is more refined and severely hydrocracked compared to group II. Group IV consists of polyalphaolefins (PAOs) and are processed by synthesizing. Group V consists of all other base oils such as esters, polyalkylene glycol (PAG), and bio-olefins. In Europe, polyinternal olefins (PIO) were added by the representative body for the European lubricants industry, ATIEL, as a group VI base oil. Currently, Group II is the most common type of base oil used in engine oils.

Most current base oils are made from crude oil, a nonrenewable resource, but more renewable alternatives for base oils are being researched and introduced into newer engine oil products. Bio olefins are alternative base oils categorized in Group IV. Alternative base oils such as esters and PAGs are categorized as group V base oils. These alternative base oils have different characteristics with varying strengths and weaknesses. With different characteristics, each base oil will have specific and slightly different applications [6]. Table 1 compares the strengths and weaknesses of different base oils. The maximum operating temperature is an indication of thermal stability and depends upon the antioxidant additive packages used.

Group V base oils stray from common market base oils and refining practices that are found in group I–III base oils. However, many Group V base oils are researched more in-depth as of late for the creation of new low-viscosity and low-friction engine oils. Alternative base oils may be needed, because the Noack evaporation of low-viscosity hydrocarbons increases with decreasing viscosity. Esters and PAGs have much lower volatility due to their molecular polarity.
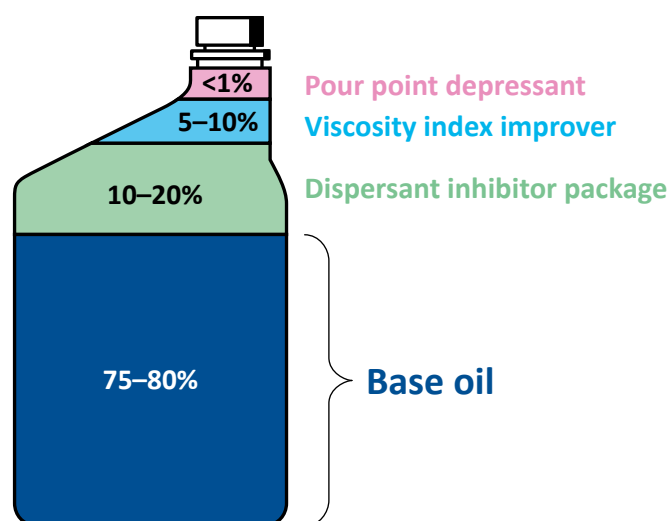
<1%   Pour point depressant

5–10%   Viscosity index improver

10–20%   Dispersant inhibitor package

75–80%   Base oil

FIG. 1. Base oil makes up most of the engine oil. [3]

THE ALTERNATIVES

Base oils other than the typical hydrocarbon base oils appeared in aircraft engines during WWII. The German synthetic lubricant base oils consisted of a blend of poly(ethylene)-ester concept, generated more or less no soot, and was a low-viscosity grade in the range of a 0W–20 engine oil of today. The kinematic viscosity at 100°C of SS1600 used in air cooled radial BMW 801 was ~6.2 mm²/s (SS = synthetischer Schmierstoff = synthetic lube). The ester was a reaction product of methyladipic acid with branched C8-C14 alcohols. The US Air Force used a polypropyleneglycolmonobutylether (Union Carbide Prestone 200, LB-550 base oil) from March 1944 onward with a viscosity at 100°C of 18.5 mm²/s (safer design!). The PAG based aircraft engine oil was ash-free and formed no deposits. Until today, the price level of such base oils limited the market penetration.

The key differences in properties among hydrocarbons, esters, and polyalkylene glycols originate from the presence of oxygen in their carbon backbones. Esters have one, two, or three "ester" links with two oxygen atoms each, whereas PAGs have in each monomer an "ether" link. The oxygen represents an increase in molecular polarity, which increases the lubricity and viscosity index as well as reducing the Noack evaporation. However, the polarity due to the oxygen atoms makes the compatibility with polymeric materials challenging. Esters are one of the Group V base oils added as a co-base stock for engine oils. There were many past attempts to market engine oils using 100% esters as a base oil stock. The use of esters in engine oil sparks interest in creating low-viscosity, high-viscosity index and reduced friction engine oils that can improve fuel economy and reduce greenhouse gas emissions [7].

Esters are being introduced into low-viscosity engine oils due to their low-volatility and high-viscosity index (VI) characteristics. Esters further exhibit great thermal and oxidative stability properties which allow them to perform well at high temperatures [8] associated with low (reduced) Noack volatilities (physical evaporation). Esters may go through a thermal degradation where oxygen is not involved [9]. This reaction, in which the ester typically goes through beta-elimination at temperatures of 275° to 315°, does not occur as metals act as a catalyst and lower the temperature to 200°. Since there is "no hydrogen present in the beta position of oxygen" [9], beta-elimination is unlikely to occur and therefore increases thermal stability. Esters also have great solvency, as they can solubilize complex additives into the formulation [10]. Due to esters' high VI and great oxidative stability, the formulas of the engine oils that incorporate esters will use less VI improvers and dispersants, both of which are known to increase the viscosity of the finished engine oil product [7].

Esters can meet the criteria of environmentally acceptable lubricants (EALs) as per Vessel General Permit [10]. There are three different classes of esters that are commonly found

TABLE 1. Base oils have differing characteristics from one another. [6]

Synthetic	Strengths	Weaknesses
Polyalphaolefins (PAOs) Maximum operating temperature 399°F/ up to 204°C	High VI, high thermal oxidative stability, low volatility, good flow properties at low temperatures, nontoxic, compatible with mineral oils	Biodegradability depends from molar mass, limited additive solubility, seal shrinkage risk
Di-, tri-, tetraesters, polyolesters Temperature 399°F/ up to 204°C	Nontoxic and biodegradable, high VI, good low-temperature properties, miscible with hydrocarbons	Hydrolytic stability and miscibility with hydrocarbons can be an issue, limited seal and paint compatibility
Phosphate esters Maximum operating temperature 241°F/116°C	Highest auto-ignition temperature, excellent wear resistance and scuffing protection	Moderate VI, limited seal compatibility, not miscible with hydrocarbons, moderate hydrolytic stability
Polyalkylene glycols (PAGs) Temperature 399°F/ up to 204°C	Low friction and excellent lubricity, nontoxic and biodegradable, high VI, good thermal and oxidative stability	Limitations in soluble additives, miscibility with other base oils depends on backbone, limited seal and paint compatibility
Silicones Maximum operating temperature 486°F/252°C	Highest VI, high chemical stability, excellent seal compatibility, very good thermal and oxidative stability	Not miscible with hydrocarbons and additives, weak lubricity under mixed/boundary lubrication

in engine oils: diesters, triesters (trimethylolpropane esters (TMP)), tetraesters, and polyolesters. These esters can have great biodegradability and are miscible with other oils and additives [6]. Triesters are highly desired as they can be used in a wide range of viscosities. Esters have other properties that are suitable for engine oils. Esters induce a good seal swell in order to compensate for the shrinkage by PAOs and have low deposit formation which is good for preserving the lubricant for a long time without having to change the engine oil for a while [9]. Esters in engine oils also provide high flash points of up to 325°. Having high flash points makes the engine oil safe, as it is more resistant to ignition. An experimental engine oil was made with diesters (adipate ester or di-isotridecyl adipate) and polyolesters (TruVis™ P3020) to compare with engine oil using a Group II base oil [7]. Further testing showed that the engine oil with ester base oil had better depositing performance and friction reduction compared to the engine oil with Group II base oil. The results from the test indicates that esters used in engine oils provide low-viscosity and low-friction properties and therefore improve fuel economy and engine performance.

Another alternative base oil used to create low-viscosity and friction engine oil is PAGs. PAGs are classified as Group V base oil and have suitable properties of high viscosity indices (VI), low Noack evaporations, and high lubricity. PAGs are categorized into two different types: water-soluble and water-insoluble (newly additional also in oil-soluble) [11]. Oil-soluble PAGs have been becoming more common in the market, as they are used for high-temperature applications. PAGs have a high VI and thermal stability. PAGs have greater heat capacities compared to esters and hydrocarbons. The heat capacities of PAGs are around 22% above typical esters and hydrocarbons [12]. The heat capacity of PAGs affects viscosity loss and film-forming abilities. Since PAGs have higher heat capacities, it allows the engine oil to operate at higher temperatures without declining in lubricating performance. Figure 2 shows the heat capacities of alternative oils, such as PAGs, esters, and blends of esters with hydrocarbons (HC-Ester), compared
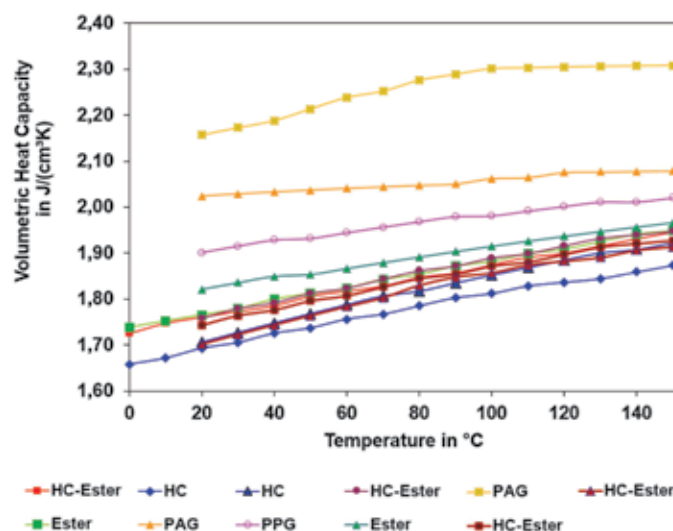

FIG. 2. Heat capacities of alternative and conventional oils. [13]

to hydrocarbon-based (HC) oils. Figure 2 displays volumetric heat capacities because an oil pump is defined by a volumetric feed. Esters offer slightly higher volumetric heat capacities surpassed by polyglycols.

The thermal conductivity of PAGs and esters are slightly higher than those of hydrocarbons, therefore improving the heat transfer properties of engine oils through the oil film [13]. With high intrinsic VIs, PAG base oils can be used to create low-viscosity and low-friction engine oils. PAGs also leave little to no residue, making engine oils clean and long-lasting due to their chemical structure [12]. Leaving little to no residue, PAGs are used to reduce friction in engine oils as co-base stock and therefore provide great film strength properties. Low volatility in high temperatures is another property of PAGs, which allows PAG-based engine oils to be used in high temperatures as well as low temperatures. PAGs also show low pour points and great oxidative stability. PAGs are used to create low-viscosity engine oils by combining them in a formulation that consists

TABLE 2. Base oil properties of different OSPs [17]

Quantity	Method ASTM	OSP-18	OSP-32	OSP-46	OSP-68	OSP-150	OSP-220	OSP-320	OSP-460	OSP-680
Kin. Viscosity at 40°C [mm2/sec]	D445	18	32	46	68	150	220	320	460	680
Kin. viscosity at 100°C [mm2/sec]	D445	4.0	6.4	8.5	11.5	23.5	33	36	52	77
Viscosity Index	D445	123	146	164	166	188	196	163	177	196
Pour point [°C]	D97	-41	-57	-57	-53	-37	-34	-37	-35	-30
Fire point [°C]	D92	220	242	240	258	258	258	260	265	270
Density at 25°C [g/ml]	D7042	0.92	0.94	0.97	0.97	0.97	0.97	0.97	0.97	0.97
Aniline point [°C]	D611	<-30	<-30	<-30	<-30	<-30	-26	n/d	n/d	n/d

of fatty alcohol. When combined with fatty alcohol, the engine oil's viscosity drops to 40° and VI increases [12, 13]. PAGs have different solvent properties compared to those of hydrocarbons. Since PAGs can be both water- and oil-soluble, they allow for more flexibility in engine oil compositions and applications. The water-soluble properties of PAGs allow for easy maintenance of equipment, as cleaning the equipment would be easier.

Oil-soluble PAGs (also known as OSPs) are derived from butylene oxides and other kinds of oxides [11, 14] or represent the combination of ethylene oxide/propylene oxide with a fatty alcohol (often C_{10}-C_{14}). OSPs have many beneficial functional properties like those of traditional PAGs. OSPs provide a lot of flexibility in the formulation, which affects molecular weights and base stock properties. Table 2 shows the variation in properties that different kinds of OSPs have, making OSPs versatile in many applications.

A unique property of OSPs is their low aniline points of less than -20°. The aniline point is typically used as an indicator when determining the solvency or polarity of the base oil. Since the aniline point of OSPs are low, the OSPs are determined to be highly polar. In general, PAGs are moderately polar. This indicates OSPs are more hydrolytically stable compared to esters, which results in a longer lifespan for the engine oil.

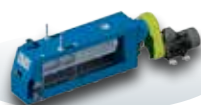Bio-olefins are another type of alternative base oil derived from renewable resources. Olefins are typically processed through crude oil refining and fluid-catalytic cracking, steam cracking, and dehydrogenation. Bio-olefins are olefins that are produced through alternative feedstock, such as biomass [15]. This alternative base oil is garnering more global interest, and technological advances are being researched and designed with new kinds of bio-olefins in mind. Bio-olefins go through processes such as fermentation, gasification, cracking, and deoxygenation. Different processes used to produce bio-olefins create different biomass intermediates. Bio-olefins can be obtained as botryococcene ($C_{30}H_{50}$) from green algae (botryococcus braunii) or as beta-farnesene ($C_{15}H_{32}$) from sugar by modified yeast cells. Both are highly branched, and their multiple double bonds need to be eliminated by hydrogenation. From such feedstocks and depending on which kind of process is used, bio alcohols, diols, and other oxygenates are created and used to produce renewable streams for synthetic base oils.

Ethylene is a type of olefin that can be produced through renewable feedstock—although it is typically produced through steam cracking from hydrocarbons. Ethylene can be further produced from bio alcohols, plants, and microorganisms [15], depending on which process is being used. The main benefit from creating a base oil of bio-olefins is the content of renewables. Bio-olefins used in base oils can have low volatility, which in turn makes them suited for low viscosity and friction in engine oils.

The next important consideration for ultralow viscosity SAE 4&8 grade oils is the Noack volatility (1h at 250°C) [16]. The physical evaporation (Noack) increases with reducing the viscosity, because at the same time the molar mass of the backbone goes down. The polarities of esters and polyglycols offer here for ultralow viscosity base oils functional benefits of reduced volatilities associated with higher viscosity indices, when compared to hydrocarbons of same kinematic viscosity at 100°C. In summary, several additional requirements and trends create an opportunity frame for alternative base oils.

Raj Shah is a Director at Koehler Instrument Company in New York, where he has worked for the last 25 years. An expert in the field of alternative energy, he is an elected Fellow by his peers at IChemE, STLE, AIC, NLGI, INSTMC, CMI, The Energy Institute, and The Royal Society of Chemistry. A Ph.D in Chemical Engineering from the Pennsylvania state University and a Fellow from the Chartered Management Institute, London, he is also a Chartered Scientist with the Science Council, a Chartered Petroleum Engineer with the Energy Institute and a Chartered Engineer with the Engineering council, UK. An ASTM Eagle award recipient, Dr. Shah recently coedited the bestseller, "Fuels and Lubricants handbook", details of which are available at https://www.astm.org/DIGITAL_LIBRARY/MNL/SOURCE_PAGES/ MNL37-2ND_ foreword.pdf.

