# INFORM

International News on Fats, Oils, and Related Materials

# LIPOSOME Moving beyond drug delivery

# **ALSO INSIDE:**

Enzyme-assisted alkaline refining Plant and protein contents in food Phospholipids from dairy waste



# **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

# Best In EDIBLE OIL FILTRATION





# SHARPLEX FILTERS (INDIA) PVT. LTD. AN ISO 9001:2008, 14001:18001 COMPANY

R-664, T.T.C. Industrial Area, Thane Belapur Road, Rabale, MIDC, Navi Mumbai - 400 701, India. Tel: +91 9136921232 till 9136921239 / 022-27696322/31/39 Fax: 022-27696325 Toll Free No. – Spares Dept.- 1800226641 E-mail : sales@sharplexfilters.com www.sharplex.com

remitrishad/219/a0cs

CE 🙉 💮 🔛 💬

# INFORM January 2021 CONT FN

# 6 The latest on liposomes

These lipid-based compounds not only deliver drugs to their intended targets; they also increase the effectiveness of beauty products and supplements, such as vitamins.







# **12** Extraction of phospholipids from dairy processing by-products

The filtration methods used to make commercial dairy phospholipids (PLs) concentrates from dairy waste do not separate polar and neutral lipids. Recent evaluations of a scalable method demonstrated that total lipids can be extracted and fractionated into neutral and polar lipid fractions using green solvents.

# **16** Determining protein and fat in plant-based foods

Learn how the fat and protein content of a wide variety of plant-based food samples can be determined with standard automated analytical methods.

**22 Phospholipase for enzyme-assisted alkaline refining** New evidence shows that incorporating this enzyme directly into the alkaline refining process results in significantly higher yields and preserves the final oil quality from caustic refining.

26 Bio-based (edible) oils: feedstock for lubricants of the future Enhanced environmental guidelines and regulations in the food-processing industry have created a need for bio-lubricants that are less toxic and more biodegradable.

# DEPARTMENTS

5 Index to Advertisers

46 Classified Advertising

- 40 AOCS Meeting Watch
- Analysis/commentary
- 32 Olio
- 35 Regulatory Review
- **37** Member Spotlight

# **Publications and more**

- 38 Patents
- 40 AOCS Journals
- 42 Extracts & Distillates



# We are proud to be a partner in success stories of our clients

One Point Solution Provider Design, Manufacture, Supply, Install, Commission & After Sales

# 165 Global References 45+ Lecithin Plants 50+ Green Field Projects









# **Edible Oil Refining**

- Degumming & Neutralization
- Bleaching
- Dewaxing / Winterization
- Deodorization

# **By-Product Processing**

- Gums Drying (Lecithin)
- Soapstock Splitting (Acid Oil)

# **Seed Crushing**

- Flash Desolventization (Soya White Flakes)
- Soapstock Desolventization

## **Fat Modification**

- Fractionation
- Interesterification
- Hydrogenation

# **Biodiesel Production**

# Pilot Plant Skid Mounted Plants

# **Customer Services**

- Technical Audits
- Existing Plant Upgrade to improve
  - Efficiencies
  - Quality
  - Yield
  - Capacity
- Automation
- Products & Spares

# **DVC Process Technologists**

Phone : +91 86699 56061 / 62 / 63 / 64 Fax : +91 21152 53970 E-mail : sales@dvcprocesstech.com, info@dvcprocesstech.com

# www.dvcprocesstech.com

# INFORM

### AOCS MISSION STATEMENT

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

### INFORM

International News on Fats, Oils, and Related Materials ISSN: 1528-9303 IFRMEC 32 (1) Copyright © 2013 AOCS Press

### **EDITOR-IN-CHIEF EMERITUS**

James B.M. Rattray

### CONTRIBUTING EDITORS

Scott Bloomer

Leslie Kleiner

### EDITORIAL ADVISORY COMMITTEE

Julian Barnes Gijs Calliauw Etienne Guillocheau Jerry King Thu (Nguyen) Landry Gary List L Jill Moser Ig Warren Schmidt

Raj Shah Utkarsh Shah Ignacio Vieitez 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 Baillely, 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.

# **INDEX TO ADVERTISERS**

Buchi	21
*Crown Iron Works Company	C3
*Desmet Ballestra Engineering NA	C2
DVC Process Technologists	4
*French Oil Mill Machinery Co	21
*Oil-Dri Corporation of America	C4
Pope Scientific, Inc	11
Sharplex Filters (India) Pvt. Ltd.	1

\*Corporate member of AOCS who supports the Society through corporate membership dues.

2710 South Boulder Drive P.O. Box 17190 Urbana, IL 61803-7190 USA Phone: +1 217-359-2344 Fax: +1 217-351-8091 Email: publications@aocs.org

### ADVERTISING INSTRUCTIONS AND DEADLINES

Closing dates are published on the AOCS website (www.aocs.org). Insertion orders received after closing will be subject to acceptance at advertisers' risk. No cancellations accepted after closing date. Ad materials must be prepared per published print ad specifications (posted on www.aocs.org) and received by the published material closing dates. Materials received after deadline or materials requiring changes will be published at advertisers' risk. Send insertion orders and materials to the email address below.

**NOTE:** AOCS reserves the right to reject advertising copy which in its opinion is unethical, misleading, unfair, or otherwise inappropriate or incompatible with the character of *Inform*. Advertisers and advertising agencies assume liability for all content (including text, representation, and illustrations) of advertisements printed and also assume responsibility for any claims arising therefrom made against the publisher.

AOCS Advertising: Sterling Bollman Phone: +1 217-693-4901 Fax: +1 217-693-4864 sterling.bollman@aocs.org

Formerly published as *Chemists' Section, Cotton Oil Press,* 1917–1924; *Journal of the Oil and Fat Industries,* 1924–1931; *Oil & Soap,* 1932–1947; news portion of *JAOCS,* 1948–1989. The American Oil Chemists' Society assumes no responsibility for statements or opinions of contributors to its columns.

Inform (ISSN: 1528-9303) is published 10 times per year in January, February, March, April, May, June, July/August, September, October, November/ December by AOCS Press, 2710 South Boulder Drive, Urbana, IL 61802-6996 USA. Phone: +1 217-359-2344. Periodicals Postage paid at Urbana, IL, and additional mailing offices. **POSTMASTER:** Send address changes to *Inform*, P.O. Box 17190, Urbana, IL 61803-7190 USA.

Subscriptions to Inform for members of the American Oil Chemists' Society are included in the annual dues. An individual subscription to Inform is \$195. Outside the U.S., add \$35 for surface mail, or add \$125 for air mail. Institutional subscriptions to the Journal of the American Oil Chemists' Society and Inform combined are now being handled by Wiley. Price list information is available at http://olabout. wiley.com/WileyCDA/Section/id-406108.html. Claims for copies lost in the mail must be received within 30 days (90 days outside the U.S.) of the date of issue. Notice of change of address must be received two weeks before the date of issue. For subscription inquiries, please contact Julie May at AOCS, julie.may@aocs.org. AOCS membership information and applications can be obtained from: AOCS, P.O. Box 17190, Urbana, IL 61803-7190 USA or membership@ aocs.org.

**NOTICE TO COPIERS:** Authorization to photocopy items for internal or personal use, or the internal or personal use of specific clients, is granted by the American Oil Chemists' Society for libraries and other users registered with the Copyright Clearance Center (www.copyright.com) Transactional Reporting Service, provided that the base fee of \$15.00 and a page charge of \$0.50 per copy are paid directly to CCC, 21 Congress St., Salem, MA 01970 USA.

# The latest on Rebecca Guenard

Since 1961, when images from an electron microscope first revealed the bilayer lipid structure of cell membranes, researchers have been intrigued by their potential. The scientist who made the discovery, Alec Douglas Bangham, at Babraham Institute in Cambridge, Massachusetts, USA, observed that, like a microscopic parcel, the closed structure released its contents when exposed to a detergent. He called the structures "liposomes."

- The courier capability of liposomes is being fine-tuned for drug delivery, while researchers continue to imagine new applications.
- Supplement makers are touting the potential benefit of liposomes for increasing the circulation time of vitamins in the blood.
- Researchers have developed a liposome that is capable of promoting cell attachment and growth by delivering an extracellular matrix to cells.
- Liposomes could eventually lead to artificial cells that reveal the origin of life.

The lipid bilayer of liposomes creates a unique vessel that's utility was immediately recognized as a means for stealthily delivering drugs to sites of disease in the human body. The hydrophobic membrane encapsulates an aqueous core where hydrophilic molecules can be contained. Liposomes can also encapsulate molecules that do not dissolve in water. Hydrophobic molecules trapped inside them will cling to the interior bilayer walls. In fact, the physical properties of liposomes allow for a diversity of compounds and pH ranges to reside within the structure without detection by the body's immune system.

"Lipids are biologically friendly molecules," says Kwanwoo Shin, professor at Sogang University in Soul, Korea. "They are very stable in our biological systems."

In the decades since their discovery, pharmaceutical companies have developed a range

# **CELLULAR LIPIDS**



of clinically approved liposome-based medicines. They are ideal for drugs with poor pharmacokinetics, limited bioavailability, or a high degree of toxicity. As the field of nano-medicine grows, the biocompatibility of liposomes and lipid nanoparticles will continue to make them a valuable asset for drug delivery.

Cellular lipid

In fact, lipid nanoparticles are a critical part of the COVID-19 vaccines being produced by Pfizer and Moderna. Both manufacturers use lipids to shield delicate mRNA long enough for it to be delivered to cells. There, the mRNA signals the cell to produce a coronavirus protein that induces a mild immune response, prepping the body for future COVID-19 exposure. A Moderna representative has stated that its unique combination of four different lipids sufficiently protect mRNA from degrading, thus requiring a lower storage temperature than the Pfizer vaccine (https://tinyurl.com/covidmRNAvaccines).

Along with their established role in pharmaceuticals, such lipid-based compounds have enhanced the effectiveness of products for the beauty and supplement industries. And researchers are getting more innovative about liposome applications, even using them to make artificial cells. Here are some of the latest commercial and research applications.

# **A NANOMEDICINE**

"Liposomes prolong circulation of a drug in the body, breaking down without being expelled through the kidneys, for a slow release of the active ingredient," says Claudia Zylberberg, CEO at Akron Biotech, in Boca Raton, Florida, USA. The company provides products and services to biopharmaceutical companies (https://www.akronbiotech.com/). The US Food and Drug Administration (FDA) approved liposomes as the first nanoscale drug in 1995. Since then, development has centered on delivering a target compound to a specific location, within a specific time, and at a desired permeation rate. Researchers alter the composition of phospholipids in the bilayer to tailor the drug-release profile for specific applications.

Early on, interactions with circulating blood proteins destabilized liposomes, and they were cleared from the body shortly after injection. The drug carriers became more effective after they were coated with inert hydrophilic polymers, such as polyethylene glycol (PEG). The improvement increased circulation time and, thus, bioavailability, earning these modified versions the name, "stealth liposomes."

Coating liposomes has led to their improved effectiveness in targeting specific treatment areas. Ligands attached to the stealth liposome shell home in on unique disease environments and selectively deliver a drug. Cancer therapies center on overexpressed cell receptors that occur with malignancy. Connecting ligands of certain enzymes, folic acid, or monoclonal antibodies to the exterior of the liposomes is like providing a key that can access cancer cells. Once the liposomes have gained access, they unleash their cancer-killing cargo.

"It is also possible to have them break down in a certain environment," says Zylberberg. "Like in a capillary where there is a lot of oxygen and CO<sub>2</sub> exchange actively changing the local environment to have a lower pH."

Customized liposome coatings have resulted in successful clinical trials for liposome-based drugs to treat fungal infections, meningitis, hepatitis, as well as a variety of cancers. In clinical studies, the pharmacokinetics and biodistribution surpass traditional treatments while minimizing toxicity.

Zylberberg says she will not be surprised if the success of liposomes as delivery vehicles that can be modified for cell penetration will lead to their application in more and more commercialized products.

# **ENCAPSULATING SUPPLEMENTS**

Many of the vitamins sold as supplements do not produce as much benefit as they could because of a lack of bioavailability. The majority are not absorbed by the body. They are broken down during digestion and expelled.

Some supplement makers have caught on to the potential benefit of liposomes for increasing the circulation time of vitamins in the blood. A company called SOMEGA, in Cork, Ireland, has just launched a liposome-coated vitamin C supplement. One of the company's co-founders claims the new technology



FIG. 1. (A) Comparison of blood plasma concentrations of vitamin C after intravenous administration versus oral and liposomal oral doses (B) Comparison of oral doses only showing an increase in blood plasma concentration with liposomes. Source: Davis, J.L., Nutrition and Metabolic Insights, January 2016

improves absorption by enhancing delivery of vitamin C to the cells (https://tinyurl.com/supplementswithliposomes).

A company in Rehoboth Beach, Delaware, USA, is making a similar claim. Manna is promoting a liposomal curcumin that they say enhances powdered turmeric's pain-reducing and anti-inflammation capabilities (https://curcumin.mannaboost. com). The company cites scientific studies that support their claims. In fact, recently published research on small groups of participants show an increase in bioavailability with liposomeencapsulated supplements.

Researchers compared intravenous, oral, and liposomal vitamin C supplements (Fig. 1) and found that encapsulating the vitamin resulted in a higher concentration in the blood when taken orally than occurred with non-encapsulated supplements (https://doi.org/10.4137/NMI.S39764). Typically, vitamin C must be administered intravenously to achieve a significant amount in blood plasma. Oral doses of the supplement do not make it through the renal system without decomposing. However, the longer vitamins are in the blood stream, the greater the chance they will be absorbed into cells and counter the effects of oxidative stress. The conclusion of liposomal vitamin studies is that these nanoparticles provide users with the critical benefit of a longer duration in the blood stream (https://doi.org/10.1080/08982104.2019.1630642).



### **BEAUTY AND HEALING**

The beauty industry has also taken advantage of liposomes as a means for delivering molecules to cells. The market has myriad anti-aging products encapsulated in liposomes to improve effectiveness.

Shin says it is not enough to coat an anti-aging ingredient. The synthesis should be strategic. The properties of the liposome and its contents must be planned properly to achieve the desired outcomes for a specific application, he adds. For precisely controlled delivery to a target, you need to make the exact sized nanoparticle containing the exact compound with the exact molecular structure.

"If the particle size is much smaller than the cell size of human skin, then the particles start to penetrate the cell directly," Shin explains. "If the size is bigger, then the cell naturally repels it." He is currently collaborating with Harvard researchers on a new liposome delivery system for collagen.

The problem is collagen is a huge molecule, according to Shin. "Sometimes people drink it or apply it directly, but it is too big to penetrate inside a cell." Shin and his collaborators have developed liposomes containing tropocollagen molecules, smaller collagen subunits that link together to make up a full collagen molecule. They are small enough to penetrate deep into the skin and enter cells. An external stimulation source, like the light from an LED mask, dismantles the liposomes, spilling their contents, which cells then use to build collagen fiber *in situ*. Skin cells excrete the collagen into intercellular space, replacing what is lost naturally with age.

Shin says this liposomal collagen product has been successfully tested on animals. The group hopes to commercialize it next year. Concurrently, they are applying their process for developing this product to create a treatment for healing wounds.

"Healing is a natural process, but to facilitate it we are adding liposomes with extra cellular matrix proteins," Shin says.

Fibronectin, laminin, and elastin are some of the proteins that compose the body's connective tissue. When an injury occurs, they remodel the skin and formulate scar tissue.

Shin and his team have combined these proteins with liposomes and found that the additional cellular matrix proteins alone accelerate healing. "Even without drugs we can make the cells more active, more fast moving so they can heal much faster," he says.

This is particularly important for burn victims. After a burn, skin scars more quickly than cells can rebuild the skin's original shape, permanently changing its structure. But if you activate the cells with liposomes containing the necessary proteins, then you can entice it to rebuild the skin before scarring occurs, Shin says. Perhaps all bandages could be infused with this treatment to improve wound healing.

# **ARTIFICIAL CELLS**

Shin views the applications he described as a means to an end. What he is most interested in studying is the fundamental mechanism for cellular function. To do that, he uses liposomes to recreate the organelles and membranes that comprise a living cell.

Shin explains that a classic liposome study focuses on optimizing the shape of the lipid bilayer for a specific target. His research group is working with shape as well as function, so the liposome behaves like an actual cell.

"This is really complex, but we went through one function at a time. Then we combined them incrementally," he says. "The whole architecture is just made of lipids and proteins."

Building a cell from the ground up inevitably involves the challenge of having to duplicate the complex metabolic reactions that cells need to operate. Shin's team addressed the challenge by using light, in a type of photosynthesis, to initiate cell processing. They embedded proteins and enzymes in a lipid membrane which produced adenosine triphosphate (ATP) when activated. Switching the light to a different wavelength stops the reaction. ATP provides the energy necessary for the cells to generate actin, a protein that forms filaments of muscle cells. The experiment, published in *Nature Biotechnology* (https://doi.org/10.1038/nbt.4140), proved that liposomes could be used to make a functioning artificial cell (Fig. 2).

Shin plans to continue his international collaboration with the Harvard researchers in pursuit of creating fully operational artificial cells. He says that these types of cells could be used to identify how a disease manifests and, ultimately, how to cure or eliminate that disease. Primarily, he is curious about life.

"There was a protein; there was a lipid; there were many other things," says Shin. "Somehow they assembled to become bacteria, to become viruses, to become mammalian cells." In the future, he wants to perform fundamental studies to understand how multi-celled organisms developed. "We could use artificial cells to learn how human life originated," says Shin.

The human body is teeming with lipids. Liposomal cloaks take advantage of that fact and allow researchers to sneak crucial drug treatments past the body's immune system. Over the decades, they have gotten better at disguising drugs so that nefarious cells do not suspect the threat that lies inside. It is remarkable what scientists can do with a group of amphiphilic molecules. They may even someday provide the answer for the origins of life on Earth.

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



FIG. 2. Image of an artificial cell with a liposomal membrane (red) embedded with artificial organelles (green) that produce the protein actin (white) using ATP that forms when the cell is activated by light. Source: Kwanwoo Shin, Sogang University, Soul, Korea

# **Information**

Tumor repolarization by an advanced liposomal drug delivery system provides a potent new approach for chemo-immunotherapy, Ringgaard, L., *et al., Sci. Adv. 6*: 36, 2020.

Photosynthetic artificial organelles sustain and control ATP-dependent reactions in a protocellular system, Lee, K.Y., *et al., Nat. Biotechnol. 36*: 530–535, 2018.

Pharmaceutical liposomal drug delivery: a review of new delivery systems and a look at the regulatory landscape, Zylberberg, C. and S. Matosevic, *Drug Delivery* 23: 9, 2016.

Liposomal-encapsulated ascorbic acid: influence on vitamin C bioavailability and capacity to protect against ischemia–reperfusion injury, Davis, J.L., *Nutrition and Metabolic Insights*, January 2016.

# Hybrid

Wiped Film/Fractional Still Systems Combine Technologies To Provide Highest Purity, Yield & Value

Pope Scientific's world leadership in hybrid technology evolved from decades of experience in toll distillation, pilot process development and lab studies along with continuous innovation of equipment including wiped film and fractional stills.

Our breakthrough systems incorporate short duration, high vacuum wiped-film evaporation with efficient multiple plate column fractionation to:

- Allow the purification of heat-sensitive materials similar in volatility, which could not otherwise be separated; and
- Advance the quality of your product to levels not previously possible.

To your advantage, we're not just providing equipment; we're processing in-house as well. It's the synthesis of theoretical knowledge and handson expertise that truly separates us from the competition.



Tri-functional hybrid pilot plant is configurable for molecular distillation, evaporation and hybrid separation.

# Successfully developed separation & purification applications include:

- Edible and Essential Oils
- Foods, Flavors & Fragrances
- Vitamins & Nutraceuticals
- Pharmaceutical Intermediates & Cosmetics
- Polymers, Waxes, Lubricants & Bio-based Materials
- Many other temperature sensitive separations [Fish, Citrus, Mint, Wood, Other Botanical Oils, Omega-3, FAME]

# Concept > Lab > Pilot Plant > Commercialization

For 50+ years Pope Scientific has provided a full range of process solutions.

### Toll Processing Services:

- Laboratory Feasibility Testing Process Development/Pilot Trials
- Applications Assistance Any Time/Any Stage
- Contract Processing 
   CGMP & Kosher Certified

-----

### Chemical Processing Equipment (for Laboratory, Pilot & Large Scale Production):

- Wiped-Film Molecular (Short Path) Stills & Evaporators
- Batch and Continuous Fractional Distillation Systems
- Hybrid Wiped-Film/Fractional Distillation Systems
- Pressure Vessels, Reactors, and Process Vessel Systems





3-stage stainless steel production scale hybrid distillation plant. Built with PLC system and used for the purification of nutritional supplements and intermediates.



www.popeinc.com 1-262-268-9300

# Extraction of Tong Wang phospholipids from dairy processing by-products

- PLs from dairy processing by-products can be extracted using safe solvents.
- Fractionation of the total lipids into neutral and polar lipid fractions using green solvents is possible and feasible.
- Salt precipitation of the milk fat globule membrane fragments from low-solid and low-fat content dairy processing by-products is effective; PLs can then be feasibly separated from the precipitate.