A 2020 recipient of the illustrious Tau Beta Pi eminent engineer title, he is currently an adjunct professor in the department of Material Science and Chemical engineering at State University of New York, Stony Brook, NY. He has over 250 publications and volunteers on advisory board of directors at several US universities. More information on Raj can be found at https://www.che. psu.edu/news-archive/2018/Alumni-Spotlight-Raj-Shah.aspx.

Dr. Mathias Woydt is managing director of MATRILUB Materials I Tribology I Lubrication, with more than 34 years of experience in R&D and product development. He has published more than 340 publications and has filed 51 priority patents. He is also board member of the German Society for Tribology. He is an adjunct professor for tribology at the Technical University of Berlin. He is recipient of the ASTM award of Excellence. He can be reached at m.woydt@matrilub.de.

Ms. Hillary Wong is a graduate student at State University of New York (SUNY), Stony brook, and an intern at Koehler Instrument Company.

References

[1] Keegan, Matt, "Valvoline at 150: America's Oldest Branded Motor Oil, *Auto Trends Magazine*, May 24, 2016, autotrends.org/2016/05/24/ valvoline-at-150-americas-oldest-branded-motor-oil/

[2] Lee, David, Base oil basics: Quality starts at the base, *Chevron Lubricants*, February 1, 2018, www. chevronlubricants.com/en_us/home/learning/from-chevron/personal-rec-vechicles-and-equipment/base-oil-basics-quality-starts-at-the-base.html

[3] Modern base oils and blending for optimal performance, *Lube-Tech*, February 2012, pp. 1–7

[4] Base oil groups explained," *Machinery Lubrication*, Noria Corporation, October 9, 2012, www. machinerylubrication.com/Read/29113/base-oil-groups

[5] "Hydrocracking." Hydrocracking—an overview, ScienceDirect Topics, 2019, www.sciencedirect.com/ topics/engineering/hydrocracking

[6] Fitch, Bennett, Understanding the Differences Between Base Oil Formulations, *Machinery Lubrication*, February 6. 2017, www.machinerylubrication.com/Read/30730/ base-oil-formulations

[7] Esche, C., *et al.*, Esters for engine oils, *Tribology and Lubrication Technology*, November 2018, p. 80–82

[8] Esters. BASF AG, Ludwigshafen, www.basf.com/ global/en/products/segments/industrial_solutions/ performance_chemicals/business/fuel-and-lubricant-solutions/esters.html

[9] S. Lucazeau, From jet engine oils to high temperature industrial lubricants, *LUBE Magazine 154*: 27–29, December 2019

[10] Standard Guide for Selection of Environmentally Acceptable Lubricants for the U.S. Environmental Protection Agency (EPA) Vessel General Permit, ASTM work item WK68688

[11] M.R. Greaves, "Oil Soluble Polyalkylene glycols," In: Rudnick, L.R. (Ed), *Synthetics, Mineral Oils and Biobased Lubricants*

[12] Woydt, M., Polyalkylene glycols as next generation engine oils, *J. ASTM Int. 8* (6), paper ID JAI103368 and ASTM STP1521, 2012, ISBN: 978-0-8031-7507-5

[13] Schmidt R., G. Klingenberg, and M. Woydt, Thermophysical and viscosimetric properties of environmentally acceptable lubricants, *Industrial Lubrication and Tribology 58*: 210–224 , 2006, https://doi. org/10.1108/00368790610670809

[14] Greaves, Martin, Oil soluble polyalkylene glycols, *LUBE-Tech*, Dec. 2013, pp. 1–4.

[15] Zacharopoulou, V. and A.A. Lemonidou, Olefins from biomass intermediates: a review, *Catalysts 8*(1): 2, 2018, https://doi.org/10.3390/catal8010002

[16] H.R. Henderson, Solving the engine oil puzzle, part 2, *Compoundings*, February 2018, p. 16–19

Oleofoams: toward new food products and soft materials

A-L. Fameau

- **Research on edible oleofoams is still scarce compared to research on aqueous food foams, even though oleofoams present promising applications and have attracted considerable interest from the scientific community and industry during the last five years.**

- **Edible oleofoams are currently based on heating a vegetable oil solution containing a high-melting-point component, which crystallizes upon cooling. After aeration, the crystals adsorb to air bubble surfaces, and those remaining in excess in the continuous phase form an oleogel.**

- **Understanding the formation and stabilization of oleofoam mechanisms will contribute to the engineering of new food products and soft materials.**

Foams are based on gas bubbles dispersed in a liquid phase containing surface-active species. Two different categories of liquid foams exist depending on the nature of the continuous phase: aqueous (water-based) and non-aqueous (oil-based). Aqueous foams have been widely used for industrial applications for years, whereas oil foams were first discovered in the 1970s and essentially ignored for about 30 years.

In the last five years, interest in using oil foams for edible applications has grown tremendously [1] due to their potential as novel structuring materials to replace solid fats. It is very difficult to replace solid fats with liquid oils since this leads to a loss of texture and mouthfeel. Oleofoams, on the other hand, are seen as a promising option for food technologists who are seeking to develop products with both a reduced fat content and new appealing textures and sensorial properties [1].

Oleofoams offer other advantages which are not only of interest to food technologists, but also to companies that are developing cosmetic and pharmaceutical applications [2]. Oleofoams exhibit very long-term stability and can be stored for months, even at temperatures above room temperature. Enhancing product shelf life is an important way for the food, cosmetic, and pharmaceutical industries to reduce waste and preserve product quality during storage. Also, because oleofoams are based on oil without water, they drastically reduce microbial spoilage and the need for preservatives. Moreover, few or even no additives are needed to produce them. Therefore, oleofoams appear to be a perfect solution for meeting consumer demand for a "clean label" [1].
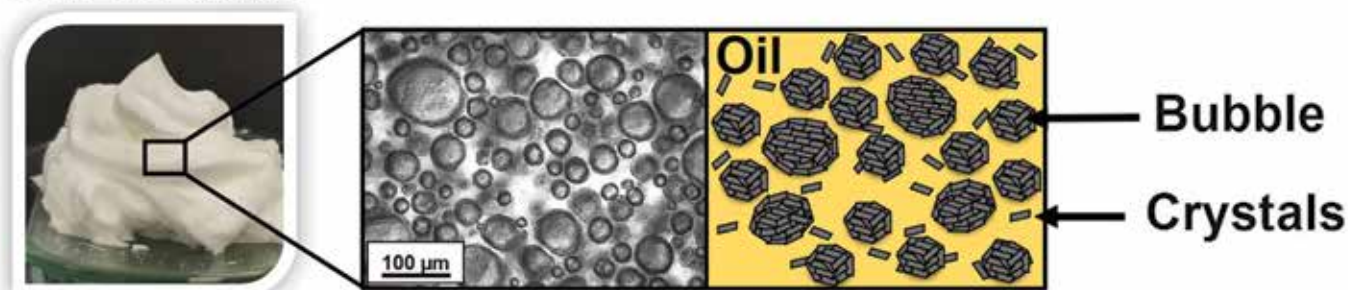
In the last two years, oleofoams were successfully described in several patents as a partial replacement for butter in food products, such as sponge cakes, biscuits, laminated pastry, and mayonnaise [1]. Oleofoams helped

OLEOFOAM

FIG. 1. Illustration of oleofoam stabilized by the presence of crystals both in bulk and at the interface as observed by optical microscopy

to decrease the fat content, while preserving a similar taste in the products. Similarly, several cometic- and pharmaceutical-related patents described the use of oil foams to provide new sensorial textures in formulations consumers apply on the skin and the hair, and to help deliver drugs more efficiently due to the continuous phase made of oil [1,2].

Interest in oil foams is relatively new in comparison to aqueous foams [1]. Although their stability mechanisms are very similar, it is much more difficult to produce and stabilize oil foams, since the common surfactants used for aqueous foams are considerably less surface-active in oils [3]. Most of the oils already have a surface tension so low that it makes the adsorption of hydrocarbon-based surfactants energetically unfavorable. Until now, oil foams could only be obtained when the hydrocarbon-based surfactant formed crystalline particles in the oil phase, stabilizing the gas bubbles [4]. Therefore, the strategy developed to prepare stable edible oil foams is to combine two main components: an edible oil for the continuous phase, and an edible oil-soluble molecule that will crystallize upon cooling (Fig. 1) [4]. When the concentration of the edible oil-soluble molecule is high, leading to high concentration of crystals, the mixture between these two components forms an oleogel. By aerating the oleogel, the crystalline particles adsorb to air bubbles on the surface, leading to the formation of oleofoams (Fig. 1).

The particles protect the foams against the destabilization mechanisms: coalescence and coarsening. The presence of the oleogel suspends buoyancy-driven creaming of air bubbles within the foam. The oil drainage is directly linked to the rheological properties of the crystal network inside the liquid oil phase. Therefore, both the presence of the crystalline particles at the interface and in the continuous phase stabilize the oil foam by reducing the three main mechanisms of foam destabilization: drainage, coarsening, and coalescence.

To obtain edible crystalline particles leading to edible oil foams, the first systems described in the literature for food applications were based on mono- and/or diglycerides [4]. Then, in our group, we used long-chain alcohols in sunflower oil to describe the formulation rules and stabilization mechanisms [5]. Following this work, various edible components used to produce oleofoams were described in the literature, including long-chain fatty acids, triglycerides, and phytosterols [1]. Recently, another simple approach to producing oleofoams was described and is based on the use of oils, such as coco-

nut oil, which is naturally composed of crystalline particles dispersed inside a continuous oil liquid phase [1].

In oleofoams, the long-term stability comes from the crystals, which govern overall foam stability due to both the contributions at interfaces and in bulk [1]. However, the structure at the interface, the thickness of the interfacial layer, and the resulting interfacial rheological properties are still unknown. The main problems to drawing clear conclusions about the interfacial stabilization mechanisms stem from the fact that the fatty components used to stabilize the oil foams—as well as their purity—were different in each study, and the processes used to produce the foam also varied [1].

A better understanding of oleofoams could be obtained by making links with similar soft matter systems, such as Pickering aqueous foams and emulsions, as well as oleogels. Our group recently demonstrated that the weight ratio (R) between long-
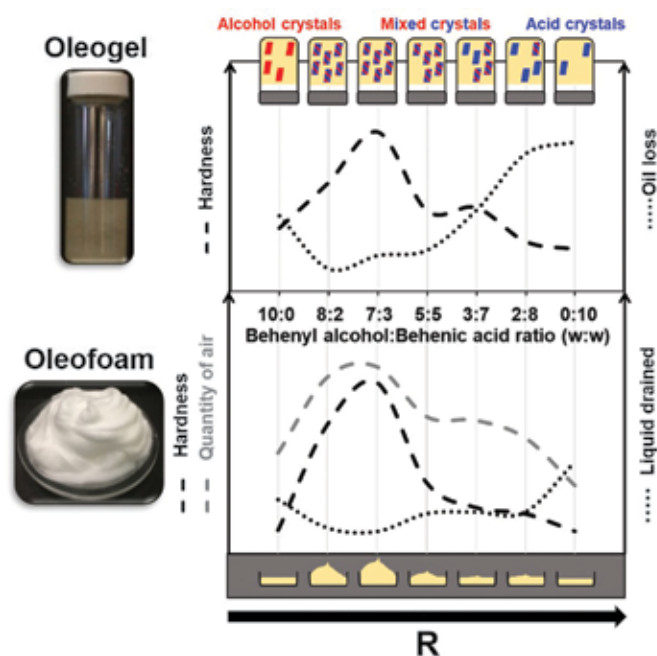
FIG. 2. Illustration of the links between oleogel and oleofoam properties as a function of the weight ratio (R) between fatty alcohol and fatty acid in sunflower oil [2]. When mixed crystals of behenyl alcohol and behenic acid are formed inside the oleogel, the hardness is maximum and the oil loss is minimum, leading to the highest oleofoam hardness and quantity of air, and the lowest liquid drained inside the foam.
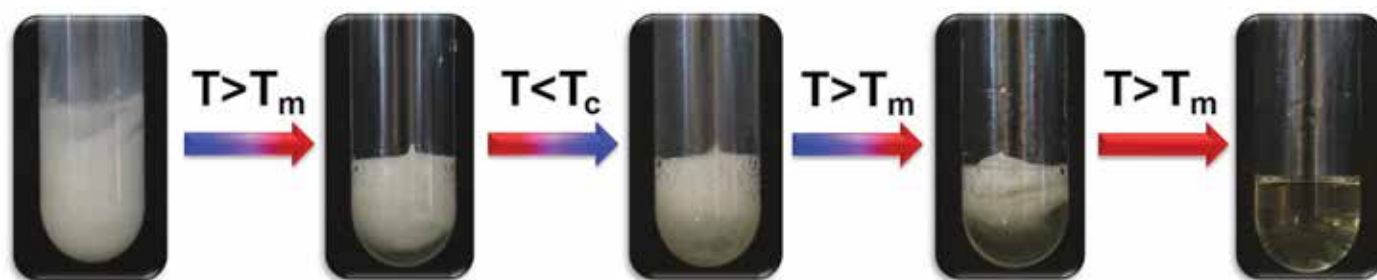
FIG. 3. Illustration of the ability of oleofoams to be destabilized in a few minutes by increasing the temperature above the temperature at which the crystals melt (T_m). By decreasing the temperature below the temperature at which the crystals form (T_c), the oleofoams destabilization is stopped. The transition between stable and unstable oleofoam is completely reversible.

chain fatty acids and fatty alcohols could be used to tune both the oleogels and their oil foam properties (Fig. 2) [2]. Two optimal R, for which mixed crystals are present, produced the best foams in terms of overrun, foam firmness, and foam stability. R not only affected the crystal size, but also the number of crystalline particles present in the oleogel. Mixed crystals with a small size helped to produce and stabilize the foams. We highlighted that there is a link between oleogel stability and hardness, with resulting oleofoam properties (Fig. 2) [2].

New fundamental insights will also offer the possibility to push further the applications of oil foams as smart soft materials. For example, our research group highlighted that oleofoams can be thermoresponsive (Fig. 3) [5]. When oleofoams are stored at temperatures at which crystals dissolve, they completely collapse in a few minutes due the disappearance of the crystals—both at the interface and in the continuous oil phase. No foam remains, and only a clear molecular solution is present. Since the crystals reform from the molecular solutions on cooling, the foam stability can be easily alternated between high and low by tuning the temperature above or below the crystals' melting temperatures through multiple temperature cycles [5].

Moreover, we produced photoresponsive oleofoams [5]. The concept is based on the use of carbon black particles as internal heat sources incorporated into the foam matrix to generate heat. Carbon black particles are known to absorb UV light and dissipate the adsorbed energy as heat. Without UV, the oleofoams are ultrastable, but under UV illumination, the foam is destabilized. Under UV illumination, the carbon black particles absorb the light and act as photothermal heat generators, leading to a foam temperature increase. The crystals melt inside the foam, leading to foam destabilization. By removing the UV light, the foam destabilization process is stopped. This example demonstrates the possibilities offered by oleofoams as smart soft materials.

This brief overview about oleofoams provides a framework for understanding the formulation rules and stabilization mechanisms. In the future, new fundamental insights will make it possible to optimize existing edible formulations and extend the use of oleofoams for other applications.