Dairy phospholipids (PLs) are milk fat globule membrane (MFGM) lipids. Milk fat globule (MFG) lipids are a complex mixture containing about 70% neutral lipids and 26 to 30% polar lipids. MFGM PLs are functional components in infant formulas and health-promoting foods. They are unique compared to other sources of PLs, because of the presence of sphingomyelin (SM, 4.1–29.2% of total PLs) and phosphatidylserine (PS, 2.0–16.1% of total PLs). These polar lipids protect against infection, contribute to the healthy development and maintenance of the brain and gastrointestinal immune systems in early and late life, and improve memory (Conway, et al., 2014). SM is reported to play important roles in cell regulation and tumor suppression. PS has been attributed to cognitive performance improvement.

The low content of PLs in dairy products makes it difficult to extract and concentrate PLs on an industrial scale. Milk contains only 0.01–0.04% PLs, but dairy by-products contain much higher concentrations of PLs, as shown in Table 1. These data indicate that PLs associated with the MFGM fragments tend to move toward the aqueous portion during processing. Therefore, these processing by-products are ideal feedstocks for PLs extraction. Commercial dairy PLs concentrates are mostly produced using microfiltration, ultrafiltration, supercritical fluid extraction, or a combination of these technologies. However, the filtration methods do not specifically separate the lipid classes (i.e., polar and neutral lipids), and the concentration of PLs in the product is relatively low. The proteins in the aqueous system tend to interfere with membrane separation, decreasing operation efficiency. Because of this challenge, we have evaluated scalable methods of extraction and fractionation that are summarized in this article.

# **ETHANOL EXTRACTION OF DAIRY PLs**

Previously, we successfully used hot ethanol to extract PLs from egg yolk by injecting a stream of yolk liquid in a solvent vessel. The protein instantaneously coagulates and forms a network, and the lipids are leached out. Multiple stages of extraction can be used, and various extraction parameters can be tested. Figure 1 shows how the procream, a lipid-rich by-product removed from whey to make whey protein isolate (also referred to as whey protein phospholipid concentrate), behaved in this extraction system. We found that aqueous ethanol in the concentration of 70–90% and at temperature 60-80°C can extract both neutral and polar lipids, but with different preferences depending on the extraction conditions. Overall, if a high ethanol concentration and temperature are used (90% at 70°C), 94 and 100% of the total and PLs can be recovered through 5-stage extraction (most are extracted with two sequential extractions).

However, the PLs concentration in the product is low (32%) as it is in the original material (~29% relative to total lipids). If lower ethanol concentration and temperature are used (70% at 70°C), the solvent will have a preference for PLs, leading to a product with 46% PLs, but with much lower total and PLs recovery of 41 and 58% (Price, *et al.*, 2018). Therefore, this method is modestly successful. To further concentrate the PLs, an additional fractionation method is needed.

# SOLVENT FRACTIONATION OF THE TOTAL DAIRY LIPIDS

To investigate suitable solvent and conditions to separate the neutral lipid from PLs, a total dairy lipid extracted from beta stream—a by-product of making anhydrous milk fat from butter—was used in a study (Price, *et al.*, 2020a). A fixed lipid:solvent ratio of 1:10 (g/mL) was used at various fractionation temperatures (-20, 2, 15, 23, 40, and 60°C) depending on the solvents. The use of acetone at 23°C, not at any lower temperatures, led to a dairy PLs product with high PLs content of 71.5% (Fig 2, page 14). The more aqueous ethanol, i.e., at 70% concentration at 60°C compared to 95%, was able to preferentially extract PLs to form products with up to 74.7% PLs, but the PLs yield was much lower (26.3%) compared to acetone precipitation (97.9%). The 95% ethanol at 23°C led to a PLs product with 59.7% yield and 60.7% PLs content, compared to

### TABLE 1. Dairy phospholipids (PLs) content in various products

	PLs % in product	PLs % in dry matter	PLs % in total lipids
Whole milk	0.0	0.2	0.8
Skim milk	0.0	0.3	19.1
Cream	0.2	0.4	0.5
Butter	0.2	0.3	0.3
Buttermilk	0.1	1.5	25.6
Buttermilk whey	0.1	1.8	23.7
Butter serum	1.3	11.5	48.4
Cheddar	0.2	0.3	0.5
Cottage cheese	0.4	1.6	5.3
Whey (cheddar)	0.0	0.3	5.3
Whey (Swiss)	0.0	0.3	33.6

Data obtained from Rombaut and Dewettinck (2006).



FIG 1. Procream liquid is injected in the heated ethanol vessel, at the equilibrium state for stage-1 extraction (left) and the five sequentially extracted dairy lipids (right).



# FIG. 2. Fractionation of neutral and polar lipids of total dairy fat at various temperatures using 70% ethanol (top), 95% ethanol (middle), and acetone (bottom).

the initial beta stream lipid fraction that contained 35.2% PLs. Therefore, acetone precipitation at ambient temperature is an effective way to separate neutral and polar lipids.

Fatty acid (FA) composition of the separated polar and neutral lipid also yielded interesting observations. As expected, PLs extract contained more unsaturated FA (mainly C18:1) compared to the neutral lipid in the respective product. Phosphatidylethanolamine (PE) is the most saturated PL class, followed by SM. It is very interesting to observe that



FIG 3. Dairy PLs precipitation with Ca acetate and Zn acetate at 30 and 60°C at different salt concentrations (left); and protein precipitation along with PLs (right, insert shows clarification of beta stream dispersion with increasing salt concentration).

C23:0 is found primarily in the PLs, concentrated in SM (up to 18–22% of the total FAs) and PE (up to 12.6%). SM contained the highest amount of long-chain fatty acids, such as C20:0, 22:0, 22:1, 23:0, and 24:0. In addition, the branched-chain fatty acids (BCFAs) were primarily found in the NL fraction. We also aimed to enrich BCFAs during the process, but it proved to be very challenging due to their overlapping melting points with other fatty acids. The BCFAs concentration in the initial beta stream was about 1.0% of the total lipid fraction, which was composed of 13.1% iso C15:0, 28.9% anteiso C15:0, 24.0% iso C17:0, and 34.0% anteiso C17:0 fatty acids.

Overall, the use of solvent fractionation was shown to be an effective way to concentrate dairy PLs from a high-PLs content dairy fat. The solvent type and fractionation temperature are important factors for PLs concentration. Future work can be done to fractionate directly from the solid and high-solid content dairy by-products using modified conditions identified in this study.

# SALT PRECIPITATION OF THE DAIRY PLS FROM DILUTE OR LOW-SOLID LOW-FAT CONTENT BY-PRODUCTS

To obtain a PLs-rich fraction from a low-fat or low-solid content aqueous system, a salt precipitation method was explored. The thermocalcic aggregation of MFGM has been used to remove lipid interference in producing whey protein concentrate (Rombaut and Dewettinck, 2007; Damodoran, 2010). We expanded the scope and investigated the type of salt, salt concentration, and temperature on the precipitation efficiency of PLs in the beta stream of 5% solid concentration





FIG. 4. Recovery of the first and second extraction and residual total lipid (top) and PLs (bottom) from the calcium acetate-precipitated membrane lipid fragments in dairy processing waste stream. H: hexane, IP: isopropanol, ETH: ethanol, EA: ethyl acetate, DCM: dichloromethane, C: chloroform, M: methanol.

(Price, *et al*, 2020b). The use of zinc acetate and calcium acetate, along with mild heat treatment and pH adjustment, was shown to be effective in precipitating PLs and proteins into a pellet. The effective precipitation conditions were zinc acetate of 25 mM concentration at pH greater than 6.5 and 30°C that led to a >96% of both total and PLs recovery. Calcium acetate of greater than 75 mM concentration at pH greater than 6.5 and 60°C was able to precipitate >88% total lipid and >85% PLs (Fig 3). Even though the calcium salt is less effective compared to zinc salt, it precipitated less protein, and it has fewer health concerns. Extracting using 90% ethanol at 70°C, PLs recovery of >95% from the precipitate was achieved. However, salt was also extracted in the PLs fraction. This work demonstrates that calcium acetate precipitation has a great potential for producing valuable lipid fractions from the beta stream or other similar dairy processing by-products. But, an improved PLs extraction from the precipitate without salt co-extraction is needed.

# EXTRACTION OF PLS FROM THE PLS-SALT PRECIPITATE

We are now investigating the extraction of PLs from this MFGM fragment that was previously precipitated with salt. We began by using acetone to remove only the neutral lipids first, then tested solvents of different polarity to extract PLs. The calcium acetate precipitate has protein, salt, moisture, and neutral and polar lipids contents of 31, 32, 10, 17, and 10%, respectively. Acetone removed 13% of the mass as neutral lipid, and the residual was extracted with solvents. As shown in Figure 4, two sequential extractions with dichloromethane:methanol (2:1), hexane: isopropanol (3:2), and hexane: ethanol (1:2.5) (polarity index of 3.8, 1.6, and 3.7) resulted in relatively high total and PLs recovery in the first and second extraction compared to extraction with the standard chloroform:methanol (2:1, polarity index of 4.4). In contrast, the extraction with hexane: isopropanol (1:4) and ethyl acetate:ethanol (75:20) (polarity index of 3.2 and 4.6) was poor. Therefore, for such a PLs extraction, solvent polarity alone is not the determining factor.

This work is still ongoing, and more results will be forthcoming.

Tong Wang is a professor in the Department of Food Science at the University of Tennessee, Knoxville, USA. Her research programs focus on fats and oil chemistry, quality and functionality, and processing of commodity products or by-products, such as corn, soybean, egg, dairy, microbial biomass, and hemp.

# **Further reading**

Conway, V., S.F Gauthier, and Y. Pouliot, Buttermilk: much more than a source of milk phospholipids, *Anim. Frontiers 4*: 44–51, 2014.

Damodaran, S., Zinc-induced precipitation of milk fat globule membranes: a simple method for the preparation of fat-free whey protein isolate, *J. Agric. Food Chem. 58*: 11052–11057, 2010.

Price, N., T. Fei, C. Clark, and T. Wang, Application of zinc and calcium acetate to precipitate milk fat globule membrane components from a dairy by-product, *J. Dairy Sci.* 103: 1303–1314, 2020 b.

Price, N., T. Fei, S. Clark, and T. Wang, Extraction of phospholipids from a dairy by-product (whey protein

phospholipid concentrate) using ethanol, *J. Dairy Sci.* 101: 1–10, 2018.

Price, N., Z. Wan, T. Fei, S. Clark, and T. Wang, Development of industrially scalable method for phospholipids and branch-chain fatty acids of dairy by-product, *J. Am. Oil. Chem. Soc.*, 2020 a., DOI 10.1002/aocs.12377.

Rombaut, R. and K. Dewettinck, Thermocalcic aggregation of milk fat globule membrane fragments from acid buttermilk cheese whey, *J. Dairy Sci.* 90: 2665–2674, 2007.

Rombaut, R. and K. Dewettinck, Properties, analysis, and purification of milk polar lipids, *Int. Dairy J.*, *16*: 1362–1373, 2006.



# Determining Claudia Blum and Nicolai Kraut protein and fat in plant-based foods

- The fat and protein contents of five commercially available plant-based foods were determined using BUCHI's standard automated fat extraction and Kjeldahl solutions.
- The determined values for fat and protein content were in close agreement to the labelled nutrient content and delivered good repeatability with a relative standard deviation of 2% or less.
- The standard application parameters can be used, and no specific adaptation for these types of samples was necessary.

Plant-based burgers and other meat alternatives have become very popular, not only with vegans and vegetarians but also with consumers who are concerned about sustainability and health. Although these products are vegan, they have wide appeal and are viewed as trendy products for a contemporary lifestyle [1]. A meat substitute or meat analogue approximates certain qualities (primarily texture, flavor, and appearance) or chemical characteristics of a specific meat. The texture of meat is imitated by processing pea, soy, or wheat proteins, and red beet root juice or plant heme provide the color [2]. A special focus is placed on the proteins, as the protein ingredients are the most important components for product identity and product differentiation. The role of fats and oils in meat analogue formulations is to contribute to the juiciness, tenderness, mouthfeel, and flavor release of the product [3]. The content of proteins and fat are important for controlling the production process as well as for quality control of the final product. Nutritional labelling is a legal requirement and of interest to many health-conscious consumers.

We recently determined the fat and protein content of a wide variety of plant-based food samples with BUCHI's standard automated fat extraction and Kjeldahl solutions.

The samples were chosen to represent different protein sources (soy, wheat, pea) and various ways in which the proteins were processed (dried, extruded, and so on). The food samples used in the study are shown in Figure 1.

# FAT DETERMINATION WITH FATEXTRACTOR E-500

The fat content of foods is usually determined by solvent extraction according to different extraction methods. Other methods, such as near-infrared (NIR) or nuclear magnetic resonance (NMR), can also be used. The choice for a method depends on the purpose of the analysis (e.g., official nutrition labeling or rapid quality control), the daily sample load, and the need for compliance with official standard methods.

Crude fat or free fat is determined by pure solvent extraction. When the total fat content is determined, an acid hydrolysis prior to the solvent extraction is carried out. The acid hydrolysis releases all the encapsulated or bound fat from the sample matrix.

Soxhlet extraction is still the most widely used method for fat determination. It is a very robust and easy method that is demanded by many official standard methods. Fat determination according to the Weibull-Stoldt method includes acid hydrolysis and Soxhlet extraction.

The FatExtractor E-500 is designed for quick and compliant fat extraction according to Soxhlet, Randall, or



FIG. 1. Plant-based food samples used for this study prior to homogenization: soy schnetzel (top row, left); fish fingers based on wheat (top row, right); boiled sausage based on Tofu and wheat protein (bottom row, left); steak based on soy and wheat (bottom row, middle); and cold cuts based on pea and soy protein (bottom row, right)



FIG. 2. HydrolEx H-506 in combination with the FatExtractor E-500 Soxhlet for compliant and fast solvent extraction of fat according to the Weibull-Stoldt method

Twisselmann (Fig. 2). The HydrolEx H-506 offers a smooth and safe process with convenient system handling for acid hydrolysis as a sample preparation step prior to fat extraction for total fat determination.

The total fat content of the samples, as well as the free fat content (without prior acid hydrolysis), were determined. The results are shown in Table 1 and Figure 3.

All determined fat contents were higher when the samples were hydrolyzed before the fat extraction. This preparation step helps to release bound fats from the matrix and is advisable for plant-based foods.

# PROTEIN DETERMINATION WITH KJELMASTER K-375

The three-step Kjeldahl protein determination was established at the end of the 19th century by Johan Kjeldahl at the Carlsberg Breweries in Copenhagen, Denmark.

Despite its age, the method is still widely used as the reference according to most official norms worldwide. The method consists of three main steps, which include digestion to convert organic nitrogen to inorganic ammonium sulphate, distillation preceded by alkalization to separate ammonia from the digest, and titration for quantification of the ammonia. Due to the automation of the process, the method is safer and more application-friendly, with an impact on chemical savings.

The BUCHI KjelDigester K-449 along with the KjelMaster K-375 adjacent to the KjelSampler K-377 (Fig. 4, page 20) is designed for automated, high-sample throughput and a potentiometric or colorimetric titration for compliance to the broadest range of official methods.

The BUCHI standard method was applied [5] according to the freely available Kjeldahl Optimizer App [6]. Overall, the results (Table 2, page 20) for the protein content are in close accordance with the labelled values with only minimal deviations. TABLE 1. Free fat and total fat content determined in plantbased foods. (n=3, RSDs % in bracket) The samples were extracted with petroleum ether 40–60°C using Soxhlet extraction for 20 cycles. For total fat, the samples were hydrolyzed with 4 M HCl for 30 min prior to extraction.

	Free fat [g/100g]	Total fat [g/100g]
Soy schnetzel	0.10 (10.2)	1.66 (4.45)
Boiled sausage	15.8 (0.64)	16.8 (0.59)
Steak	10.7 (0.92)	11.8 (0.77)
Fish fingers	12.9 (1.67)	14.1 (0.89)
Cold cuts	11.2 (0.30)	12.1 (0.64)



FIG. 3. Mean values of the determined fat contents for total and free fat of five plant-based food samples. The error bars indicate the standard deviation (n=3)



# Proximate analysis without breaking the rules

- Innovative design for highest sample throughput in proximate analysis
- Full compliance to official methods according to AOCS, AOAC, and ISO
- Swiss-made quality ensures longevity



Extraction Solutions for fat and other extractables



Kjeldahl Solutions for protein determinations



Benefit from trade-in promo





FIG. 4. KjelDigester K-449 connected to the Scrubber K-415 for neutralisation of acidic fumes for increased longevity of the instruments and environmental protection; KjelMaster K-375 in combination with the KjelSampler K-377 for highest sample throughput

# TABLE 2. Comparison between labelled and determined protein content in g/100g (n=3, RSDs % in brackets)

	Labelled content [g/100g]	Determined content [g/100g]
Soy schnetzel	49	47.4 (0.04)
Boiled sausage	17	16.4 (0.11)
Steak	20	19.7 (0.95)
Fish fingers	12.6	12.8 (0.44)
Cold cuts	6.5	6.9 (1.32)

The consistency of the results (RSD < 2%) was exceptionally good, demonstrating that fat and protein contents of plant-based food matrixes can be determined with standard automated analytical methods.

Claudia Blum is the Global Product Manager for Extraction solutions at BUCHI Labortechnik AG in Switzerland. She has a background in food engineering and did her PhD in food chemistry. BUCHI is a leading solution provider in laboratory technology for R&D, quality control and production worldwide.

Nicolai Kraut works as a Product Specialist for Kjeldahl and Extraction at BUCHI Labortechnik AG in Switzerland. He has a background in pharmaceutical chemistry, holds a PhD in bioanalytical chemistry, and has experience in R&D and quality control in the food and pharmaceutical industry. He can be contacted at Kraut.N@buchi.com.

# References

[1] Rützler, H. 2019. Article from Food Report 2019, translated into English https://www.zukunftsinstitut. de/artikel/food/plant-based-food-der-neue-spin-beiersatzprodukten/.

[2] Wikipedia, definition of meat analogue, as seen on 10.03.2020. https://en.wikipedia.org/wiki/ Meat\_analogue.

[3] Bohrer, B. An investigation of the formulation and nutritional composition of modern meat analogue products, *Food Sci. Hum. Wellness 8*: 320–329, 2019.

[4] Application note 387/2020. Determination of total fat content in plant-based meat substitutes. Available for download at: https://www.buchi.com/ch-de/ node/10871.

[5] Application note 395/2020. Protein determination in plant-based meat alternatives. Available for download at: https://www.buchi.com/ch-de/ node/11642.

[6] Kjeldahl Optimizer App. Available for download at: https://www.buchi.com/en/service-support/ scientific-mobile-apps/kjeldahl-optimizer.

Additional Information

- Operating manual FatExtractor E-500
- Operating manual HydrolEx H-506
- Operating manual KjelMaster K-375
- Operating manual KjelDigester K-449
- Microsite Classical Extraction https://www.buchi.com/en/ classical-extraction-system





French Achiever presses offer outstanding features and reliability for full pressing to produce full press cake and crude oil. The press design yields high quality oil and cake with residual oils among the lowest in the industry when using the single pressing process. The Model 44, 55, and 66 presses can also be supplied for prepressing oilseeds to produce prepress cake prior to solvent extraction.

Since 1900, we have supplied durable equipment and systems for most commercial food and industrial uses. Our process solutions have a worldwide reputation for years of reliable operation with low life cycle costs.

Let us be Your Partner in Processing. Contact us for more information.



French Oil Mill Machinery Co. Piqua, Ohio, U.S.A. • 937-773-3420 www.frenchoil.com

# Per Munk NielsenDataDer Munk NielsenData

Phospholipase C (PLC) products have been on the market for several years already and are wellestablished in the water degumming of soybean oil to provide additional oil yield (Galhardo and Dayton). While developing the new PLC Quara<sup>®</sup> Boost for water degumming, my research group discovered that the enzyme could be integrated with the alkaline refining process. The result is a significant increase in oil yield and preservation of the final oil quality from caustic refining (Holm and Nielsen). Recently, the process efficiency has been confirmed in full-scale trials.

- Phospholipase C (Quara<sup>®</sup> Boost) enables higher oil yields in water degumming of vegetable oils.
- My research group at Novozymes recently documented that incorporating this enzyme directly into the alkaline refining process results in significantly higher yields and preserves the final oil quality from caustic refining.
- Enzyme-assisted alkaline refining is simple to establish and has a short payback time, as the typical oil yield is 15–20kg per ton.

# **THE PROCESS**

The typical alkaline refining process is seen in Figure 1. Some plants start the refining with a water degumming process and continue the process once the oil is degummed. They may already have considered using phospholipase C during the water degumming step to get a higher oil yield. Either way, water degumming includes a gum centrifugation step where some oil is inevitably lost.

Other plants go directly from the crude oil to the caustic step, which eliminates both the gums and the soap stock. Another option is to incorporate a chelating step that uses phosphoric acid to achieve the lowest possible phosphorous content (P-content) in the final oil.

The process our group developed for incorporating PLC Quara<sup>®</sup> Boost into the alkaline refining process is seen in Figure 2.

# PROCESSING



FIG. 1. Alkaline refining process



FIG. 2. Enzyme-assisted alkaline refining with phospholipase C (Quara Boost)

# THE ENZYME

The Quara Boost enzyme consists of two enzyme molecules that hydrolyze the phosphatidyl choline/ethanolamine (PC/PE) and phosphatidyl inositol (PI), respectively. The phosphatidic acid (PA) is not hydrolyzed. The extra oil yield is the sum of the released diglycerides (DG) and the neutral oil, typically in ratio 2:1. The critical parameters for an efficient enzyme reaction are pH in the water phase, temperature and time of reaction, and an efficient emulsification of the water phase in the oil. The preferred parameters are shown in the flow chart (Fig. 3).