Anne-Laure Fameau works for L'Oréal Company in Research & Innovation, Saint-Ouen, France. She received her Ph.D. in Physical Chemistry from the University of Nantes, France, in 2011. At the end of 2011, she obtained a permanent staff position at the French National Institute of Agricultural Research (INRAE). In 2013, she spent six months as visiting scientist in the group of Prof. Velev at North Carolina State University, USA. In 2014, she was awarded an Agreenskills Fellowship from the European Union to spend six months as visiting scientist in the group of Prof. von Klitzing at Technische Universität Berlin, Germany. In 2015, she joined L'Oréal company. In 2018, she received the European Young Lipid Scientist Award and the Langmuir Prize at ECIS Conference. In 2021, she received the AOCS Young Scientist Research Award. Her research interests are in the field of responsive soft materials based on lipids and green surfactants, with a particular emphasis on foams and interfaces. Her research is focused on the development of these systems and their detailed structural characterization using scattering techniques such as Small Angle Neutron and X-ray scattering. Fameau also enjoys transmitting her passion for science to young students, especially to promote science for girls. She can be reached at anne-laure.fameau@rd.loreal.com.

References

[1] Fameau, A.-L. and A. Saint-Jalmes, Recent advances in understanding and use of oleofoams, *Front. Sustain. Food Syst.*, August 2020.

[2] Callau, M., K. Sow-Kébé, N. Jenkins, and A.-L. Fameau, Effect of the ratio between fatty alcohol and fatty acid on foaming properties of whipped oleogels, *Food Chem. 333*: 127403, 2020.

[3] Fameau, A.-L. and A. Saint-Jalmes, Non-aqueous foams: current understanding on the formation and stability mechanisms, *Adv. Colloid Interface Sci. 247*: 454–464, 2017.

[4] A.-L. Fameau, Chapter 13: Non-aqueous foams based on edible oils, in: *Edible Oil Structuring: Concepts, Methods, and Applications*, The Royal Society of Chemistry, London, 2018: pp. 275–307.

[5] Fameau, A.-L., *et al.*, Smart nonaqueous foams from lipid-based oleogel, *Langmuir. 31*: 13501–13510, 2015.

Physicochemical and thermal behavior of tucumã oil and butter

Mayanny Gomes da Silva, Larissa Magalhães Grimaldi, Kamila Ramponi Rodrigues de Godoi, Fernanda Luisa Lüdtke, Thais Jordânia Silva, Renato Grimaldi, and Ana Paula Badan Ribeiro.

The tucumã (*Astrocaryum vulgare, Arecaceae*) is a native tree of the Amazon region that grows in northern Bolivia, Colombia, Venezuela, Guyana, Suriname, and Brazil. It is primarily found in the state of Amazonas (Santos, Sagrillo, Ribeiro, and Cruz, 2018). The tree's fruits (Fig. 1) are enclosed within a pulp that is yellow to orange in color, and the orange oil from this pulp is rich in polyunsaturated fatty acids and carotenoids. The kernel, in turn, produces a lauric fat that resembles palm kernel oil. Currently, applications for tucumã and its lipid fractions are limited to the Amazonian people (Bora, Narain, Rocha, De Oliveira Monteiro, and De Azevedo Moreira, 2001; Cordenonsi, *et al.*, 2020; Mambrin and Barrera Arellano, 1997; Pereira, *et al.*, 2019; Santos, *et al.*, 2018).

- **Pulp from the tucumã fruit is a source of oil rich in polyunsaturated fatty acids and carotenoids, while the tree's kernel produces a butter that resembles palm kernel oil.**

- **Applications for these lipid fractions are currently limited to the Amazonian people.**

- **This article reviews the physicochemical characteristics and potential applications.**

TUCUMÃ PULP OIL

Table 1 presents the results for acid value, peroxide, iodine, and saponification for tucumã pulp oil. The values of acidity and peroxide are reference parameters for the quality of oils and fats. As seen in the table, high acid peroxide values were observed for tucumã pulp oil. These high values are related to the handling, processing, and storage of the fruits. High temperatures, humidity, and other climate characteristics of the Amazonian region also have a significant influence on these parameters, favoring hydrolysis and oxidation (Rodrigues, Silva, Marsaioli, and Meirelles, 2005). Meanwhile, the iodine and saponification indexes are in line with what would be expected for an oil with the fatty acid profile of this oil.

The major fatty acids are oleic acid (60.86%) and palmitic acid (21.88%), totaling 71.44% of the unsaturated fatty acids.

The color observed for the tucumã pulp oil is orange-red, confirmed by the Cor Lovibond analysis, with red (R) as the predominant parameter. The reddish-orange color is related to the high content of carotenoids observed for this oil: 1,094.10 mg/100g. Carotenoids have an important functional role as antioxidants. No significant values were observed for chlorophyll A and B. Small amounts of alpha- and beta-tocopherols, but no values for tocotrienols were observed.

Tucumã pulp oil had a high content (28.16%) of monoacylglycerols (MAG) and free fatty acids (FFA) as well as 9.84% diacylglycerols (DAG).

FIG. 1. Tucumã fruit (Embrapa, 2018)

TABLE 1. Physicochemical characterization of tucumã pulp oil

Parameter	Tucumã pulp oil
Acid value (g of oleic acid/100g)	11.94
Peroxide value (meq.O_2/kg)	18.59
Iodine value (g I_2/100 g)	72.60
Saponification value (mg KOH/g)	194.27

This corroborates with the high acidity value observed for this oil.

The thermal profile for tucumã pulp oil is shown in Fig. 2. Three peaks of crystallization were observed, a more saturated fraction with higher onset crystallization temperature (15.89°C), related to TAGs with palmitic acid, an intermediate fraction with crystallization temperature at -6.79°C, and another fraction with a crystallization point at -43.96°C, probably associated to more unsaturated TAGs with oleic acid. Regarding melting profile, two peaks were observed (Table 2, page 26).
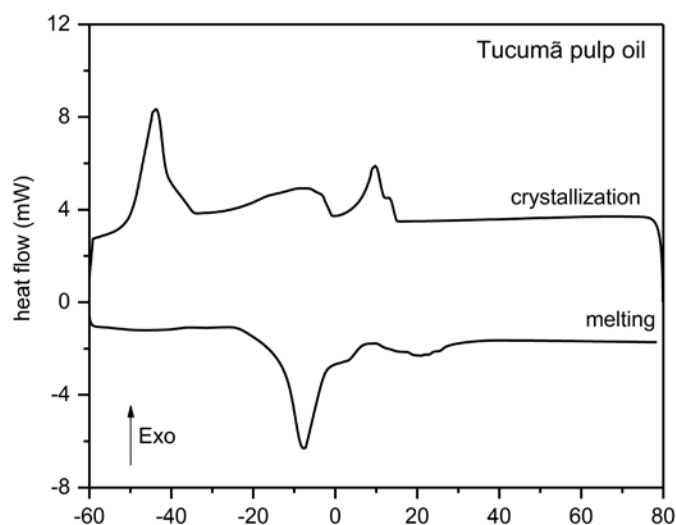

FIG. 2. Crystallization and melting curves obtained by differential exploratory calorimetry (DSC) as a function of the temperature for tucumã pulp oil

TUCUMÃ BUTTER

The tucumã butter was characterized by low acidity and low peroxide content. These reduced values are related to the low levels of unsaturated fatty acids (19.61%) present in this butter, which makes the fat less susceptible to hydrolysis and oxidation. The low content of unsaturated fatty acids is also related to the low iodine value (16.71 g, I_2/100 g). The saponification value, in turn, is related to the average chain length of all the fatty acids present in an oil or fat. Short-chain fatty acids have higher saponification values than long-chain fatty acids. As the tucumã butter has medium-chain fatty acids (lauric and myristic acids), a high saponification value was observed.

The tucumã butter presented 80.39% of saturated fatty acids, the majority being lauric acid (C12:0) and myristic acid (C14:0). Oleic acid (C18:1) was the predominant unsaturated fatty acid. This fatty acid composition is similar to the palm kernel oil composition (Alimentarius, 1999).

The tucumã butter presented 90.47% of TAG and 9.53% MAG and FFA. These low levels of MAG and FFA in comparison with tucumã pulp oil is in accordance with the lowest level of acidity found for tucumã butter.

The predominant TAG chain sizes were 36 (25.24%), 38 (27.93%), and 40 (13.90%), which are in accordance with the predominant lauric, myristic, and oleic fatty acids observed for tucumã butter.

Tucumã butter has a slightly yellowish-white color, with yellow (Y) and red (R) being the main colorimetric parameters observed. Low tocopherol and tocotrienol contents were observed. These values are expected since predominantly saturated fats have lower contents for these components (Kamal-Eldin, 2005).

The crystallization and melting curves and thermal parameters for tucumã butter are shown in Fig. 3 and Table 4, respectively. Crystallization showed two overlapping peaks with the beginning at 13.08°C and end at -17.19°C. The melting also presented a wide temperature range, from 9.96°C to 36.85°C, thus indicating complete melting at body temperature.

The solid fat content (SFC) is a measure of the solid fat in a sample at a specific temperature. Fig. 4 shows the solid fat profile for tucumã butter determined by low- field magnetic resonance imaging. With the increase in temperature, the solid content showed a non-linear reduction. At 25°C, the solid con-

TABLE 2. Temperature parameters of crystallization and melting for tucumã pulp oil: crystallization start temperature (Tsc) and melting (Tsm); crystallization temperature (Tc) and melting (Tm); crystallization end temperature (Tec) and melting (Tem); crystallization enthalpy (ΔHc) and melting (ΔHm)

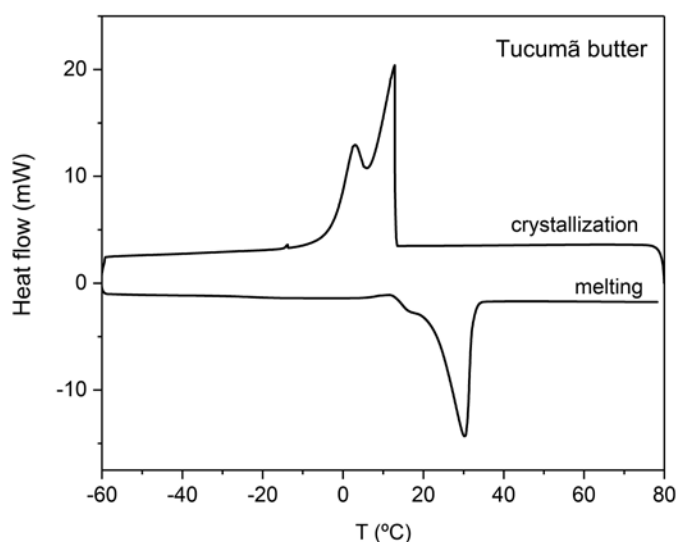| Tucumã pulp oil	Peak I	Peak II	Peak III
Tsc (°C)	15.89	-0.37	-34.04
Tc (°C)	9.76	-6.79	-43.96
Tec (°C)	-0.37	-34.04	-55.57
ΔHc (J/g)	8.59	12.04	22.88
Tsm (°C)	-27.44	8.08	-
Tm (°C)	-7.82	20.88	-
Tem (°C)	8.08	33.09	-
ΔH*m* (J/g)	62.80	8.40	-

TABLE 3. Physicochemical characterization of tucumã butter

Parameters	Tucumã butter
Acid value (g of oleic acid/100g)	0.37
Peroxide value (meq.O_2/kg)	3.58
Iodine value (g I_2/100 g)	16.71
Saponification value (mg KOH/g)	236.35

FIG. 3. Crystallization and melting curves for tucumã butter obtained by differential exploratory calorimetry

TABLE 4. Thermal parameters of crystallization and melting for tucumã butter: crystallization start temperature (Tsc) and melting (Tsm); crystallization temperature (Tc) and melting (Tm); crystallization end temperature (Tec) and melting (Tem); crystallization enthalpy (ΔHc) and melting (ΔHm)

Tucumã Butter	
Tsc (°C)	13.08
Tc (°C)	11.95
Tec (°C)	-17.19
ΔHc (J/g)	93.26
Tsm (°C)	9.96
Tm (°C)	30.50
Tem (°C)	36.85
ΔH*m* (J/g)	112.70

tent was 50%, and at 35°C, the fat was liquid. This behavior at body temperature is determinant for the aroma properties and softness of the products in the mouth.

Crystallization kinetics has a significant influence on the final structure of fats and is intrinsically related to their plasticity, besides being important in determining the limitations of fat for industrial applications. The isotherm of crystallization at 20°C for tucumã butter is presented in Fig. 5. A crystallization induction time of 20.5 minutes and maximum solid content of 63% was observed.
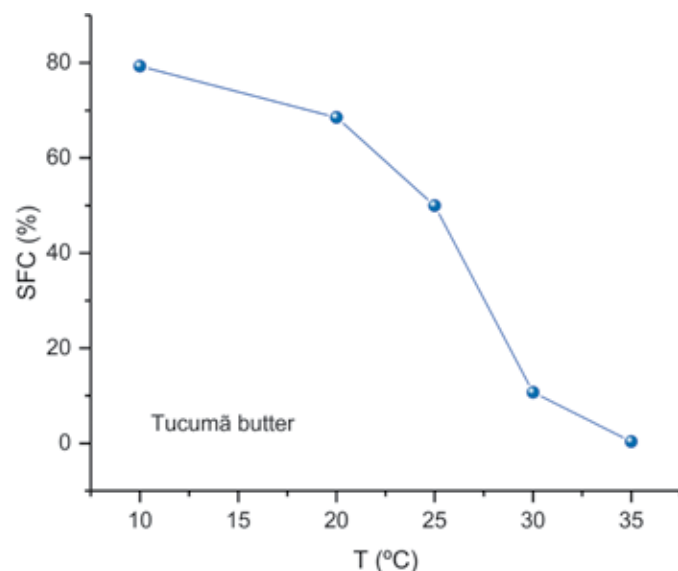
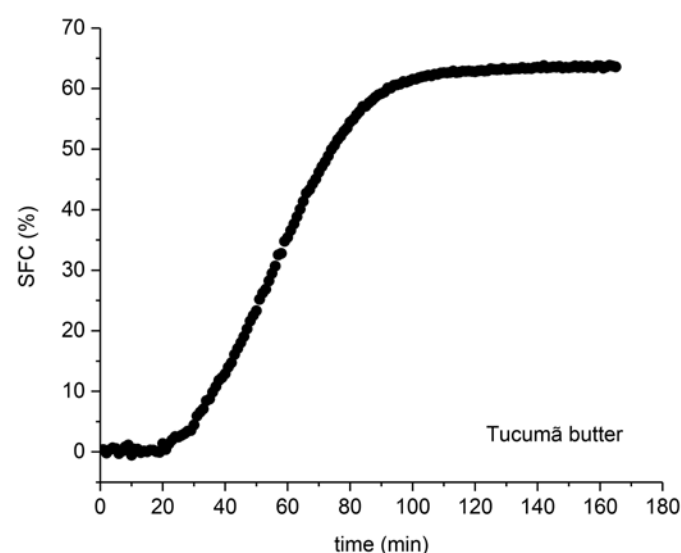**FIG. 4. Solid fat content profile (SFC) for tucumã butter**

FIG. 5. Crystallization isotherm at 20°C for tucumã butter

Tucumã butter is a perfect match for body temperature and can be applied to products that require this melting profile, such as coverings, chocolates, spreads, and ice creams.