## EXPECTED OIL YIELD INCREASE

The increase in yield depends on the amount of phospholipids in the crude oil, which varies considerably depending upon the location, bean/seed quality, and process technology. Phosphorous from the phospholipids in crude oil typically ranges from 400–1400ppm. Since PA is not converted, the amount of PA must also be taken into account.

From our experience working with phospholipase, we typically get a 75% conversion of the PC + PE + PI in the oil. The limitations in the hydrolysis are caused by different parameters, such as the extent to which the phospholipids are hydrated, variations in the process parameters, the efficiency of mixing, plus the duration of the reaction. Recalculating the expected increase in DG from the phospholipid hydrolysis provides an estimate, as seen in Figure 4 (page 24), based on the phosphorous numbers in the oil (5% PA in the case of the provided example). It should be noted that the numbers used in this recalculation are based upon <sup>31</sup>P-NMR data, which typically show a slightly lower P-content than the ICP-P method. In addition to a DG-increase, you can expect a yield of neutral



FIG. 3. Process parameters for the enzyme-assisted alkaline refining process



FIG. 4. Estimation of delta diglyceride (DG) from hydrolyzing the PC + PE + PI in soybean oil with 5% PA

oil in the order of magnitude of about 50% of the delta DG. For example, an oil with 900ppm P measured by the <sup>31</sup>P-NMR will have an estimated DG increase of 1.20%. With the release of neutral oil of 0.5\*1.20%, you may expect a yield increase of 1.8% oil.

The outcome of a laboratory experiment comparing conventional alkaline refining to enzyme-assisted alkaline refining is shown in Figure 5.

# **PROCESS CONSIDERATIONS**

Retrofitting an existing alkaline refining plant will require enzyme reactors and dosing and mixing equipment. The suggested holding time for the enzyme reaction is 1-2 hours in a tank system with some extent of plug flow. Plug flow can be generated in a tank divided into at least four compartments as shown in Figure 2. Another important detail is NaOH dosing, which is done at two places in the line—after the acid chelating step to adjust pH, and in the saponification step to neutralize the acid from the pre-treatment, the FFA, and the phosphatidic groups formed during the enzyme reaction. The amount of NaOH added at those two points are calculated based on the amount of acid added, FFA in the crude oil plus a small excess, and the amount of phospholipids that are hydrolyzed. This will be a slightly higher dosage than would be used without the enzyme process, as the phosphatidic groups formed as result of the enzyme reaction must be neutralized. Since the enzyme-assisted alkaline process eliminates water degumming, there will be additional yield savings from omitting one centrifugation step.

Per Munk Nielsen is senior science manager, Application Research Oils & Fats, Novozymes, Denmark. He can be contacted at pmn@novozymes.com.



FIG. 5. Centrifuge test showing soap stock in the enzyme-assisted alkaline refining compared to control

# References

1. Galhardo, F. and Dayton, C. Enzymatic degumming. https://lipidlibrary.aocs.org/edible-oil-processing/ enzymatic-degumming.

2. Holm, H.C, *et al.* 2018. Method for degumming and refining vegetable oil. Patent application WO 2018/171552.



# The second printing of the 7th edition of **Official Methods and Recommended Practices of the**

**AOCS** is now available! The updated AOCS Methods has four new official methods and revisions to fifteen previously approved methods.

Ensure your lab maintains proficiency and accuracy in testing by ordering your copy today!

# aocs.org/methods



**Setting the Standard** | Since the 1920s, the global fats and oils industry has relied on the analytical integrity of the *Official Methods and Recommended Practices of the AOCS*. AOCS has set the standard for analytical methods critical to processing, trading, utilizing, and evaluating fats, oils, lipid products, and proteins. Worldwide acceptance has made the *AOCS Methods* a requirement wherever fats and oils are analyzed.

### **ADDITIONS**

AOCS Official Method Ca 17a-18 Determination of Trace Elements in Oil by Inductively Coupled Plasma Optical Emission Spectroscopy

Joint JOCS/AOCS Official Method Cd 29d-19 2-/3-MCPD Fatty Acid Esters and Glycidyl Fatty Acid Esters in Edible Oils and Fats by Enzymatic Hydrolysis

Joint JOCS/AOCS Recommended Practice Cd 29e-19

2-/3-MCPD Fatty Acid Esters and Glycidyl Fatty Acid Esters in Fish Oils by Enzymatic Hydrolysis

Joint JOCS/AOCS Official Method Ch 3a-19 Determination of the Composition of Fatty Acids at the 2-Position of Oils and Fats-Enzymatic Transesterification Method using *Candida antarctica* Lipase

### REVISIONS

AOCS Standard Procedure Ba 6a-05 Crude Fiber in Feed by Filter Bag Technique AOCS Official Method Cc 7-25 Refractive Index of Fats and Oils AOCS Official Method Cd 26-96 Stigmastadienes in Vegetable Oils AOCS Official Method Cd 27-96 Steroidal Hydrocarbons in Vegetable Oils AOCS Official Method Cd 3d-63 Acid Value of Fats and Oils AOCS Official Method Cd 29c-13 2- and 3-MCPD Fatty Acid Esters and Glycidol Fatty Acid Esters in Edible Oils and Fats by GC/

MS (Difference Method) **AOCS Official Method Ce 8-89** Tocopherols and Tocotrienols in Vegetable Oils

Tocopherols and Tocotrienols in Vegetable Oils and Fats by HPLC

AOCS Official Method Ch 3-91 Fatty Acids in the 2-Position in the Triglycerides of Oils and Fats

AOCS Official Method Ch 5-91 Specific Extinction of Oils and Fats, Ultraviolet Absorption

AOCS Analytical Guidelines Ch 7-09 International Trade Standard Applying to Olive and Olive-Pomace Oils

AOCS Official Method Ch 8-02 Wax Content by Capillary Column Gas-Liquid Chromatography

AOCS Procedure M 1-92 Determination of Precision of Analytical Methods

AOCS Procedure M 3-82 Surplus Status of Methods

AOCS Criteria M 5-09 Approved Chemists (Criteria)

AOCS Criteria M 6-09 Certified Laboratories (Criteria)

New and revised methods included in the 2020 Additions and Revisions may also be purchased individually as PDF downloads.

# **Bio-based (edible) oils:** Raj Shah, Nathan Aragon, and John Calderon feedstock for lubricants of the future

There has recently been a shift in research toward improving bio-lubricants that use base oils made from biodegradable feedstocks. Vegetable oils are attractive base oils for bio-lubricants because they are mostly biodegradable and are made from edible feedstocks. However, vegetable oil-based lubricants are not capable of completely replacing the more standard petroleum-based lubricants, because vegetable oils lack the physical properties (oxidative stability, thermal stability, and viscosity range) that give petroleum-based lubricants their high performance. The physical properties of bio-lubricants can be enhanced using additives, blending, or modification by chemical means, but such enhancements can increase the total cost and toxicity, and decrease biodegradability [1]. Due to enhanced environmental guidelines and regulations in the food-processing industry, the global market for bio-lubricants is expected to grow from \$2 billion to \$2.4 billion in the next five years [2]. The estimated growth is seen in Figure 1. For that to occur, there is a wide gap that must be bridged between the petroleum-based lubricants and bio-lubricants, and this seems to be where the bulk of the research will go. Recent growth in the healthcare and food processing industries demonstrates a need for lubricants that are less toxic and more biodegradable, so research aimed toward enhancing current bio-lubricants will be key in the near future [1].

- The global market for bio-lubricants is expected to grow by 20% in the next five years.
- Recent growth in the healthcare and food-processing industries demonstrates a need for lubricants that are less toxic and more biodegradable.
- Research aimed at enhancing the performance of current bio-lubricants will be key to meeting this need.

The term "vegetable oil" was introduced previously, but this is a broad term that encompasses several oils made from different feedstocks and used for different applications. Canola oil is used to make food-grade lubricants, hydraulic oils, and metal working fluids. Coconut oil is mainly used in gas engines. Palm oil is used in the steel industry and is used to make greases. Rapeseed oil is used in farm equipment and in the fabrication of biodegradable greases. Soybean oil has a wide variety of uses, including fabrication of soaps, shampoos, detergents, and pesticides, and acts as an ingredient in various lubricants, biodiesel fuel, and hydraulic oil. Sunflower oil is used for greases and diesel fuel [3]. As such, the versatility of vegetable oils spans a multitude of industries,

# More Informed poster summary

A poster summary of this topic with additional figures and graphics is available at bit.ly/biolubricant.

# LUBRICANTS



and many express key interests in utilizing vegetable oils as bio-lubricants—especially industries that are environmentally aware and face strict environmental regulations.

Key industries that apply bio-lubricants include the marine, food processing, agricultural, and pharmaceutical industries, as well as underground mining. Other industries that show a large interest are commercial transportation and the automotive industry [2]. This is due to the biodegradability and low toxicity of bio-lubricants in addition to the benefit of lower emissions from using them [4]. However, there are some factors to consider when comparing vegetable oils and mineral, or petroleum-based, oils. Mineral oils tend to have better stability with respect to hydrolysis and oxidation than vegetable oils. They also tend to have superior low-temperature performance [5]. For example, one study found the pour points of soybean oil and 90% sunflower oil to be -9°C and -12°C, respectively, while the pour point of mineral oil was -21°C. The same study also found the percentage of micro-oxidation after 30 minutes of soybean oil and 90% sunflower oil to be 48% and 13%, respectively, while the percentage of micro-oxidation of mineral oil after the same time was found to be 5% [6]. The extent of micro-oxidation is an important factor, because any oil that undergoes more oxidation will be less stable.



### FIG. 1. Estimated growth of the bio-lubricant industry by 2025 [2]

Economic and social concerns about the effects of large-scale usage of edible oils on seasonal-dependent food markets are a major concern as well. The food markets are examples of environments where non-toxic and biodegradable lubricants are required. Research and development of TABLE 1. NSF International registers the formulations of lubricants for food-processing applications. Food-grade lubricants are understood to be NSF H1-registered lubricants for incidental contact with food and beverages [1–4].

Description and Use

Desen	
H1	<ul> <li>Lubricants with incidental contact with edible products</li> <li>Food-grade lubricants</li> <li>Contain only chemicals that meet CFR 21, Section 178.3570</li> <li>Registered for incidental contact with food and beverages</li> <li>Used for equipment in direct contact with food products</li> <li>May be used as protective anti-rust film, release agent, lubricant</li> </ul>
H2	<ul> <li>Lubricants with no contact with edible products</li> <li>Not food-grade lubricants</li> <li>Not required to meet CFR 21, Section 178.3570</li> <li>Contain no carcinogens, mutagens, teratogens, mineral acids, odorous substances, or intentionally added heavy metals</li> <li>For use on equipment in food processing facilities where there is no possible contact with food and beverages</li> <li>May be used as protective anti-rust film, release agent, lubricant</li> </ul>
НЗ	<ul> <li>Soluble oils with incidental contact with edible products</li> <li>Contain only chemicals that meet CFR 21, Section 178.3570</li> <li>Registered for incidental contact with food and beverage</li> <li>Chemically acceptable for application to hooks, trolley, etc.</li> <li>Used to clean and prevent rust</li> <li>Portions of equipment that contact edible products must be cleaned before re-use</li> </ul>
HT-1	<ul> <li>Heat transfer fluids with incidental contact with edible products</li> <li>Registered for incidental contact with food and beverages</li> <li>Contains only chemicals that meet CFR 21, Section 178.3570</li> </ul>
HT-2	<ul> <li>Heat transfer fluids with no contact with edible products</li> <li>Not required to meet CFR 21, Section 178.3570</li> <li>Contains no carcinogens, mutagens, teratogens, mineral oils, odorous substances, or intentionally added heavy metals</li> <li>For equipment in food processing facilities where there is no possible contact with food or beverages</li> </ul>
HX-1	<ul> <li>Food-grade lubricant components/ingredients</li> <li>Registered and listed by NSF International for use in H1 formulations</li> <li>Comply with CFR 21, Section 178.3570</li> </ul>

improved edible oil-based bio-lubricants is driven by these sensitive environments and the push for safe and sustainable lubricants. Consequently, many industries are poised to benefit from such research.

### **STANDARDS**

Depending on the industry, the production and use of lubricants must follow specific standards for performance and environmental regulations established by numerous international standards organizations and environmental protection regulators. ASTM issues international standards for lubricants through their D02 Committee [7]. Other institutions that set standards for lubricants and the petroleum industry include SAE International, the American Petroleum Institute (API), the International Lubricant Standardization and Approval Committee (ILSAC), and the International Organization for Standardization (ISO) [8]. The US Environmental Protection Agency (EPA) lists different categories of environmentally acceptable lubricants, which include vegetable oils, synthetic esters, polyalkylene glycols, and water [9]. The US Department of Agriculture (USDA) and the National Sanitation Foundation (NSF) also have food grades specifically for lubricants categorized as H1–H3, with each category representing the lubricant's acceptable exposure levels for food as shown in Table 1 [10]. The H1 category is for lubricants that may come into direct contact with food, the maximum limit of lubricant in food being 10 ppm. The H2 category applies to lubricants that are used in equipment where there is no possibility of direct contact with food, while the H3 category is for lubricants used for cleaning and preventing rust on equipment [1].

Although performance and environmental/toxicity specifications for food-grade lubricants are well defined and heavily regulated, what specifically constitutes a "biolubricant" may not be defined and can change depending on the industry and country [1]. Some organizations may consider only biodegradability as a criterion, while others may also incorporate renewable feedstocks as a requirement. Consequently, biodegradable synthetic-based lubricants may be classified as "bio-lubricants." According to the EPA's designation of Environmentally Acceptable Lubricants (EALs) used to determine environmental limits for lubricants used in marine environments, at least 90% of the formulation must be readily biodegradable for oil lubricants and 75% for grease lubricants, with the rest being either inherently biodegradable or non-biodegradable but non-bio accumulative [11]. The EU's Ecolabel has similar requirements but separates lubricants into the following classes: Total Loss Lubricant (TLL), Partial Loss Lubricant (PLL), and Accidental Loss Lubricant (ALL). Each class represents lubricants based on their application and have different biodegradable specifications/limits as summarized in Table 2 [12]. As such, bio-lubricants and bio-lubricant manufacturers must have these specifications in mind when forming bio-lubricant compositions alongside general lubricant specifications, such as ISO/SAE grades.

	ALL	PLL	TLL	Greases (ALL,PLL,TLL)
Readily aerobically biodegradable	> 90	> 75	> 95	> 80
Inherently aerobically biodegradable	≤ 10	≤ 25	≤ 5	≤ 20
Non-biodegradable and non-bioaccumulative	≤ 5	≤ 20	≤ 5	≤ 15
Non-biodegradable and bioaccumulative	≤ 0,1	≤ 0,1	≤ 0,1	≤ 0,1

TABLE 2. EU Ecolabel biodegradability limits of lubricants classified by loss cases [12]. Cumulative mass percentage (%w/w) limits for substances present in the product with respect to their biodegradability and bio-accumulation potential.

# ADVANCEMENTS IN BIO-LUBRICANT RESEARCH

As mentioned before, it is well known that bio-lubricants, in comparison to mineral oils, show a large degree of biodegradability, a low amount of toxicity, high flash points, high viscosity indices, and better adherence to metal, but suffer from low pour points and oxidative stability. Table 3 compares the viscosities and pour points of many common mineral oil-based lubricants with numerous edible and non-edible natural oil-based bio-lubricants. Higher grades of petroleum lubricants possess significantly lower pour points down to -54°C, whereas the lowest pour points of the bio-lubricants only reached -27°C, indicating reduced low temperature performance. Oxidative stabilities of a few petroleum lubricants were orders of magnitude better than those of the bio-lubricants as well [13].

To take advantage of the high viscosities, biodegradability, and flash points, much of the research done so far has attempted to improve the weaknesses of the vegetable oil base, i.e., the thermal and oxidative stabilities through chemi-

# TABLE 3. Relevant physiochemical properties of common mineral and vegetable oils, including viscosities, flash and pour points, and oxidative stabilities [13]

Lubricant	Viscosity 40°C (cSt)	Viscosity 100°C (cSt)	Viscosity Index <sup>1</sup>	Pour Point (°C)	Flash Point (°C)	Oxidative Stability (min)	Coecient of Friction	Wear (mm)
Mineral oils								
ISO VG32	>28.8	>4.1	>90	-6	204	-	-	-
ISO VG46	>41.4	>4.1	>90	-6	220	-	-	-
ISO VG68	>61.4	>4.1	>198	-6	226		-	-
ISO VG100	>90.0	>4.1	>216	-6	246	1640.26	-	-
Paran VG45	95	10	102	-	-	-	-	-
Paran VG460	461	31	97	-	-	-	-	-
R150	150	-	-	-	195	931.16	-	-
SAE20W40	105	13.9	132	-21	200	-	0.117	0.549
AG100	216	19.6	103	-18	244	-	-	-
75W-90	120	15.9	140	-48	205	-	-	-
75W-140	175	24.7	174	-54	228	-	-	-
80W-140	310	31.2	139	-36	210	-	-	-
Vegetable oils								
Castor oil	220.6	19.72	220	-27	250	-	-	-
Coconut oil	24.8	5.5	169	21	325	-	0.101	0.601
Cottonseed oil	33.86	7.75	211	-	252		-	-
Jatropha oil	35.4	7.9	205	-6	186	5	_	_
Lesquerella oil	119.8	14.7	125	-21	_	-	0.045	0.857
Moringa oil	44.9	-	-	-	204	28.27	-	-
Palm oil	52.4	10.2	186	-5	_	_		
Passion fruit oil	31.78	_	_	_	228	7.5	_	
Pennycress oil	40.0	9.3	226	-21	-	-	0.054	0.769
Olive oil	39.62	8.24	190	-3	318	-	-	-
Rapeseed oil	45.60	10.07	180	-12	252	-	-	-
Rice bran oil	40.6	8.7	169	-13	318	_	0.073	0.585
Sesame oil	27.33	6.3	193	-5	316	-	_	_
Soybean oil	28.86	7.55	246	-9	325	-	_	_
Sunflower oil	40.05	8.65	206	-12	252	-	-	-

<sup>1</sup> Viscosity index: (<35) low, (35–80) medium, (80–110) high, (>110) very high.

# References

[1] https://www.newfoodmagazine. com/article/100207/ the-growth-of-the-bio-lubricant-industry/.

[2] https://www.marketsandmarkets.com/Market-Reports/biolubricants-market-17431466.html.

[3] Panchal, T.M., Patel, A., Chauhan, D.D., Thomas, M., and Patel, J., "A methodological review on biolubricants from vegetable oil-based resources," *Renew. Sust. Energ. Rev.* 70: 65–70, 2017.

[4] Salimon, J., Salih, N., and Yousif, E., "Biolubricants: raw materials, chemical modifications, and environmental benefits," *Eur. J. Lipid Sci. Technol.* 112: 519–530, 2010.

[5] Mobarak, H.M., *et al.*, "The prospects of biolubricants as alternatives in automotive applications," *Renew. Sust. Energ. Rev. 33*: 34–43, 2014.

[6] Erhan, S.Z. and S. Asadauskas, "Lubricant basestocks from vegetable oils," *Ind. Crop. Prod.* 11: 277–282, 2000.

[7] https://www.astm.org/ABOUT/ OverviewsforWeb2014/chemical\_overview\_2016.pdf

[8] https://www.thelubricantstore.com/ important-lubricant-organizations

[9] https://www3.epa.gov/npdes/pubs/vgp\_ environmentally\_acceptable\_lubricants.pdf

[10] https://www.newfoodmagazine.com/ article/1417/food-grade-lubricants-are-theyenvironmentally-friendly-and-biodegradable/

[11] https://www.lelubricants.com/lubricants/ environmentally-acceptable-lubricants/

[12] https://eur-lex.europa.eu/legal-content/EN/ TXT/?qid=1542112371100&uri=CELEX:32018D1702

[13] Cecilia, J.A., *et al.*, "An Overview of the Biolubricant Production Process: Challenges and Future Perspectives," *Processes 8*: 257, 2020.

[14] Balakrishnan, M., *et al.*, "Novel pathways for fuels and lubricants from biomass optimized using lifecycle greenhouse gas assessment," *Proc. Natl. Acad. Sci. USA*. *112*: 7645–7649, 2015.

[15] Liu, S., *et al.*, "Renewable lubricants with tailored molecular architecture," *Science Advances 5*: eaav5487, 2019.

[16] https://bioplasticsnews.com/2019/12/24/ neste-full-industrial-biolubricant/ cal modification or additives. The inherent unsaturation found in plant oils makes them perform poorly in extreme temperature conditions, so chemical modification, such as epoxidation of the double bonds, has been used to overcome this. Epoxides are generated by forming a peroxy acid and then reacting the peroxy acid with the carbon-carbon double bond. The epoxidation has been shown to increase resistance to heat in vegetable oil-based polymer PVC plasticizers/additives, allowing for high-temperature applications [3]. Further esterification of the epoxides by a ring-opening reaction using fatty acids and *p*-toluenesulfonic acid (PTSA) has been shown to improve low-temperature activity due to long mid-chain esters disrupting macrocrystallization at low temperatures. Some fatty acids used in this reaction include octanoic, non-anoic, lauric, and myristic acids.

Transesterification has also been used to synthesize biodegradable organic polyesters using plant oils and molecules such as trimethylolpropane (TMP) and pentaerythritol (PE). Biodegradable TMP was formed from rapeseed oil fatty acids using sodium methylate as a catalyst. Transesterification of TMP with esters from palm oil was done using sodium methoxide as a catalyst. Polyesters made from PE and erucic acid were formed using *p*-PTSA in xylenes as a catalyst. All these polyesters have displayed improved low-temperature properties and oxidative stabilities [4].