The authors of this article are with Fats and Oils Laboratory— UNICAMP (University of Campinas), Brazil. For more information, contact Mayanny Gomes da Silva at mayannygomes@hotmail.com.

References

Alimentarius, C. (1999). Codex standard for named vegetable oils. *Codex stan, 210*, 1–13.

Bora, P., N. Narain, R. Rocha, A. De Oliveira Monteiro, and R. De Azevedo Moreira, Characterization of the oil and protein fractions of tucuma (*Astrocaryum vulgare* Mart.), *CYTA-J. Food 3*: 111–116, 2001, https://doi.org/10.1080/11358120109487654.

Cordenonsi, L.M., *et al.*, Amazonia products in novel lipid nanoparticles for fucoxanthin encapsulation, *AAPS PharmSciTech 21*: 32, 2020, https://doi.org/10.1208/s12249-019-1601-y.

Embrapa. (2018). Prosa Rural fala sobre as potencialidades do tucumã. <https://www.embrapa.br/busca-de-noticias/-/noticia/33036613/prosa-rural-fala-sobre-as-potencialidades-do-tucuma>. Accessed in October 2020.

Kamal-Eldin, A. (2005). Minor components of fats and oils. *Bailey's industrial oil and fat products*, https://doi.org/10.1002/047167849X.bio012.

Mambrin, M. and D. Barrera Arellano, D., Caracterización de aeites de frutos de palmeras de la región amazónica del Brasil, *Grasas y aceites*, 1997.

Pereira, E., *et al.*, Physical properties of Amazonian fats and oils and their blends, *Food Chem. 278*: 208–215, 2019, https://doi.org/10.1016/j.foodchem.2018.11.016

Rodrigues, C.E., F.A. Silva, A. Marsaioli, and A.J. Meirelles, Deacidification of Brazil nut and macadamia nut oils by solvent extraction: liquid–liquid equilibrium data at 298.2 K, *J. Chem. Eng. Data 50*: 517–523, 2005, https://doi.org/10.1021/je049687j

Santos, R.C., Sagrillo, M. R., Ribeiro, E. E., and Cruz, I. B. (2018). "The Tucumã of Amazonas—Astrocaryum aculeatum". In *Exotic Fruits* (pp. 419–425), Elsevier, https://doi.org/10.1016/B978-0-12-803138-4.00056-3

Estolides as environmentally friendly lubricants

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

Rebecca Guenard

In the early days of the green chemistry movement, when sustainability pioneers were attempting to wean the world off its dependence on petroleum, Mark Miller did not have much luck selling biodegradable and biobased lubricants. An entrepreneur and chemical engineer, Miller says that in the late 1990s, potential clients were reluctant to switch to plant-based products. Now, his products are so mainstream they are sold on Amazon. "Today, it is a no-brainer," he says. "Everybody gets it."

Green manufacturing experts believe they have finally reached a tipping point now that sustainability is accepted as a global priority. This is true across a variety of different market sectors. Personal care, in particular, has shifted to a primarily ecofriendly, sustainable industry with the majority of labels replacing their unsustainable ingredients and packaging. Even the plastics and elastomers industries, conceived out of petrochemicals over 50 years ago, increase the number of green formulations each year.

"Early on, there was resistance, but as technologies like ours have become more mature people are realizing these biobased, synthetic products work," says Miller, who credits household cleaners with helping consumers make the green shift. "Household cleaners have really shown consumers that sustainability does not mean that you have to give up anything."

One area where performance is crucial is lubrication. Lubricants act as a barrier between two sliding interfaces in contact with each other. The moving parts of machines cause friction that leads to wear, and will eventually fail without lubrication. A variety of petroleum-based formulas address the unique friction environment of specific applications, just as a car's engine and transmission use two different lubricants.

Extensive research on petroleum lubricants and their additives has led to product reliability consumers have grown to expect, hence the hesitation to adopt plant-based lubricants. However, technology developed by the US Department of Agriculture (USDA) has resulted in exceptional plant-based lubricants, known as estolides. "The industry has come such a long way that we are able to create sustainable, environmentally safe products that can compete on a performance basis with petroleum and even petroleum synthetics," says Miller, now CEO of Biosynthetic Technologies, based in Indianapolis, Indiana, USA. Through a Cooperative Research and Development Agreement (CRADA) with the USDA's Agricultural Research Service, Biosynthetic Technologies continues to develop estolide functionality for sustainable, ecofriendly consumer goods.
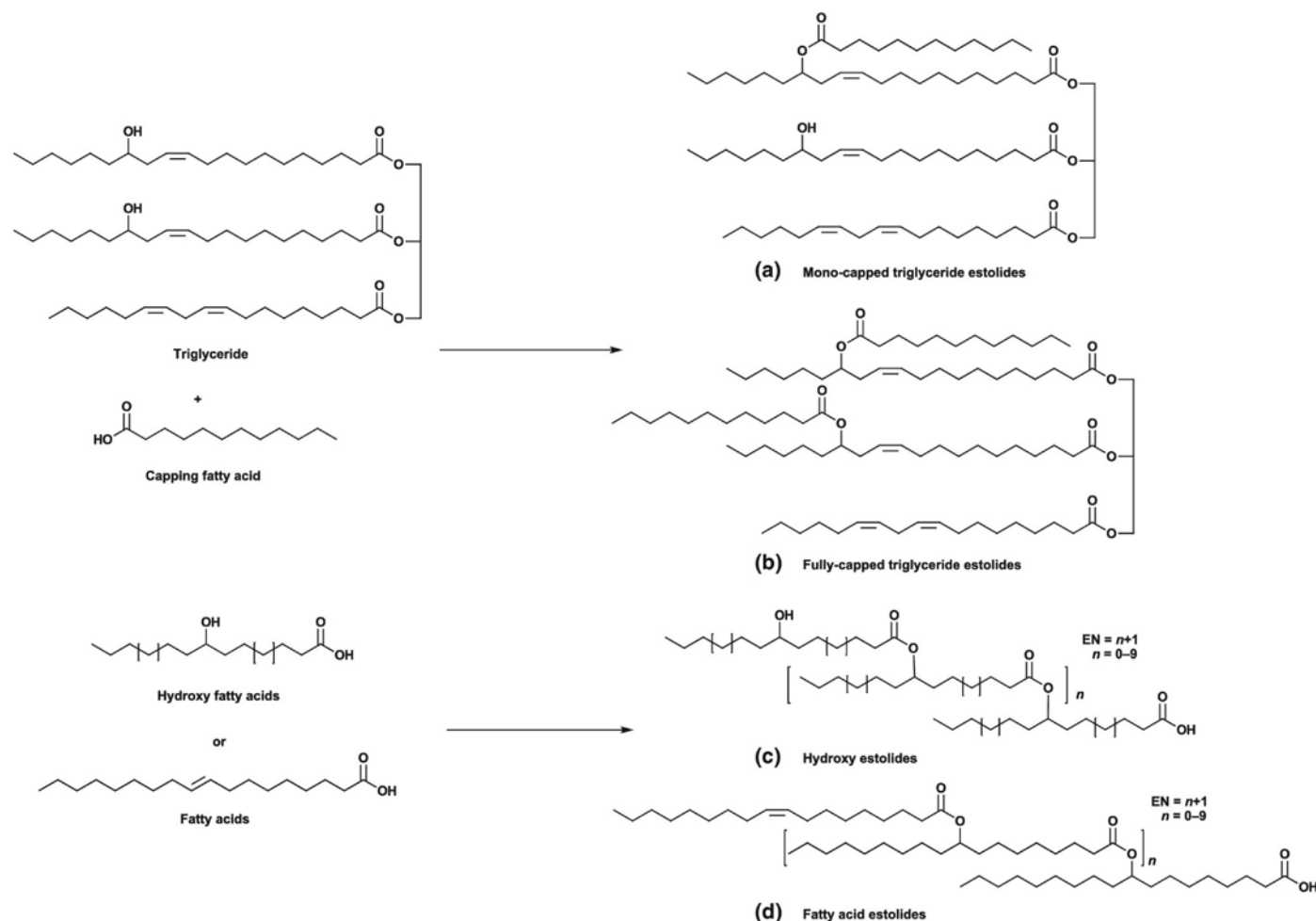
FIG. 1. Estolides synthesized from triglycerides (a,b) and fatty acids (c,d). Source: Yunzhi C., *et al., J. Am. Oil Chem'. Soc.* **97**: 231–241, 2020

Estolides are fatty acid esters that act as naturally occurring lubricants in a variety of plants and animals. In the human eye, for example, over two dozen estolide-producing glands in both lids make a wax ester that keeps tears from evaporating. Estolides can also be made synthetically by reacting vegetable oil triglycerides with fatty acids or by reacting two fatty acids, as long as one contains the necessary functional groups (Fig. 1).

Depending on the type of fatty acids used in the estolide synthesis, the resulting oil can be tuned for a range of physical properties. Researchers have produced new types of estolide lubricants by altering the chain length, molecular weight, degree of branching, or degree of oligomerization of the starting fatty acid. Since the synthesized molecule results in a secondary ester linkage, it is protected sterically from decomposition by heat or oxidation by water. Therefore, estolides exhibit exceptional stability, yet remain biodegradable. In addition, the compounds have low volatility, high viscosity, and act as natural detergents. Aside from lubricants, they are used in food, cosmetics, paints, coatings, rubber, and plastics.

Miller says they have made estolides from several sources, such as castor oil, soybean oil, and palm oil, as well as animal oils, such as fat and lard. They are also keeping track of the biotechnology associated with the production of algal oils in case it becomes an economically viable source. The company has developed an engine oil and a food-grade hydraulic fluid from high-oleic soybean oils, but Miller says he prefers making commercial products from the fatty acids of castor oil because it is highly sustainable, does not use GMOs, does not compete with food sources, and does not require deforestation.

"In India, where most castor is grown, it is the entry crop for the poorest class of farmers, creating an economics of sustainability," says Miller.

So how do estolide lubricants compare to petroleum when it comes to performance? Miller says their soy-based engine oils were tested in a fleet of Las Vegas taxi cabs. They drove 150,000 miles, and the parts came back pristine compared to the cabs with conventional petroleum oil whose parts came back gummed up with slug. A similar positive finding resulted in a study of the oil in US Department of Defense vehicles. He says his company's engine oil has been selling on Amazon for two years for use in commercial cars and trucks without any problems.

Aside from establishing the effectiveness of estolides as lubricants, Miller says Biosynthetic Technologies has shown that the compounds work well as emollients in cosmetics and hair-care. They are also exploring the UV adsorption properties of estolides for possible application in ecofriendly sunscreens. He says his research team has shown they could replace naphthenic oils in elastomers, rubbers, and tires, making these products less toxic and more biodegradeable. Currently, cost is prohibiting their entry into that market, since estolides are slightly more expensive than naphthenic oils. In addition, the company sees an opportunity to make plastics more sustainable by replacing current petroleum-based phthalates with estolide compounds. "Again, anywhere you are using petroleum-based products, you can substitute in estolides," Miller says.

Of course, consumer demand is not the only reason to transition bio-based lubricants into the mainstream. Government regulations have certainly helped the industry. In 2012, the US Environmental Protection Agency mandated that all merchant ships use environmentally acceptable lubricants. The Europe Union established criteria to place an Ecolabel on sustainable lubricant products. And, the USDA created the bio-preferred program, which mandates that federal agencies use bio-containing materials to support the agricultural community.

Miller says he would like to see more regulations aimed at protecting the environment from petroleum products. For now, his company is developing an engine oil by combining re-refined base oil with the more sustainable estolides that will improve the environmental impact of these lubricants. He feels positive that biolubricants will continue to overtake the lubricant and petrochemical industries.

"Today, major multinational companies are looking at themselves and asking, 'How can we be more sustainable,'" he says. He predicts continued growth over the next 10 years.

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

Information

Fatty acid estolides: a review, Yunzhi, C., *et al., J. Am. Oil Chem'. Soc. 97*: 231–241, 2020.

Nature-guided synthesis of advanced bio-lubricants, Romsdahl, T., *et al., Sci. Rep. 9*: 11711, 2019.

Physical properties of estolides and their ester derivatives, Isbell, T.A., *et al., Ind. Crop. Prod. 13*: 11–20, 2001.

Plant proteins for use in personal care products:
regulatory aspects

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

Karl Lintner

For more than 50 years, the cosmetic industry has been using naturally occurring proteins as ingredients in product formulations. To a large degree, these proteins were of animal origin, extracted with acids from bovine or fish skin (collagen, elastin) or obtained from sheep's wool (keratin), from bovine or donkey milk, horse blood serum (fibronectin), hen egg albumin, or possibly some other more exotic sources.

Most, but not all, of these proteins were subjected to hydrolysis *via* acid or enzymatic treatment, leading to a blend of protein fragments (sometimes referred to as "peptides") of variable size distribution, undefined amino acid sequences, and generally undefined characteristics. Two suppliers of

"hydrolyzed collagen" or "hydrolyzed milk protein" (very popular ingredients at the time) would not propose identical substances, given that acid strength, duration of hydrolysis, temperature, and down-stream processing might be substantially different. Specifications in the Certificate of Analysis

might have been large enough to accommodate both (or several) materials from different suppliers. The major concern for the formulators would have been low microbial count, solubility, odor, and color of the material. Only rarely were some of these proteins/peptides tested for sophisticated biological or physical benefits on the skin or hair.

Much has changed over the last 30 years. But then, as now, no harmonized global legislation on the manufacturing and selling of cosmetic products to consumers worldwide existed.

The term "cosmetic" itself is ambiguous. In the United States, it often refers mainly to "color cosmetics", i.e., makeup items (foundation, eyeshadow, lipstick, and so on), whereas shampoos and shower gels are labelled as "toiletries". More specialized formulations (wrinkle creams, moisturizers, hair straighteners, and the like) are placed in the categories of "skin care", "hair care", and "body care". Overlap between these categories occurs very often and renders market analysis rather difficult. Nevertheless, *all* these products are considered "cosmetics" under the Food, Drug, and Cosmetics Act of 1938, and are monitored by the US Food and Drug Administration (FDA). CFR Title 21 , Chapter 9, § 321 line (i) defines the nature and purpose of a cosmetic product: "*The term 'cosmetic' means (1) articles intended to be rubbed, poured, sprinkled, or sprayed on, introduced into, or otherwise applied to the human body or any part thereof for cleansing, beautifying, promoting attractiveness, or altering the appearance, and (2) articles intended for use as a component of any such articles; except that such term shall not include soap.*"

In this article, I shall use the terms "personal care" and "cosmetic" interchangeably, including all items that are thus defined by FDA and EU regulations. The following analysis will touch upon the two major markets (the United States and the European Union) and their regulatory situations which, alas, change rather often locally, even if the texts of the regulations are quite similar from region to region (United States, European Union, China, Korea, and Japan), creating some tricky traps for the unaware.

The present article is not destined to describe in detail all major cosmetic regulatory documents and rules. The purpose is to focus on the use of plant proteins for use in cosmetics (as defined by FDA, EU, Korean, or Japanese legislation). The reader is expected to know the basics of these rules for oils, lipids, and other ingredients.

The question is thus: Does there exist any specific legislation/regulation concerning the use of plant-derived proteins for use in cosmetic (personal care) products?

The short and easy answer to this question is: "No!" To the best of my knowledge, there is nothing special about plant protein's use in cosmetic formulations, compared to other proteins or any other cosmetic ingredients. End of story? Not quite. Let us look at some of the intricacies of this field.

WHAT IS A "COSMETIC INGREDIENT" AND HOW IS IT NAMED?

Curiously enough, there is no *a priori* definition of a cosmetic ingredient, except in the Chinese CFDA regulation (covered later in this section). Literally, anything can be used as an ingre-

dient in a cosmetic product *unless* it is prohibited as indicated in a country's regulation (included in a "negative" list of forbidden substances, such as the Annex II of the European Cosmetic Regulation).

Therefore, a substance becomes a "cosmetic ingredient" solely by being included in a formulation. For this to occur legally, the substance must not be on any "negative list" and must be listed on the consumer packaging in the "ingredients" list by the name it has been given in the INCI dictionary (International nomenclature of "cosmetic ingredients").

It is important to understand that the INCI document is *not* a "registration" or an authorization; all it represents is the (almost) globally harmonized *name* of the ingredient. The COSING data base (https://tinyurl.com/yy9mltnr) is the equivalent document in the EU. Japan and Korea have similar regulations.

For the last 10 years, China has closed the door to innovation in cosmetic ingredients of any type. It is the only country where 8,738 cosmetic ingredients are labelled as "permitted" to be used in a cosmetic, to the exclusion of every other possible substance or material. This Inventory of Existing Cosmetic Ingredients in China (IECIC), https://tinyurl.com/y57dfxry, undergoes extremely little change (only 4 or 5 new ingredients added in 10 years).

A plant protein proposed for inclusion in a cosmetic formulation must have an "INCI name"; for a new protein (not yet used in cosmetic products), the INCI name is acquired by submitting a dossier (precise or approximate chemical structure, brief manufacturing process description, purpose and function of the substance, suggested name) to the INCI committee. The suggested name is not always accepted by the committee, to the chagrin of the submitting entity. A lot could be said about the obscure and sometimes illogical decisions of this naming body, but that is not the scope of this article.

WHAT ARE THE SAFETY REQUIREMENTS?

Both consumers (and their advocates) and authorities want cosmetic products to be safe. The notion of "benefits vs risks" that applies in the pharmaceutical domain is generally not accepted for cosmetics. Although it is absurd to require zero risk for a cosmetic product which is impossible to attain, anyone providing cosmetic products to the public (free samples, products for purchase) has an obligation to evaluate and minimize risks to consumers.

In the United States, this requirement is alluded to in the official documents, but no specific rules are given about the methods and protocols employed to guarantee the safety of a consumer product. The responsibility (and liability) lies entirely on the shoulders of the provider (marketer) of the final products on the shelves.

In the EU, the Annex I document of the regulation is a detailed guideline of safety assessment. Each product put on the market must be accompanied by a PIF (product information file) which must contain all the data, arguments, and reports useful for asserting the safety. It is specified that in general "the safety of the product is based on the toxicological profile of the ingredients." Great, but what does that mean?

Thank you for supporting and partnering with AOCS

AOCS CORPORATE MEMBER 2021

AOCS Corporate Members: innovators and influencers — supply chain authorities for fats, oils and surrounding markets.

Current AOCS Corporate Members as of January 15, 2021.

Platinum

AAK

Archer Daniels Midland Co

Barrow-Agee Laboratories, LLC

Bunge Loders Croklaan

Cargill, Inc

Louis Dreyfus Co

Novozymes

Richardson International

Gold

Artisan Jet-Vac

Clariant (Mexico) SA de CV

Colgate-Palmolive Co

Crown Iron Works Co

Dallas Group of America Inc

Desmet Ballestra

EP Minerals LLC

Fuji Vegetable Oil Inc

Kao Corporation

Oil-Dri Corp of America

Stratas Foods

Wiley Companies

Silver

Ag Processing Inc

Alfa Laval Inc

Anderson International Corp

BASF Corp

Buss-ChemTech Ag

Canola Council of Canada

Catania Oils

CI SIGRA SA

Colonial Chemical Inc

Corbion

Corteva Agriscience

EFKO

Eurofins

Farmet AS

French Oil Mill Machinery Co

Hershey Co

Intertek USA Inc

Kalsec Inc

Kemin Industries Inc

LEEM Filtration

Lion Corp

Nisshin OilliO Group Ltd

Plus Group

PQ Corp

Procter & Gamble Co

Solex Thermal Science Inc

Wilmar International Ltd

Young Living Essential Oils

Bronze

American Emu Association

American Lecithin Co

Arkema Inc

Avatar Corp

Bioriginal Food & Science Corp

Caldic Canada Inc

Canadian Food Inspection Agency

Caribbean Products Co Ltd

Carribex SA

Center for Testmaterials BV

Darling Ingredients Inc

Epax Norway AS

Evonik Corp - Household Care

Golden Agri-Resources Ltd

Goulston Technologies

Henkel

Industrial de Oleaginosas SA de CV

Integro Foods Australia Pty Ltd

Intermed Sdn Bhd

ITS Testing Services (M) Sdn Bhd

JST Global, LLC

Kuala Lumpur Kepong Bhd

Larodan AB

Liberty Vegetable Oil Co

Lipsa SA

Lovibond North America

Lovibond Tintometer

Modern Olives

MSM Milling PL

Myande Group Co Ltd

Natec Network

Nippon Yuryo Kentei Kyokai

Nutriswiss AG

Pattyn Packing Lines NV

Peerless Holdings Pty Ltd

Pennakem Europa

Perimondo LLC

Perry Videx LLC

Pompeian Inc

Sanmark Ltd

Silverson Machines Inc

Simmons Grain Co

Testfabrics, Inc.

Tsuno Food Industrial Co Ltd

AOCS
Your Global Fats and Oils Connection

The Annex I spells out the type of toxicity studies that are recommended; the responsible person in the company must decide, as a function of the chemical nature of the ingredients, which studies are necessary and reasonable.

As long as finished products do not turn out to cause noxious effects on consumers, this practical approach is fine. In the case of sudden outbreaks (adverse effects) on people's skin or other body parts that can be traced to a specific ingredient, e.g., allergies sufficiently serious to attract the attention of the authorities, other questions will arise: What studies have been performed on the guilty ingredient? What were the results? Why has not more been done to avoid the problem, and so on …

WHO IS RESPONSIBLE FOR PROVIDING THE SAFETY DATA?

It turns out that the burden of proof for the safety of a final formulation, and thus the liability for any adverse effects, lies essentially with the formulating entity, not with the supplier of the ingredient. A supplier of chemicals (for whatever purpose the chemicals are destined) is subject to the EU regulation on chemical substances called REACH and TSCA (https://tinyurl.com/y5g2chyf) in the United States.

The supplier company is also bound by the rules of transport of dangerous substances. It must supply a material safety data sheet (MSDS) and any required warning labels, logos, and signs. The company must also respect the Globally Harmonized System (GHS) legislation on classification, labelling, and transport. The supplier is *not*—and cannot be—required to produce safety data of cosmetic interest for an ingredient, given that the supplier has no control over how the ingredient will be used in the final product! The MSDS is a general document which does not take specific uses for whatever purpose into account.

In practice, however, it is almost impossible for a supplier of an ingredient destined for cosmetic usage to sell material without a minimum of safety data. The extent and scope of a safety file can be negotiated between supplier and buyer. For example, if an ingredient will be used in a foot cream, they may negotiate such details as whether one or two genotoxicity studies is needed, if a 90-subchronic toxicity study should be done, or if ocular irritation is a relevant parameter. Buyers pressure and make demands about the extent of the safety profile supplied by the vendor without a legal basis for those demands. The motto of the EU's REACH exercise, "No data, no market," seems to have been adopted by the formulators.

What all of this means is that a plant protein proposed to the cosmetic industry as an ingredient will have difficulty finding buyers without supplying a safety profile for the material. A succinctly edited MSDS with little data will usually not be sufficient. The amount and scope of safety data that the supplier should provide is not specified by legislation. Common practice suggests skin and eye irritation, and sensitization (next section) as the basic requirement. For plant proteins, additional data (mutagenicity, genotoxicity, chronic toxicity, and so on) are more rarely requested and should require justification based on perceived risks.

SENSITIZATION AND ALLERGY RISK

The term sensitization refers to the acquisition by the body of an allergic reaction to a specific allergenic/sensitizing substance. It should not be confused with sensitivity, which is a usually harmless reversible reaction (redness, slight itching) to a formulation. However, a great number of chemical families of substances are known to be sensitizing on the skin and should be avoided as much as possible in cosmetic products. Contrary to irritation (which is often misinterpreted to be an "allergy" by consumers with "sensitive" skin), sensitization is generally not reversible. Skin that has been sensitized by a substance will react increasingly strongly with each new contact with that substance, in rare cases leading to severe health problems. Certain surfactant substances of amphiphilic nature have a tendency toward allergenicity. The complexities of this risk were discussed in detail at the international symposium, "Sensitizing Properties of Proteins," held in April 2012, in Prague, Czech Republic (https://tinyurl.com/yamjpbwg). Proteins, particularly plant proteins, are known to present a certain level of allergic risk that could sensitize susceptible skin to the protein. The "gluten-free" buzz is a case in point.

As mentioned before, while concerned with consumer safety, neither the FDA nor the Scientific Committee on Consumer Safety (SCCS) of the European Union specify beforehand which ingredients are allowed in a cosmetic or precisely how their safety should be evaluated and assessed; but both organizations keep an eye on the matter. A good overview of this topic is found on the Personal Care Products Council (PCPC) website (https://tinyurl.com/y43wo7uw).

In the United States, the Cosmetic Ingredient Review Committee (CIR) is a voluntary emanation of the Personal Care Product Council (PCPC, ex CTFA) composed of independent toxicologists, dermatologists, and chemists, where a representative of the FDA is present at committee meetings and appraised of the committee's work. Based on alerts or its own perception of risks, the committee selects ingredients found in cosmetic formulations and carries out a thorough safety analysis, considering not only all available safety study reports supplied by the industry (both suppliers and formulators) but also those read across references, use level in the formulations, and sites of application on the body. The outcome of such risk analysis is published in the *Intl. Journal of Toxicology*. There are several possible outcomes, including: safe for cosmetic use at the use level of … x%, safe with qualifications, insufficient data, and unsafe.

As an example, the CIR status report on the "Safety Assessment of Plant-Derived Proteins and Peptides as Used in Cosmetics" of Oct. 2017 (https://tinyurl.com/y4wzvysz) concludes that the 18 (hydrolyzed) plant proteins and peptides tested were all safe for use in the conditions described in the various documents, with two caveats: 1. hydrolyzed wheat protein and hydrolyzed wheat gluten were safe with the qualification of not exceeding 3,500 daltons of average molecular mass and 2. Data on hydrolyzed maple sycamore protein are insufficient to determine safety.

A CIR report conclusion of "insufficient data" for an ingredient has two consequences: 1. It alerts the FDA to a potential

problem of an insufficiently tested ingredient and 2. The formulation containing the ingredient may be required to label the cosmetic product with a phrase such as, "This product contains an ingredient that has not been fully tested for safety." (Not the best advertising slogan!)

The *only* regulatory issue that applies specifically to plant proteins and peptides concerns the average molecular mass of the hydrolyzed protein fragments (peptides) of wheat and wheat gluten: They are considered safe provided this value does not exceed 3,500 daltons. This requires suppliers to check for this parameter, preferably for each batch. Although there is no prohibition against using higher molecular weight protein fragments, the risk of allergic reaction increases with increased size of the protein; as the example of the Hydrolyzed Maple Sycamore Protein shows, its status of "insufficient data" in the CIR assessment requires any product using this material to alert consumers to this safety concern.

The suppliers of hydrolyzed proteins and the formulators using them appear to have taken this molecular size limitation to heart, not only for wheat proteins, but as a general recommendation for all proteinaceous material. Exceptions do exist, though.

PLANT PROTEINS: EFFICACY

Once the safety of a plant protein has been determined, it is time to consider the function and usage that the protein will display in the final formulation. Are there specific restrictions to the activities and functions plant proteins may exert and claim in consumer directed advertising?

The answer is a resounding "No." Like any other cosmetic ingredient, plant proteins and peptides may possess and display physical benefits (e.g., moisturization, superficial skin tightening, hair straightening, hair protection) and/or biological benefits (antioxidant, protein synthesis, enzyme inhibition, and many more bioactivities).

Specifically in the US market, the crucial factor is the language (the "intention") of the publicly made claims of these activities. In brief, claims on physical activities that do not change the nature and function of the body are generally allowed if they are true (based on credible data).

Claims of biological benefits in skin care and body care applications are subject to FDA scrutiny and may be viewed as drug claims (https://tinyurl.com/yy7om4ax), hence rejected on a cosmetic. Information on claim substantiation methods and language can be found in a variety of sources (see "*Inform*ation about claim substantiation")

With respect to Korean and Japanese markets, the nature of the claims of efficacy determines the type of consumer product regulations. Any claims of UV protection, skin lightening, or antiwrinkle efficacy puts a product into the "functional cosmetics" category in Korea (https://tinyurl.com/y2pfedjc).

Very similar rules exist in Japan, where such specific claims lead to a product's classification as Quasi Drug, with specific regulations (https://tinyurl.com/y3l4hysq). Again, all this applies to all potential cosmetic ingredients, and plant proteins/peptides are not singled out in any way to the best of my knowledge. Another type of regulation protocol covers biological materials (plants, algae, microorganisms, and genetic assets…) obtained from natural sources: the Nagoya protocol on biodiversity. Plant proteins have no special status but are subject to the protocol's rules for any usage (https://www.cbd.int/abs/).

TO SUM IT ALL UP

The cosmetic industry is a peculiar branch of industrial activity. In some ways, it is totally free to be creative and dynamic. In other ways, it is highly overregulated for no apparent reason. Navigating the various administrations, regulations, rules, and recommendations can seem difficult. The present article scratches only the surface of some arcane or recent regulations, but I hope it has shown that the use of plant proteins in cosmetic formulations does not face specific higher regulatory obstacles than any other ingredient destined for creams, serums, gels, shampoos, varnish, makeup, lipsticks, or other beauty products.

Karl Lintner obtained a Chemical Engineering degree and a PhD degree in Biochemistry from Vienna University (Austria). After 10 years of research on biological peptides at the Nuclear Research Centre in Saclay, France, he became Laboratory Manager, then Marketing Manager (product development and worldwide technical support) at Henkel Co., Düsseldorf, Germany. He joined SEDERMA S.A.S. (France) in 1990 as Technical Director and later headed the company for 10 years as Managing Director/CEO. Currently, Karl Lintner is an independent consultant to the cosmetic industry (President of KAL'IDEES S.A.S.).