Various antioxidants have been used as additives, but more research is needed to find antioxidants with lower toxicity. Antioxidants, such as vitamin C, vitamin E, esters from gallic acid, and derivatives of citric acid, are all naturally occurring and can serve as possible replacements to more toxic antioxidants, but more investigation is needed on the effectiveness of these replacements [4]. Other additives, such as plant-derived cysteine Schiff base esters, have shown excellent anticorrosion, antiwear, and antifriction properties, while non-toxic inorganic nanoparticle additives, such as ZnO and CuO, provide similar benefits in regards to antiwear and friction performance and are already used in many petroleum based lubricant formulations [13].

Regarding the commercial application of bio-lubricants, many current advancements have attempted to bridge the wide gap between the bio-lubricants and mineral oils. An American company named Novvi LLC has developed a cycloalkane base oil using lignocellulose derived from sugar cane that is completely renewable and meets the H1 certification from the NSF [1]. Biomass-based lubricants made from the condensation of ketones were synthesized that have good pour points, but they still need improvement with respect to the viscosity index values [14]. Researchers at the Catalysis Center for Energy Innovation (CCEI) at the University of Delaware have synthesized renewable base oils from biomass and natural oils that have specialized functional groups and molecular architectures. Their pour points and viscosity indices are comparable to commercially available base oils, but their extent of biodegradation needs to be analyzed [15]. A recent collaboration between Neste and AT-Tuote claims to have the world's first 100% bio-based MORE RESEARCH NEEDS TO BE DONE TO DETERMINE THE ACTUAL BIODEGRADABILITY OF NEW RENEWABLE BASE OILS AND THE COST-EFFECTIVENESS OF THE CURRENTLY KNOWN CHEMICAL MODIFICATIONS.

industrial lubricant. The lubricant uses Neste's MY Renewable Isoalkane<sup>™</sup> as a key component and can comply with the OECD 31 test for biodegradability [16]. Similar commercial investments will only continue to grow as bio-lubricants improve in performance and research and development and environmental regulations grow stricter.

# **MORE RESEARCH NEEDED**

Significant progress has been made with regard to the performance and commercial investment of bio-lubricants. Although petroleum-based lubricants are still preferred for low-temperature performance and oxidative stability, numerous methods have been explored and developed to allow bio-lubricants to compete with them. Chemical modification of plant oil-derived fatty acids by saturating the carbon-carbon double bonds through epoxidation and esterification has shown to be effective in the improvement of extreme temperature properties and oxidative stabilities of bio-lubricants. Possible replacements for antioxidant additives used in bio-lubricants that are more natural and less toxic have been proposed, which can preserve the enhancement of oxidative stability. More focus has also been placed on renewable sources of base oils, and this has brought base oils made from sugar cane and other types of biomass.

For the future, more research needs to be done to determine the actual biodegradability of new renewable base oils and the cost-effectiveness of the currently known chemical modifications. There is also much consideration regarding the use of bio-lubricants in the automotive industry, but for this to move forward, further studies on the tribological performance of bio-lubricants need to be completed [5]. While the greater financial cost of bio-lubricants is a concern, newer bio-lubricant formulations will be safe to use in industries with many environmental regulations and can be applied to total-loss systems without harming the environment. Continued commercial investment and advances in bio-lubricant performance and lower-cost synthesis methods will enable wider bio-lubricant applications and contribute to a more sustainable and clean future.

Raj Shah is a Director at Koehler Instrument Company in New York, where he has worked for the last 25 years. With a PhD in Chemical Engineering from Penn State University and a Fellow from the Chartered Management Institute, London, Raj has been an active member of AOCS for the last 2 decades. An Adjunct Professor in the Department of Material science and Chemical engineering at State University of New York, Stony Brook, Shah has been elected a Fellow by his peers at STLE, AIC, NLGI, INSTMC, CMI, IChem E, The Energy Institute, and The Royal Society of Chemistry. He is also a Chartered Scientist with the Science Council, a Chartered Petroleum Engineer with the Energy Institute, A Chartered Chemist with the Royal Society, and a Chartered Engineer with the Engineering council, UK. He has over 250 publications and is a co-editor of various books related to Fuels and oils. He can be reached at rshah@koehlerinstrument.com. More information on Dr. Shah can be found at https://www.petro-online.com/news/fuel-for-thought/13/ koehler-instrument-company/dr-raj-shah-director-at-koehler-instrument-companynbspconferrednbspwith-multifarious-accolades/53404.

John Calderon and Nathan Aragon are chemical engineering students at Stony Brook University, where Shah is the Chair of the external advisory board of directors. Both students are also part of a thriving internship program at Koehler instrument Company in Holtsville, New York, USA.

# Is herd immunity possible without a vaccine? Probably not.

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

# **Rebecca Guenard**

Every fall, as the weather cools and we pull out our sweaters, we also begin dreading the thought of cold and flu season. By January—after greeting treat-or-treaters, gathering with family for Thanksgiving, attending holiday parties, and taking part in all the other end-of-year festivities— we have usually already caught a cold.



FIG. 1. Top—A community outbreak with few people infected (red), and the rest healthy but unimmunized (blue); the illness spreads freely through the population. Middle—a population where a small number have been immunized (yellow); those not immunized become infected. Bottom—a large proportion of the population have been immunized; this prevents the illness from spreading significantly, including to unimmunized people. Source: Tkarcher. In a recent *Nature* article, Caroline Buckee, an epidemiologist at Harvard T.H. Chan School of Public Health in Boston, Massachusetts, USA, explained the reason colds and flus return every winter (https://tinyurl.com/heardimmunitynature). Seasonal coronaviruses that cause common colds provoke a waning immunity that seems to last approximately a year, Buckee told the journal. "It seems reasonable as a hypothesis to assume this one will be similar," she said. By "this one," she is of course referring to the novel coronavirus, or COVID-19.

As the pandemic drags past its twelfth month, there is a growing assertion that the more people who contract the virus, the faster it will be extinguished by mass immunity. However, no scientific evidence supports that theory. The only way to achieve mass immunity to COVID-19 is mostly likely through vaccination.

In the early 2000s, the first highly pathogenic, deadly coronavirus swept around the world. Researchers designated it SARS-CoV, which stands for Sever Acute Respiratory Syndrome. Ten years later, the more deadly MERS-CoV emerged. Each of these viruses were identified as having the ability to cross from animal to human. Both also have the same spiked membrane with single-stranded RNA tucked inside that can replicate quickly in their host's cells. With the arrival of SARS-CoV-2 (also called COVID-19), researchers are realizing that, despite similarities, each new virus emerges and spreads differently.

The novel coronavirus has been spreading around the world for more than a year now, yet so much is still unknown. We do know that the virus is highly infectious and spreads through contact with aerosol droplets. We also know the virus thrives in cold, dry air with limited sunlight. As winter sets in, and these ambient conditions become routine, epidemiologists predict that the rate of infection will increase.

Unlike many government officials and some researchers who tout herd-immunity, they are concerned this will have a deadly outcome.

The herd-immunity concept is based on an idea typically associated with vaccinations. A population with a large number of vaccinated people protect those who cannot receive a vaccine—such as someone with a compromised immune system—by reducing the chance that they will come into contact with the virus.

The concept should not be applied to the novel coronavirus, at least not yet. The general population is assuming COVID-19 acts like the chicken pox or measles whose antibodies induce a lifelong immunity. However, more research is needed to determine how long someone's immunity lasts after they have contracted COVID-19.

A man in Hong Kong recently reported contracting the virus a second time nine months after the initial outbreak in Wuhan, China. Another individual in Nevada, USA, also reported reinfection (https://tinyurl.com/naturereinfection). The two cases showed widely varying immune responses. In the first case, the person was asymptomatic when reinfected. In the second, the symptoms were worse the second time around.

These results trouble epidemiologists. Either the immune system recognizes the virus a second time and neutralizes it, or a revisit by the virus sends the immune system into overdrive again and it attacks tissue and organs. As is common with COVID-19, there are still many questions. "If the people who are infected become susceptible again in a year, then basically you will never reach herd immunity" through natural transmission, Caitlin Rivers, an epidemiologist at the Johns Hopkins, in Baltimore, Maryland, USA, told *Nature*.

Studies on the duration of COVID-19 antibodies seem to indicate that the yearly infection Rivers describes is likely. In a recent study, conducted by researchers at Massachusetts General Hospital in Boston, the blood of 343 individuals known to have had a SARS-CoV-2 infection was analyzed for antibodies (https://doi.org/10.1126/sciimmunol.abe0367). The goal was to determine the persistence and decay of antibody responses following infection. The study lasted for 122 days and found that at 90 days only three of the participates showed antibody decay. The authors of the study conclude that immunization from natural transmission lasts about five months. Similar findings have been reached in other experiments, though this research was conducted on individuals with severe cases. Studies on individuals with mild or asymptomatic cases show that their antibodies decay more rapidly (https: doi. org/10.1056/NEJMc2025179).

These results are too new to influence policy on dealing with SARS-CoV-2 should the virus be a yearly occurrence. The novel coronavirus has not been in existence long enough for a critical mass of research to have accumulated. By the same reasoning, there has not been enough time to assume herd-immunity can be achieved if more people are exposed to the virus. The growing evidence actually indicates the opposite.

# **Information**

From SARS and MERS to COVID-19: a brief summary and comparison of severe acute respiratory infections caused by three highly pathogenic human coronaviruses, Zhu, Z., *et al., Respir. Re.s 21*: 224, 2020.

Scientific consensus on the COVID-19 pandemic: We need to act now, Alwan, N.A., *et al.*, October 15, 2020.

Orthogonal SARS-CoV-2 serological assays enable surveillance of low prevalence communities and reveal durable humoral immunity, Ripperger, T.J., *et al.*, *Immunity*, October 13, 2020.

Persistence and decay of human antibody responses to the receptor binding domain of SARS-CoV-2 spike protein in COVID-19 patients, Iyer, A.S., *et al.*, *Science Immunology 5*: 52, 2020.

To counter a growing belief in herd-immunity among the general public and government entities, a group of scientists published a memorandum in *The Lancet* (https://tinyurl.com/ y65hjv5l). They called the hope for herd-immunity a "dangerous fallacy unsupported by scientific evidence."

As of press time, COVID-19 had claimed 1.48 million lives globally. Early data indicated that vaccines being developed by Pfizer and Moderna were more than 90% effective, and both companies applied for emergency use authorization from the US Food and Drug Administration. Vaccination of health care workers and at-risk populations may already be underway by the time this issue is published. But much testing remains to be done, and experts say that even after a COVID-19 vaccine is found to be effective it could still take a year to reach the general public.