He has filed several dozens of patents, published numerous articles and book chapters on cosmetic ingredients, and is an active member of SCC (US) and SFC (France). He served five years as Editor-in-Chief of the International Journal of Cosmetic Science. *He was awarded the prestigious Maison de Navarre Medal of the SCC in 2012, and the In-Cosmetics Lifetime Achievement Award in 2013. Karl Lintner can be contacted at karl.lintner@kalidees.com.*

*Inform*ation

ABOUT CLAIM SUBSTANTIATION

[1] https://cosmeticseurope.eu/files/4016/0015/2480/Guidelines_for_Cosmetic_Product_Claim_Substantiation.pdf

[2] https://www.cosmeticsbusiness.com/news/article_page/How_to_substantiate_cosmetic_claims/151883

[3] https://www.kobo.com/nz/en/ebook/global-regulatory-issues-for-the-cosmetics-industry-3

Meet Mike Williams

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

Mike Williams beside his favorite pinball machine, which he fully restored to "better than new condition" in 2020

Fast facts

Name	Mike Williams
Joined AOCS	2005
Education	M.Sc. from the University of Oregon (Eugene, Oregon, USA)
Job title	Research, Development & Innovation group leader, Care Solutions, North America
Employer	Evonik Corp., Milton, Wisconsin, USA
Current AOCS involvement	Vice-chair, Surfactants and Detergents Division

PROFESSIONAL

What's a typical day like for you?
My new COVID--times day looks a lot different than my pre-COVID day. Currently, I am at the lab one to two days per week making sure that my group has what they need to get their jobs done, touching base to help solve any chemistry or mechanical issues, interacting with our plant personnel to see what has been happening in the field, and going through the labs to make sure everyone is following our COVID-19 protocols. The other days, I head down to my basement to settle in for a day of remote video interaction.

Flash back to when you were 10 years old. What did you want to be when you grew up?
My mom was the ultimate record-keeper; she kept everything, including my "baby book." When one thumbs back to look at what I wrote I wanted to be, starting in second grade (and ever after) you will find the word "chemist."

Why did you decide to do the work you are doing now?
Before I joined Tomah Products in 1998, I was working at a contract research organization. I decided I wanted to get out of pharmaceuticals and go for something with more long-term stability, so I left Illinois and moved to Wisconsin to join Tomah Products. We've been through four name changes in my time here, but I have been doing (approximately) the same job ever since Tomah bought the Reserve, Louisiana, USA, alkoxylation plant from Shell in 1999.

What event, person, or life experience has had the most influence on the direction of your life?
I had a chemistry teacher in high school who was the kind of teacher that everyone fears. She was a first-year teacher who did a terrible job of explaining concepts. I remember early on in the first quarter when I challenged her on the first question of her test and why her chemical stoichiometry was wrong, which meant all her subsequent answers were wrong. We did the math together in front of the class and showed how my logic was right. From then on, I took it as a personal challenge to know more about whatever topic we were covering than Ms. T. knew. This meant I spent hours each night working through chemistry problems or reading what I could find in the library. She was gone after one year, but I still found myself digging in at the library as much as possible. This experience ultimately helped me see the need for choosing a college and grad school where I enjoyed working with the professors.

PERSONAL

How do you relax after a hard day of work?
I collect and restore pinball machines. Rebuilding drop targets, pop bumpers, and flippers can be an exercise in three-dimensional thinking. However, the most challenging part about restoring machines often is working through any circuit board issues. When a machine sits untouched in someone's basement for years, the moisture can wreak havoc on the circuits.

YEARS OF MEMBERSHIP

aocs.org/membership

25–29

Okey Abara
Syed Husain Abbas
Ruben Abril
S. Patrick Adu-Peasah
Naseer Ahmed
Jocelyn D. Alfieri
Archer Daniels Midland Co
Hector Carlos Autino
Eddie L. Baldwin
Constantin Bertoli
Bryan L. Bertram
Douglas M. Bibus
Bioriginal Food & Science Corp
Scott D. Bloomer
William L. Boatright
Patrick E. Brown
William C. Byrdwell
Mark S. Caddigan
Charles D. Carr
Carlos Caruso
Douglas Cater
Meng-Tsan Chiang
Raj K. Chopra
Crown Iron Works Co
Lorin R. De Bonte
Lorenzo De Ferra
Wim De Greyt
Gerardo de la Piedra
Bernd W. K. Diehl
Michael J. Dobson
David A. Dolde
Michael K. Dowd
Robert O. Dunn, Jr
Paresh Dutta
Bert Engelhard
Larry C. Erickson
Andrew M. Evans
Fabio Favati
Tripp Fezler
Kelley C. Fitzpatrick
Ralph Franklin
Satoshi Fujita
Sam F. Gabriel
James F. Gadberry
Terri Germain
Nadir B. Godrej
David Grande
Stephen F. Gross
Mark A. Gulden
Barry M. Gursky
Clifford A. Hall, III

C. Ross Hamilton
Steven L. Hansen
Richard W. Hartel
Gregory G. Hatfield
Douglas G. Hayes
Jan P. Hazebroek
Richard W. Heiden
Jeffrey L. Henderson
Ernesto Hernandez
Maria Lidia Herrera
Steve A. Howell
Keum Taek Hwang
Amadeo Ibarra-Hallal
Richard Ihasz
Katsumi Imaizumi
Robert K. Johnson
Howard Jordi
Angeles Kantun
David R. Karsa
Marc J. Kellens
Phillip S. Kerr
In-Hwan Kim
Chong Kim Fook
Howard R. Knapp
Kazunori Koba
Dana Krueger
Michael K. Landis
Lee B. Lasher
Patrick J. Lawler
Eric L. Lien
Keshun Liu
Linsen Liu
Lovibond Tintometer
Hector A. A. Maduro
Maria Makulska
Timothy E. Maneely
Alejandro G. Marangoni
Felix Maritano
John L. Massingill, Jr.
Alison Jane Mathews
Shuichi Matsumura
Thomas P. Mawhinney
Thomas E. McBrayer
Frank P. McGovern
Juan Jose Mendez
Luis R. Mercado
Jens Mesa-Dishington
Serpil Metin
Jurgen O. Metzger
Clarence A. Miller
William S. Minor
Ann Moser

Magdi M. Mossoba
Mototake Murakami
Dilip K. Nakhasi
Ramanathan Narayanan
Ted Neuman
Gregg Newman
Ian S. Newton
Peter D. Nichols
Anthony Nock
Daisuke Ono
Pattyn Packing Lines NV
Perry Videx LLC
Norman E. Pfau, III
Ricardo R. Pollak
Alberto J. Pulido
John N. Rapko
Miguel Redon
Johanna D. Reske-Schumann
John M. Reuther
Mian N. Riaz
William C. Ross
Derick Rousseau
Gisbert Schall
Charles N. Serhan
Prasert Setwipattanachai
Rajesh J. Shah
Fereidoon Shahidi
Dov Shaked
Yuji Shimada
Leif Skibsted
Philip G. Sliva
George A. Smith
Robert L. Stroup
Adolfo T. Subieta
David T. Syme
Jorge R. Tanoni
Jorge F. Toro-Vazquez
Thomas Traveria Casanova
Renato D. Traversa
Masaki Tsumadori
Robert T. Tyler
Mauricio Vanegas
Tong Wang
D. Lee Warren
Michael K. Weibel
Michael T. Woolsey
Xuebing Xu
Samuel Owusu Yeboah
Bryan V. Yeh
Petrus Zaal

30–34

American Lecithin Co
Yasuhiro Ando
Franklin I. Auerbach
Orivaldo Balloni
Philip C. Benes
Robert T. Betz
David D. Brooks
Cesare Carabelli
Cheah Ping Cheong
K. B. Albert Chiu
Jerry H. Collins
Thomas G. Crosby
Jane B. Crowther
Richard A. Della Porta
Albert J. Dijkstra
Erich Dumelin
Yasushi Endo
Felix E. Escher
Shiv B. Goenka
Allan Green
Mats Hamberg
Charles E. Hammond
Michael A. Hawkins
Chris Heeb
Alberto F. Hidalgo Sr
Steven E. Hill
David C. Hoffsten
Milagros P. Hojilla-Evangelista
Alice P. Hudson
Ambrose J. Hugo
Huntsman Corp
Charles R. Hurburgh, Jr
Peter J. Huth
ITS Testing Services (M) Sdn Bhd
Timothy G. Kemper
Jerry W. King
Mark R. Knobbe
Sri Parkash Kochhar
Randal C. Kruger
Kuala Lumpur Kepong Bhd
Oi Hian Lee
Matthew Levinson
Bertha Lopez
Alastair K. H. MacGibbon
Daniel J. MacLean
F. Xavier Malcata

Rolf Mantei
Araki Masuyama
Mark Matlock
Dave B. McCall
Trevor W. Meredith
Keith A. Meyer
Martin Mittelbach
Kazuo Miyashita
Teruo Miyazawa
Saeed H. Mohseni
Robert Moreau
Trevor A. Mori
James R. Munson
Deland J. Myers, Sr
Toro Nakahara
John B. Ohlrogge
Hernan D. Paredes
Seok Kwan Park
Michael Piatko
Georg J. Pollert
Colin Ratledge
Sreeram K. Reddy
Jarrow L. Rogovin
Ralph C. Romero Sr
Harold R. Russell
W. Warren Schmidt
Philip L. Schmit
J. Duke Seibold
Leonard M. Sidisky
Randolph Sidoo
Luis Spitz
John R. Tengwall
Thomas R. Tiffany
James G. Timourian
Gregory van Buskirk
Philippe Van Doosselaere
Ken E. Visek
Gregory P. Waranica
Upali Weerasooriya
Randall J. Weselake
Ingmar Wester
Gordon L. Whitbeck, MS
J. Matthew White
Hans-Jurgen Wille
Arthur J. Wolf
Teruyoshi Yanagita

❝The best part of being an AOCS member is the networking and the quality of people that you meet. Membership in AOCS is important to me because of the opportunities it has provided to me over the course of my career in terms of new ideas to develop new products for chromatography.❞

—LEN SIDISKY, R & D MANAGER, MILLIPORESIGMA, PENNSYLVANIA, USA

❝I know there is a lot of competition for joining of trade organizations, but I really think AOCS is one of those organizations that you have to join, especially if you are in the fats and oil sector. Everybody is here. We are one of the oldest, most established and most credible organization around and you get a lot of benefit for your dollar with AOCS.❞

—DOUG BIBUS, LIPID TECHNOLOGIES, LLC, MINNESOTA, USA

OCS membership year-after-year?

Ask one of our long-standing members.

Since 1909

35–39

Ismail H. H. Abdalnabi
Yvonne V. Agustin
Casimir C. Akoh
David J. Anneken
Rob Ariaansz
Kenneth E. Beery
Philip A. Bollheimer
Harald Breivik
Nestor M. Carballeira
Pete C. Cartwright
John P. Cherry
Michael F. Cox
A. Saari Csallany
Christopher L. G. Dayton
Etienne M. Deffense
Wyatt N. Elder, Jr
Raul O. Ferrari
Murray Flashner
Alyce D. Fly
Knud A. Gerstenberg
Harish Chandra Goyal
Monoj K. Gupta
Frank M. Hahn
Gudmundur G. Haraldsson
David F. Hildebrand
Dennis H. Hogan
Hudson Tank Terminals Corp
J. Edward Hunter
Lucky Inturrisi
Yutaka Itabashi
Foon Wing Kam
Kevin M. W. Keough
Dharma R. Kodali

Semih Sefa Koseoglu
Rodney Kuss
Firouz Madadnoee
Rodney J. Mailer
Ted Matson
Richard E. McDonald
Ammanuel Mehreteab
H. Steven Misner
Albert F. Mogerley
Hans Nieuwenhuis
Kari V. V. Nurmela
Richard J. Otterson
Peerless Holdings Pty Ltd
Guy L. Posschelle
Andrew Proctor
Ian Purtle
Julius Rath
Lars Reimann
Pravit Santiwattana
Kiyotaka Sato
Edward T. Sauer
Edward S. Seguine
Bernard C. Sekula
Arnon Shani
Vijai K. S. Shukla
Patrick J. Slonecker
Jennie M. Stewart
Barrie Tan
Sanjay N. K. Trivedi
Frank Veldkamp
Earl D. Weak
Michael J. Wint
Gow-Chin Yen

40–49

Jeannene A. Ackerman
Richard Adlof
Moghis U. Ahmad
Carl Amos
George E. Anderson
Rajindra Aneja
William E. Artz
Ardin L. Backous
George T. Battaglini
W. Maurice Belcher
Marjorie L. Besemer
Michael J. Boyer
Dennis J. Breitbart
Francis R. Cala
Edward J. Campbell
Kenneth F. Carlson
Roy A. Carr
Douglas A. Cavanagh
John L. Cayias
Edith J. Conkerton
Barrie F. Cruickshank
Michael F. Czuczak
Levente L. Diosady
Aris J. Domnas
James H. Dyck
Edward Emken
John W. Erdman, Jr
N. A. Michael Eskin
Jeffrey Fine
Frank J. Flider
David G. Forster
Daniel P. French
Floyd E. Friedli
Stewart E. Gloyer
Manuel Gomez Ojeda
Stephen M. Greenberg
Mumtaz Haider
Gerard L. Hasenhuettl
William J. Hausmann
William Blake Hendrix
Bengt Herslof
Yoshio Hirabayashi

Kian Hock Ho
Sandra Holloway
Andre Huyghebaert
Lawrence A. Johnson
Edward H. King, Jr
Apostolos K. Kiritsakis
John K. G. Kramer
Andrew T. Kwong
Liberty Vegetable Oil Co
Philip A. Lofgren
Stanley C. Loft
Edmund W. Lusas
Douglas E. McLemore
Carter G. Naylor
Douglas V. Okonek
Anthony O'Lenick
Frank T. Orthoefer
Durk Pearson
Marshall Pike
Roberta L. Richards Owens
James M. Richmond
Kenneth F. Schoene
Walter A. Shaw
Rodolfo Solis O.
Carlos E. Soza Barrundia
Dennis K. Strayer
Thanakorn Vegetable Oil Prods Co Ltd
Prakit Triyanond
Ernie H. Unger
Werner E. Wagner
Gary C. Walker
Peter J. Wan
Kathleen Warner
Pamela J. White
Neil R. Widlak
James E. Willits
Richard F. Wilson
Walter J. Wolf
Suk-Hoo Yoon
George J. Zimmerman

50+

Phillip G. Abend
Don Banks
Joyce L. Beare-Rogers
Jose Becerra Rique
Daniel R. Berger
Paul D. Berger
Giovanni Bigalli
Anthony P. Bimbo
Raymond Bistline
Ram Blau
Dean K. Bredeson
Donald E. Britton
Glenn D. Brueske
Arnold Carsten
Jorge J. Castellanos
John H. Chaloud
Raoul J. P. Clarenc
Samuel Cohen
Jesse E. Covey
Richard Keith Downey
LeRoy Dugan, Jr
Jacqueline Dupont
Harold P. Dupuy
Manuchehr Eijadi
Joseph G. Endres
Giles Farmer
George C. Feighner
William H. French
Martin E. Ginn
Linsley S. Gray
Edward L. Griffin, Jr
William M. Grogan
Frank D. Gunstone
Robert C. Hastert
John E. Heilman
Murray Heimberg
Robert J. Hlavacek
Allen Howland
James M. Iacono
Glen Jacobson
Keith L. Johnson
Eric Jungermann
Nat Kessler
John S. Kilpatrick
Ernest F. Kopka

R. G. Krishnamurthy
Kenneth A. Kuiken
Arnis Kuksis
Robert E. Landers
Edward Latondress
Marcel S. F. Lie Ken Jie
Gary R. List
Norman Lovegren
Andrew M. Lubienski
Theodore K. Mag
Vaidyanath Mahadevan
Helmut K. Mangold
John F. Marsden
John R. Mitchell
Don T. Morton
John H. Nelson
Nippon Yuryo Kentei Kyokai
Richard A. Ormsbee
Conrad W. Petersen
Robert M. Pierce
James B. M. Rattray
Louis J. Roman
James L. Roorda
Logan Roots
Maurice Rosenthal
August M. Rossetto, Jr
Howard Roth
Werner H. Schmidt
William A. Singleton
Endre Sipos
Stanley Smith
Paul Sosis
Michihiro Sugano
Jack Suriano
Aloys L. Tappel
Joseph Topps
Michael L. Valletta
John Van Haften
Russell C. Walker
Arthur E. Waltking
Leamon D. Williams
F. Vernon K. Young
Gerry C. Zekert

AOCS
Since 1909
Your Global Fats and Oils Connection

PATENTS

Bio-based thermosets

Webster, D.C., *et al.*, NDSU Research Foundation, US10730998, August 4, 2020

The invention relates to novel curable bio-based thermosetting compositions of a) at least one highly-functional bio-based epoxy resin; and b) at least one carboxylic acid crosslinker selected from the group consisting of a water-soluble multifunctional carboxylic acid, a dicarboxylic acid oligomer from a melt reaction between a diol and a dicarboxylic acid or its anhydride, and mixtures thereof and which are curable with water, an alcohol or a water and alcohol mixture. The bio-based thermosets of the invention may be free of VOCs and/or organic solvents. The bio-based thermosets of the invention can be used to form coatings, composites, adhesives, and films. Methods of making the bio-based thermosets of the invention are disclosed as are coating compositions and coated objects, such as coatings, composites, adhesives, and films, using the bio-based thermosets of the invention.

Cargill An AOCS Platinum Level Corporate Member

Polymerized oils and methods of manufacturing the same

Kurth, T., *et al.*, Cargill, Inc., US10731037, August 4, 2020

Described herein is a polymerized biorenewable, petroleum based, previously modified, or functionalized oil, comprising a polymeric distribution ranging from about 2 to about 80 wt % oligomer content, a hydroxyl value ranging from about 0 to about 400, and an iodine value ranging from about 0 to about 200. Methods of manufacturing the polymerized oil as well as its incorporation into asphalt paving, roofing, and coating applications are also described.

Non-hydrogenated hardstock fat

Van Horsen, D.S.H., *et al.*, Upfield Europe B.V., US10757953, September 1, 2020

Hardstock fat having an amount of saturated fatty acids of at least 80 wt %, wherein the amount of H3mixed triglycerides is between 45 and 60 wt %, H3mixed being a group of triglycerides consisting of triglycerol esters of 3 H fatty acids wherein H is a saturated fatty acids having a length of C16 and/or C18, wherein the triglycerol ester is composed of two different fatty acids; and wherein the amount of H2Xmixed triglycerides is at least 10 wt %, H2Xmixed being a group of triglycerides consisting of H2M triglycerides and H2O triglycerides, wherein H2M triglycerides are triglycerol esters with two H fatty acid and one M fatty acid wherein M is a saturated fatty acid of a length of C12 and/or C14, and wherein H2O triglycerides are triglycerol esters with two H

fatty acid and one O fatty acid wherein O is a unsaturated fatty acid of a length of C18, wherein the H2M and H2O triglycerides are composed of three different fatty acids.

Lubricating oil compositions comprising a biodiesel fuel and a Mannich condensation product

Moniz, M.A.S., *et al.*, Chevron Oronite Company LLC, US10781394, September 22, 2020

This invention encompasses lubricating oil compositions comprising a base oil, a biodiesel fuel, and a Mannich condensation product. A method for inhibiting viscosity increase in a diesel engine fueled at least in part with a biodiesel fuel is also described.

BUNGE Loders Croklaan An AOCS Platinum Level Corporate Member

Process for extraction of oil from algal biomass

Dayton, C.L. G., *et al.*, Bunge Global Innovation LLC, US10731037, September 29, 2020

Provided here is an enzymatic process for extraction of oil from algae biomass. The oils produced by the processes herein are used in animal feed and human consumption.

Method for making a solid-supported phospholipid bilayer

Cho, N.-J., Nanyang Technological University, US10787470, September 29, 2020

A method of preparing a solid-supported phospholipid bilayer is provided. The method includes a) a first step of providing a solution comprising a bicellar mixture of a long-chain phospholipid and a short-chain phospholipid; b) at least one second step of decreasing the temperature of the solution to below 0°C., increasing the temperature to above room temperature and causing the solution to be blended; and c) a third step of depositing the solution obtained after the second step on a surface of a support, wherein the concentration of the long-chain phospholipid in the solution is at most 0.1 mg/mL, for obtaining a solid-supported phospholipid bilayer. A solid-supported phospholipid layer obtained by the method as defined above is also provided.

Potassium soaps that can be thickened with chloride salt

Smith, S.A., Vanguard Soap LLC, US10793808, October 6, 2020

Natural liquid potassium soap compositions and methods of manufacturing and using the same are provided with thickening by the addition of chloride salts, such as sodium chloride and potassium chloride. The natural liquid potassium soap compositions may contain one or more fatty acids with carbon length ranging from four (C4) to 22 (C22) or natural fatty acid mixtures with coco-

nut oil, olive oil, tallow, sunflower oil, safflower oil, and/or tall oil fatty acids which are saponified with lye. The saponification lye is preferably potassium hydroxide. Preferred embodiments contain potassium salts of fatty acids comprising at least oleic acid (C18:1 cis-9), olive oils, coconut oils, or mixtures thereof. The chloride salt is added in either solid or liquid form following saponification and neutralization.

Liquid phospholipid-containing compositions for the preparation of pharmaceuticals

Hupfeld, S., *et al.*, Aker BioMarine Antarctic AS, US10806742, October 20, 2020

Current krill oil extracts are liquid at room temperature and include only about 45–55% phospholipids. As the purity of phospholipids increases, the extracts become more viscous, making them difficult to handle and process. The invention mixes these pure materials with viscosity-reducing agents to make them amenable to processes such as encapsulation. Thus, the invention retains the useful liquid characteristics of known krill extracts while providing much higher concentrations of phospholipids.

Saturated triglyceride-containing rubber composition, tires and tire components containing the rubber composition, and related methods

Galizio, B.C., *et al.*, Bridgestone Americas Tire Operations, LLC, US10808106, October 20, 2020

Disclosed herein are rubber compositions containing a saturated triglyceride component of specified melting point, as well as tires and tire components containing the rubber composition. Also disclosed are methods for improving the performance of a tire tread (such as by improving the wet traction) containing the rubber composition.

Hydrolyzed pea protein-based nutrient composition

Laver, R., *et al.*, Kate Farms, Inc., US10806169, October 20, 2020

The present invention discloses a nutritional composition and method of using and making the nutritional composition. The nutritional composition is a hydrolyzed pea protein based nutrient composition for use in both enteral and oral feeding, and provides a non-allergenic diet for providing optimal nutrition to users. The nutritional composition is made from organic and plant-based ingredients. The nutritional composition has pea protein hydrolysates, phytochemical extracts, fatty acids, organic ingredients free of the top eight allergens and corn, and prebiotic fiber. The nutritional composition is provided in liquid form for enteral and/or oral feeding.

In-line degumming and neutralization of oils and fats using hydrodynamic flow-through cavitation reactors

Kellens, M., *et al.*, N.V. Desmet Ballestra Engineering S.A., US10808202, October 20, 2020

A process for the combined degumming and neutralization of an oil containing phosphatides and FFA, the process comprising: dosing an aqueous degumming reactant into the oil to form a mixture; introducing the mixture into a first set of flow-through hydrodynamic cavitation reactors to form at the exit of the first set of flow-through hydrodynamic cavitation reactors a mixture; dosing, at the exit of the first set of flow-through hydrodynamic cavitation reactors an aqueous caustic solution into the oil to form a mixture of aqueous caustic dispersed into the oil; introducing the mixture of an aqueous caustic dispersed into the oil into a second set of flow-through hydrodynamic cavitation reactors to form a mixture of oil containing hydrated phosphatides and neutralized FFA (soaps); and separating the mixture of oil containing hydrated phosphatides and soaps to obtain an oil phase and an aqueous phase.

Production of fatty acid alkyl esters with caustic treatment

Rancke-Madsen, A., *et al.*, Novozymes A/S, US10815506, October 27, 2020

A method for producing fatty acid alkyl esters, comprising providing a system comprising an oil phase/hydrophobic phase an a hydrophilic phase, and reacting a fatty acid feedstock present in said oil phase/hydrophobic phase with alcohol in the presence of water and one or more lipolytic enzymes.

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.

Most-cited AOCS journal articles from 2018 to 1st quarter 2021

Which AOCS journal articles have been cited the most by colleagues in your research community? The number of citations is often an indicator of a paper's scientific impact, influence, or relevance. Papers may be cited to provide background reading in a specific area, to identify a methodology, to credit pioneers, to describe an original concept, or to give credit to related work. Log in at aocs.org and visit "My Account" to access the three most-cited articles from AOCS' three journals during first quarter 2021.

TAILORING CRYSTALLINE STRUCTURE USING HIGH-INTENSITY ULTRASOUND TO REDUCE OIL MIGRATION IN A LOW SATURATED FAT

Thais L. T. da Silva, Z. Cooper, J. Lee, F. Gibon, and S. Martini
http://dx.doi.org/10.1002/aocs.12321.

High-intensity ultrasound (HIU) was used to change the crystalline structure of an interesterified soybean oil (IESBO) with 33% of saturated fats, and the effects these changes had on oil migration were evaluated. The IESBO was crystallized at different temperatures (26, 28, 30, and 32°C) with and without HIU. Results showed that oil migration was significantly affected by HIU ($P < 0.05$). HIU promoted crystallization and induced the formation of harder crystalline networks that were more resistant to oil migration with lower melting peak temperatures and sharper melting profiles. Samples processed with HIU had fewer crystalline clusters as observed by microscopy. Changes observed on the physical properties of the IESBO due to sonication that consequently improved oil migration were attributed to the ability of HIU to induce secondary nucleation and crystallize low-melting point triacyclglycerols (SUU) that would not crystallize without the HIU and

to the stronger and stable crystalline network formed capable of entrapping liquid TAG (UUU).

THE EFFECT OF ANTIOXIDANTS ON CORN AND SUNFLOWER BIODIESEL PROPERTIES UNDER EXTREME OXIDATION CONDITIONS

Sergio Nogales-Delgado, J.M. Encinar, A. Guiberteau, and S. Márquez
http://dx.doi.org/10.1002/aocs.12288.

Two antioxidants, butylated hydroxyanisole (BHA) and tert-butylhydroquinone (TBHQ), were added to corn and sunflower biodiesel, and the effect of oxidation on the parameters of biodiesel was studied using extreme oxidation conditions to accelerate the oxidation process. Both antioxidants improved the oxidation stability of biodiesel, but some parameters were altered (viscosity and acid number) which could make this biofuel unsuitable for commercialization according to standards if high concentrations of antioxidants are used.

QUALITY AND COMPOSITION OF VIRGIN OLIVE OILS FROM INDIGENOUS AND EUROPEAN CULTIVARS GROWN IN CHINA

Le Yu, Y. Wang, G. Wu, J. Jin, Q. Jin, and X. Wang
http://dx.doi.org/10.1002/aocs.12315.

The characteristics of eight varieties of virgin olive oil obtained in two successive crops in the southwest of China, were investigated. Significant differences ($P < 0.05$) were observed in physicochemical properties, fatty acid profile, minor component contents, and oxidative stability between different varieties of olive oils. The physicochemical properties of all samples met IOC standards for extra virgin olive oil. The results of hierarchical cluster analysis and principal component analysis (PCA) showed a good classification between varieties based on their qualitative characteristics. Two variet-

ies were significantly different from other varieties mainly due to color, fatty acid profile, and minor components. PCA result also showed that harvest crop influences the characteristics of samples mainly due to the variance of temperature and rainfall.
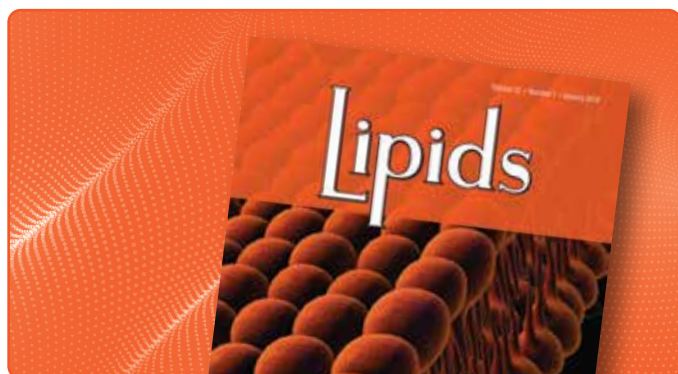
BIRTHWEIGHT AND LIPIDS IN ADULT LIFE: POPULATION-BASED CROSS-SECTIONAL STUDY

Issa Al Salmi and S. Hannawi
http://dx.doi.org/10.1002/lipd.12242.

The association of birthweight with lipid profile in a general adult population was examined. Participants in the second wave of a nationally representative cross-sectional study, Australian Diabetes, Obesity and Lifestyle *Study* (*AusDiab*), were asked to complete a birthweight questionnaire. Fasting total cholesterol (TC), LDL-C, HDL-C, and triacylglycerol levels were modeled against birthweight for the 4,502 people who reported their birthweights, mean (SD) of 3.4(0.7) kg. Females with low birthweight-LBW had higher levels of TC, LDL-C, and triacylglycerols, but no difference in HDL-C, than those with normal-birthweight (NBW;≥2.5 kg). People with LBW showed a trend toward increased risk for high TC (≥5.5 mmol/L) compared to NBW. Among females with LBW, the risk for high LDL-C (≥3.5 mmol/L) was increased compared to those of NBW. The risk for low HDL-C (<0.9 mmol/L) was increased among males with LBW compared to those with NBW. Females had higher risk for high LDL-C, whereas males had high risk for low HDL-C (<0.9 mmol/L). In addition, females with low birthweight had the highest triacylglycerol levels. High LDL-C, low HDL-C, and high triacylglycerols are well-recognized risk factors for cardiovascular disease.

THE POTENTIAL OF GENOME EDITING FOR IMPROVING SEED OIL CONTENT AND FATTY ACID COMPOSITION IN OILSEED CROPS

Udaya Subedi, K.N. Jayawardhane, X. Pan, J. Ozga, G. Chen, N.A. Foroud, and S.D. Singer
http://dx.doi.org/10.1002/lipd.12249.

This article reviews the genome-editing technology, CRISPR/Cas, and progress made thus far with respect to its use in oilseed crops to improve seed oil content and quality.

Several genes that may provide ideal targets for genome editing in this context are examined, as well as new CRISPR-related tools that have the potential to be applied to oilseed plants and may allow additional gains to be made in the future.

ACUTE HYPERCAPNIA/ISCHEMIA ALTERS THE ESTERIFICATION OF ARACHIDONIC ACID AND DOCOSAHEXAENOIC ACID EPOXIDE METABOLITES IN RAT BRAIN NEUTRAL LIPIDS

Yurika Otoki, A.H. Metherel, T. Pedersen, J. Yang, B.D. Hammock, R.P. Bazinet, J.W. Newman, A.Y. Taha
http://dx.doi.org/10.1002/lipd.12197

In the brain, approximately 90% of oxylipins are esterified to lipids. The significance of this esterification process is not known. This study validated an aminopropyl solid phase extraction (SPE) method for separating esterified lipids using 100 and 500 mg columns, and applied the method to quantify the distribution of esterified oxylipins within phospholipids (PL) and neutral lipids (NL) (i.e. triacylglycerol and cholesteryl ester) in rats subjected to head-focused microwave fixation (controls) or CO_2-induced hypercapnia/ischemia. We hypothesized that oxylipin esterification into these lipid pools will be altered following CO_2-induced hypercapnia/ischemia. Lipids

were extracted from control ($n = 8$) and CO_2-asphyxiated ($n = 8$) rat brains and separated on aminopropyl cartridges to yield PL and NL. The separated lipid fractions were hydrolyzed, purified with hydrophobic–lipophilic–balanced SPE columns, and analyzed with ultra-high-pressure liquid chromatography coupled to tandem mass spectrometry. Method validation showed that the 500 mg (vs 100 mg) aminopropyl columns yielded acceptable separation and recovery of esterified fatty acid epoxides but not other oxylipins. Two epoxides of arachidonic acid (ARA) were significantly increased, and three epoxides of docosahexaenoic acid (DHA) were significantly decreased in brain NL of CO_2-asphyxiated rats compared to controls subjected to head-focused microwave fixation. PL-bound fatty acid epoxides were highly variable and did not differ significantly between the groups. This study demonstrates that hypercapnia/ischemia alters the concentration of ARA and DHA epoxides within NL, reflecting an active turnover process regulating brain fatty acid epoxide concentrations.

BIOSURFACTANTS FROM WASTE: STRUCTURES AND INTERFACIAL PROPERTIES OF SOPHOROLIPIDS PRODUCED FROM A RESIDUAL OIL CAKE

Pedro Jiménez-Peñalver, A. Koh, R. Gross, T. Gea, and X. Font
http://dx.doi.org/10.1002/jsde.12366.

Current sophorolipids (SL) research is focused on the use of wastes to decrease the final production costs of SL and the environmental impacts. The structures and the interfacial properties of a crude SL natural mixture produced by solid-state fermentation from a residual sunflower oil cake from the oil-refining industry were studied. Liquid chromatography–mass spectrometry (LC–MS) demonstrated that the diacetylated lactonic 18:1 SL was the most abundant SL of the mixture, followed by the correspondent acidic form. The surface tension-lowering capacity was studied in a water–air interface at temperatures ranging from 15 to 50°C. The minimum surface tension and critical micelle concentration were determined. The emulsion properties were similar to those obtained for the commercial nonionic surfactant Triton X-100. The efficiency of the SL natural mixture was also proven successful for the displacement of a diesel slick. These results confirmed the effectiveness of SL produced from a real waste.

EFFECT OF NANOPARTICLES ON VISCOSITY AND INTERFACIAL TENSION OF AQUEOUS SURFACTANT SOLUTIONS AT HIGH SALINITY AND HIGH TEMPERATURE

Anastasia A. Ivanova, C. Phan, A. Barifcani, S. Iglauer, and A.N. Cheremisin
http://dx.doi.org/10.1002/jsde.12371.

Surfactant flooding has widely been used as one of the chemically enhanced oil recovery (EOR) techniques. Surfactants majorly influence the interfacial tension, γ, between oil and brine phase and control capillary number and relative permeability behavior and, thus, influence ultimate recovery. Additives, such as nanoparticles, are known to affect surfactant properties and are regarded as promising EOR agents. This study examined the influence of silica nanoparticles on the ability of surfactants to lower γ and to increase viscosity at various temperatures and salinities. Results show that the presence of nanoparticles decreased γ between n-decane and various surfactant formulations by up to 20%. It was found that γ of nanoparticles–surfactant solutions passed through a minimum at 35°C when salt was added. Furthermore, the viscosity of cationic surfactant solutions increased at specific salt (1.5 wt.%) and nanoparticle (0.05 wt.%) concentrations. Results illustrate that selected nanoparticles–surfactant formulations appear very promising for EOR as they can lower brine/n-decane interfacial tension and act as viscosity modifiers of the injected fluids.

LOW-TOXIC AND NONIRRITANT BIOSURFACTANT SURFACTIN AND ITS PERFORMANCES IN DETERGENT FORMULATIONS

Dan Fei, G.-W. Zhou, Z.-Q. Yu, H.-Z. Gang, J.-F. Liu, S.-Z. Yang, R.-Q. Y, and B.-Z. Mu
http://dx.doi.org/10.1002/jsde.12356.

Surfactin is a representative lipopeptide produced by microorganisms. In this study, surfactin was isolated from cell-free broth of *Bacillus subtilis* HSO121 and purified by reversed-phase high-performance liquid chromatography for detergent formulations. The biodegradability, acute dermal irritation, acute oral toxicity (LD_{50} and LC_{50}), surface activity, washing efficiency, and compatibility with hard water of the purified biosurfactant surfactin were studied to explore the feasibility for applications of the surfactin in detergents. Acute oral toxicity tests ($LD_{50} > 5000$ mg kg^{-1}, $LC_{50} > 1000$ mg kg^{-1}) and skin irritation tests (PII = 0) indicated that the surfactin is a low-toxic and nonirritant ingredient for detergent formulation. Moreover, it shows excellent surface and interfacial properties of emulsification and wettability, high compatibility, and stability in a wide range of temperatures, pH, and hard water and acceptable properties in biodegradability and foaming ability, which suggests that the biosurfactant surfactin is a promising ingredient for detergent formations in our daily life and for industrial applications.

May 3–14 ONLINE

2021 AOCS Annual Meeting & Expo

Advance science and technology together at the premier event in our industry.

Register now with member-only savings!

annualmeeting.aocs.org

US $149 FOR SINGLE TRACK ACCESS

US $225 FOR FULL MEETING ACCESS

Join your scientific community online at the 2021 AOCS Annual Meeting & Expo, the largest global forum on fats, oils, proteins, surfactants and related materials. The meeting, fully online in 2021, fosters collaboration across industry, academia and government.

Register now to secure your spot for popular events!

Networking activities, hosted by AOCS Divisions, Sections and Common Interest Groups, have limited space. Act quickly to secure your spot among your peers!

Learn, engage and advance with...

- 80 live sessions and interactive discussions showcasing the latest research
- Accessible online format allowing attendees to save time and travel expense
- Special events including Midweek Mixers and the AOCS Award Presentation Series

Visit **annualmeeting.aocs.org** for more information and to register.

EXTRACTS & DISTILLATES

The full version of all AOCS journal articles are available online to members at www.aocs.org/journal. This column builds on that member benefit by primarily highlighting articles from other journals.

ANA Analytical

EAT Edible Applications

H&N Health and Nutrition

PRO Processing

S&D Surfactants and Detergents

BIO Biotechnology

LOQ Lipid Oxidation and Quality

IOP Industrial Oil Products

PCP Protein and Co-Products

Original Articles

BIO **IOP** Microbial lipid and biodiesel production from municipal sludge fortified with crude glycerol medium using pH-based fed-batch strategy

Kumar, L.K., *et al.*, *J. Environ. Chem Eng. 9*: 105032, February 2021, https://doi.org/10.1016/j.jece.2021.105032.

To achieve a good performance in fed-batch fermentation, the feeding strategy is essential and important. In this study, pH-based (pH 6.0) fed-batch fermentation has been conducted on municipal sludge fortified with crude glycerol medium using *Y. lipolytica* SKY7 (YL). No additional nitrogen source and trace elements had to be added as secondary municipal sludge had sufficient nitrogen and trace elements to support growth of YL. At 72 h of fed-batch fermentation, high biomass (59.67 g/L) and lipid concentration (31.44 g/L) were obtained. The biodiesel produced using sludge cultivated YL had similar fatty acid ester profile as that of vegetable oil. It was revealed from energy balance that biodiesel production using pH-based fed-batch strategy was energetically favorable and has unit production cost of 0.67 $/L B10.

EAT Subcritical water hydrolysis of soybean residues for obtaining fermentable sugars

Vedovatto, F., *et al.*, *J. Supercrit. Fluid. 167*: 105043, 2021, https://doi.org/10.1016/j.supflu.2020.105043.

The objective of this study was to evaluate subcritical water hydrolysis (SWH) of soybean straw and hull for obtaining fermentable sugars in a semi-continuous mode. The experimental conditions investigated were temperature (180, 220, and 260 °C) and liquid/solid mass (R) ratio (9 g and 18 g water/g straw, and 7.5 g and 15 g water/g hull). The hydrolysis was performed for 15 min at 25 MPa. The characterization of residues after hydrolysis and reducing sugars yield (Y_{RS}), efficiency (E) and composition of hydrolysates were evaluated. The condition of 220 °C/R-18 and R-15 provided the highest Y_{RS} of 9.56 ± 0.53 g/100 g straw at 4 min and 10.15 ± 0.50 g/100 g hull at 3 min of hydrolysis. The efficiencies were 23.65 ± 1.32 g/100 g carbohydrates and 23.04 ± 1.14 g/100 g carbohydrates for soybean straw and hull, respectively. SWH modified the structure of the residues and allowed the production of fermentable sugars.

EAT **H&N** Enhancement of isoflavone aglycone, amino acid, and CLA contents in fermented soybean yogurts using different strains: screening of antioxidant and digestive enzyme inhibition properties

Hwang, C.E., *et al.*, *Food Chem. 340*: 128199, 2021, https://doi.org/10.1016/j.foodchem.2020.128199.

This study was the first to evaluate changes in isoflavone, amino acid, conjugated linoleic acid (CLA), antioxidant effect, and digestive enzyme inhibition during fermentation of soy milk to soy yogurt with *L. brevis* and *L. plantarum*. Total average isoflavones were reduced (1318.2 → 971.1 microgram/g) with an increase of aglycones (60.2 → 804.9 microgram/g, genistein > daidzein > glycitein) in soy powder yogurts (SPYs). Amino acids increased considerably, as did ornithine (average 4.1 → 551.0 mg/g), and CLA showed high variations from not-detected (ND) to 0.5, 0.9 mg/g (*cis*-9, *trans*-11) and ND to 0.3, 0.2 mg/g (*trans*-10, *cis*-12). Digestive enzyme inhibitions (alpha-glucosidase, alpha-amylase, and pancreatic lipase) displayed high activities (average 50.6 → 67.2, 5.2 → 46.4, 10.6 → 51.4%). Moreover, the antioxidant abilities against radicals were elevated as follows: ABTS > DPPH > hydroxyl (average 63.5 → 86.5, 50.2 → 70.3, 39.3 → 55.2%). Specifically, SPY using mixed strains exhibited the greatest enzymatic inhibition and antioxidant capacities.

IOP Bio-based polyricinoleate and polyhydroxystearate: properties and evaluation as viscosity modifiers for lubricants

Méheust, H., *et al.*, *ACS Appl. Polym. Mater.*, January 4, 2021, https://doi.org/10.1021/acsapm.0c01153.

This paper aims to design bio-based polyester as a viscosity modifier for lubricant properties. Bio-based polyricinoleate (PRic) and its saturated homologous polyhydroxystearate (PHS) have been synthesized from fatty acid methyl esters. The polycondensation performed in bulk in a one-step reaction without any purification leads to two series of polyesters within a large

range of molecular weights, with M_w between 3 and 130 kg·mol⁻¹. Their thermal properties were investigated. Good thermal stability was observed with degradation temperatures above 300 °C. As expected, PRic appeared to be amorphous with a particularly low glass-transition temperature, while PHS is semicrystalline. A rheological study determined that polyricinoleate entangled when its molecular weight was above 25 kg·mol⁻¹. These two bio-based and biodegradable polymers were then evaluated as viscosity modifiers in both organic and mineral oils. PHS with high molecular weights appeared to be an excellent thickener as well as a good viscosity index improver with a viscosity index (VI) increase above +50 in organic lubricant oil and +64 in mineral oil.

LOQ Soybean (glycine max) hull valorization through the extraction of polyphenols by green alternative methods

Cabezudo, I., *et al.*, *Food Chem.* 338: 128131, 2021, https://doi.org/10.1016/j.foodchem.2020.128131.

Soybean is one of the largest crops in the world, with about 348.7 million tons being produced in 2018. Soybean hull is a by-product produced during the processing of soybean to obtain flour and oil. Though not being actually exploited, it is a source of polyphenols with antioxidant activity. Here, the extraction of polyphenols from soybean hull was performed by means of an alkaline hydrolysis treatment, which was optimized by the response surface

methodology. At the optimal region, a total phenolic content of 0.72 g gallic acid equivalents per 100 g of soybean hull was obtained with an antioxidant activity of 2.17 mmoles of Trolox equivalents. Polyphenols responsible for the antioxidant activities were identified by LC–MS, including phenolic acids, anthocyanins, stilbenes, and the two main isoflavones of soybean, daidzein and genistein, in their non-glycosylated form. Other alternative extraction methods based on *Aspergillus oryzae* fermentation and alpha-amylase hydrolysis are also proposed.

H&N LOQ A chemical valorization of melon peels toward functional food ingredients: bioactives profile and antioxidant properties

Gómez-García, R., *et al.*, *Food Chem.* 335: 127579, 2021, https://doi.org/10.1016/j.foodchem.2020.127579.

The goal of this work was to characterize the profile of bioactive compounds and the antioxidant activity of inodorus melon peels. Melon peels were divided into three fractions: a solid fraction with a higher content of carbohydrates (84.81%); a liquid fraction with a higher ash content (11.5%); and a pellet fraction with a higher protein content (34.90%). The structural carbohydrates study revealed a composition of cellulose (27.68%), hemicellulose (8.2%), and lignin (26.46%) in the solid fraction. The liquid fraction had the highest antioxidant activity based on results from DPPH, ABTS, and ORAC assays. Flavones and hydroxybenzoic and hydroxycinnamic acids were the main phenolic classes found in all fractions. In addition, beta-carotene, lutein, beta-cryptoxanthin, and violaxanthin had also been quantified. Melon fractions were rich in nutrients and bioactive substances and could be useful in the development of novel functional products, considering the growing market demand for safe and healthy food products.

EAT LOQ Lycopene, polyphenols, and antioxidant activities of three characteristic tomato cultivars subjected to two drying methods

Tan, S., *et al.*, *Food Chem.* 338: 128062, 2021, https://doi.org/10.1016/j.foodchem.2020.128062.

The aim of this study was to evaluate the effects of freeze-drying and oven-drying on appearance, chemical components, and antioxidant activities of three cultivars of tomatoes. On the basis of appearance and polyphenol contents, freeze-drying showed better results. However, oven drying was found superior in decreasing degradation of lycopene. The effects of drying on the polyphenol contents varied depending on the cultivars. In addition, there were no significant differences in antioxidant activities between freeze-dried and oven dried tomatoes. These results also demonstrated that freeze-drying is superior in maintaining physical structure and phenolic contents of tomato slices. However, oven-drying is a viable option for drying tomatoes considering both the cost and contents of lycopene.

INFORM

International News on Fats, Oils, and Related Materials

More than a year with
COVID-19

Where do we go from here?

ALSO INSIDE:

Oleofoams

Alternative base oils

Goodbye to caustic washing

AOCS