The odds of dying from COVID-19 are still higher than the flu, but healthcare workers have learned how to treat the virus more affectively since the pandemic started. The chance of dying from COVID-19 are lower this January than they were last year. But a year of relentless cases is straining the healthcare system.

If the public assumes that contracting the virus means long-term immunity and discontinues precautions, our healthcare systems would be compromised. Washing hands, maintaining social distancing, and wearing a mask are the easiest ways to limit the spread of the novel coronavirus. It is not going away anytime soon. It shares many of the biological traits of the common cold, and like the common cold we are destined to face it yearly—until we have a vaccine.

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

# AOCS Laboratory Proficiency Program Demonstrate your technical expertise to customers



# "

The Lab Proficiency Program (LPP) is important to our business because it gives us, as well as our customers, an opportunity to see our lab's quality of testing, competency, and its proficiency level repeatability on a national scale! Being an LPP Award winner increases our professional level of confidence. Thank you AOCS!

> ESTRA BATTEN Lab Coordinator, JLA International AOCS member 2019–2020 LPP Award Winner

Each year, AOCS recognizes top labs and chemists for their rigorous dedication to achieving precise analytical results. Become a fullyear LPP participant by **May 20** to



be eligible for the 2020–2021 LPP awards and 2021–2022 **AOCS Approved Chemists** status.



**Enroll at aocs.org/series** The next enrollment deadline is February 20.

+1 217-693-4810 | technical@aocs.org | aocs.org/series

attain

Aflat

The AOCS Lab Proficiency Program is administered according to ISO 17043, and your participation may satisfy national and professional accreditation requirements.

# EU adopts Green Deal chemicals strategy

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

The European Commission has adopted a new EU Chemicals Strategy for Sustainability. The Strategy, which was adopted on October 14, 2021, is the first step toward a zero-pollution ambition for a toxic-free environment announced in the European Green Deal. According to an EU Commission press release, the Strategy will boost innovation for safe and sustainable chemicals, and increase protection of human health and the environment against hazardous chemicals. This includes prohibiting the use of the most harmful chemicals in consumer products such as toys, childcare articles, cosmetics, detergents, food contact materials, and textiles, unless proven essential for society, and ensuring that all chemicals are used more safely and sustainably.

The Strategy sets out concrete actions to make chemicals safe and sustainable by design and ensure that the most harmful chemicals for human health and the environment are avoided for "non-essential societal use," which includes use in consumer products.

# INCREASING PROTECTION OF HEALTH AND THE ENVIRONMENT

Flagship initiatives include:

- Phasing out from consumer products, such as toys, childcare articles, cosmetics, detergents, food contact materials, and textiles, the most harmful substances, which include among others endocrine disruptors, chemicals that affect the immune and respiratory systems, and persistent substances such as per- and polyfluoroalkyl substances (PFAS), unless their use is proven essential for society;
- Minimizing and substituting as far as possible the presence of substances of concern in all products. Priority will be given to those product categories that affect vulnerable populations and those with the highest potential for circular economy;
- Addressing the combination effect of chemicals (cocktail effect) by taking better account of the risk that is posed to human health and the environment by daily exposure to a wide mix of chemicals from different sources; and
- Ensuring that producers and consumers have access to information on chemical content and safe use, by introducing information requirements in the context of the Sustainable Product Policy Initiative.

# BOOSTING INNOVATION AND PROMOTING EU'S COMPETITIVENESS

In 2018, Europe was the second biggest producer of chemicals (accounting for 16.9% of sales). Chemical manufacturing is the fourth largest industry in the EU, directly employing approximately 1.2 million people. 59% of chemicals produced are directly supplied to other sectors, including health, construction, automotive, electronics, and textiles. Global chemicals production is expected to double by 2030, and the already widespread use of chemicals is likely to also increase, including in consumer products.

The Strategy envisages the EU industry as a globally competitive player in the production and use of safe and sustainable chemicals. As far as possible, new chemicals and materials must be safe and sustainable by design, i.e., from production to end of life. The actions announced in the Strategy are intended to support industrial innovation so that such chemicals become the norm on the EU market and a benchmark worldwide by:

- Developing safe-and-sustainable-by-design criteria and ensuring financial support for the commercialization and uptake of safe and sustainable chemicals;
- Ensuring the development and uptake of safe and sustainable-by-design substances, materials, and products through EU funding and investment instruments and public-private partnerships;
- Considerably stepping up enforcement of EU rules both at the borders and in the single market;
- Putting in place an EU research and innovation agenda for chemicals, to fill knowledge gaps on the impact of

chemicals, promote innovation, and move away from animal testing; and

 Simplifying and consolidating the EU legal framework—e.g., by introducing the "One substance one assessment" process, strengthening the principles of "no data, no market," and introducing targeted amendments to REACH and sectorial legislation, to name a few.

Commissioner for Health and Food Safety Stella Kyriakides said: "Our health should always come first. That is exactly what we have ensured in a Commission flagship initiative such as the Chemical Strategy. Chemicals are essential for our society and they must be safe and sustainably produced. But we need to be protected from the harmful chemicals around us. This Strategy shows our high level of commitment and our determination to protect the health of citizens, across the EU."

# **REACTION FROM INDUSTRY**

During a Chemical Watch conference which coincided with the publication of the strategy, Cefic product stewardship direc-

tor, Sylvie Lemoine, said "one of the first things we should do is define 'sustainable by design,'" adding that a definition is important because "my sustainability could be very different to your sustainability."

Many high-level discussions have taken place recently on the definition of "essential use." In the strategy, the Commission pledges to "define criteria for essential uses to ensure that the most harmful chemicals are only allowed if their use is necessary for health, safety or is critical for the functioning of society and if there are no alternatives that are acceptable from the standpoint of environment and health. These criteria will guide the application of essential uses in all relevant EU legislation for both generic and specific risk assessments."

As the Communication about the new Strategy is not a legislative proposal, it will not be subject to scrutiny by the European Parliament or the Council of Ministers. The Commission will make legislative and non-legislative proposals to implement the strategy, which are due to be presented next year, and will be carried out within the framework of existing legislation.

# AOCS Thomas H. Smouse **Memorial** Fellowship

### **Major Contributors:**

AOCS AOCS Foundation Archer Daniels Midland Foundation

### **Benefactors:**

American Fats and Oils Association Mr. and Mrs. Edward Campbell Dr. Stephen S. Chang Fabrica De Jabon la Corona, S.A. DE C.V. National Institute of Oilseed Products Mrs. Bernetta Smouse Mrs. Elizabeth Smouse and children Thomas, Deirdre, and Robert Unichema North America



# Call for Nominations for the 2021–2022 Academic Year

Nominations for the Smouse Fellowship must be submitted using the official application. The application is available at aocs.org/awards.

Completed applications must be returned to AOCS by February 1, 2021.

### Do you know an exceptional graduate student?

The purpose of this graduate fellowship is to encourage and support outstanding research by recognizing a graduate student pursuing an advanced degree in a subject matter consistent with AOCS' areas of interest. Thomas Smouse was President of AOCS in 1983 and a noted industrial researcher into the flavor chemistry of fats and oils.

The award consists of a US \$10,000 Fellowship and up to US \$5,000 for travel and research related expenditures related to the student's graduate program.

### Eligibility

This award is open to graduate students with a strong aptitude for research,

who are scholastically outstanding, and have additional interests and involvement outside the academic discipline. *Student and major professor must be current members of AOCS to be eligible.* 

For more details on eligibility requirements, please visit accs.org/awards.



# MEMBER SPOTLIGHT

# **Meet Matt Miller**

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



Matt Miller and son George heading off into the New Zealand bush

# PROFESSIONAL

### What's a typical day like for you?

I spend the majority of my time now writing and spend less time in the lab. Reports, proposals, papers, annual reviews, Zane Grey fan fiction—only kidding. For me, communication and collaboration have become very important because I work in a very dynamic space in a small team. Cawthron is an accredited commercial testing facility and we also conduct novel fundamental research. I run large multiyear, multi-provider nutritional/biotechnology research projects at the same time as I develop validated analytical methods for industry.

### My favorite part of my job is...

Discovery. I love it when a plan comes together, as well as that moment, however short it is, that it all makes sense. We develop novel methods for new matrices and there is a lot of walking—well, stumbling around—in the dark. Then a light bulb goes off, everything is illuminated, and it all makes sense... well, until the next lot of data comes in.

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

At 10, I would have wanted to play Australian rules football for the Melbourne Demons or cricket for Australia. After I finished honors in chemistry (but not as a 10 year old—I'm no child prodigy), I travelled the world for four years, trying to figure out what I wanted to do...and, more importantly, what I didn't want to do. I have always liked problem solving, and science was a way I could do that.

# **Fast facts**

Name	Matt Miller
Joined AOCS	2004
Education	Ph.D. from University of Tasmania
Job title	Research scientist
Employer	Cawthron Institute, Nelson, New Zealand
Current AOCS involvement	President of the Australasian Section and vice chair of the Health and Nutrition Division

Why did you decide to do the work you are doing now? About eight years ago I was really struggling to figure out what I wanted to do. In New Zealand we have a unique marine bivalve (Greenshell Mussels or *Perna canaliculus*) that has shown some really interesting nutritional results around inflammation and mobility. I decided I wanted to explore this further and help shape the industry into higher-value spaces. It is very challenging work but recently we are gaining some real insight and hope that through the three human clinical trials we are conducting we can elucidate the efficacy of eating this novel omega-3/unique lipid source.

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

Throughout the work on my Ph.D. I was looking at alternative sources of lipid for salmon aquaculture. I looked at sources, including SDA-rich plant oils and DHA-rich microalgal oils. I further explored the biosynthetic capability of Atlantic salmon to produce long-chain omega-3 fatty acids. I hope this research will add to the greater scientific knowledge that will lead to more environmentally sound practices in aquaculture and more sustainable feeds while retaining salmon/cultured fish as a great source of EPA and DHA.

# PERSONAL

### How do you relax after a hard day of work?

In an ideal world I would love to spend my weekends up the rivers of New Zealand fly fishing or on my sea kayak targeting large snapper. I have a drum kit that has been dusted off during COVID lockdown. But mostly I chase my 4- and 6-year olds (Hazel and George) around while playing LEGOs or ponies.

# PATENTS

# Vegetable oil extraction improvement

Kellens, M, *et al.*, Desmet Ballestra Engineering, US20200512, May 12, 2020

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

# Composition containing highly unsaturated fatty acid or alkyl ester thereof and a method for producing the same

Doisaki, N., *et al.*, Nippon Suisan Kaisha, Ltd., US10696924, June 30, 2020

To provide a composition comprising highly enriched PUFA or its alkyl esters while containing fatty acid esters of 3-MCPD at adequately low concentrations and to provide an efficient method for producing the composition. A composition that contains fatty acids or fatty acid alkyl esters as its major component, the composition containing highly unsaturated fatty acid or alkyl ester thereof, wherein the proportion of the highly unsaturated fatty acid in the constituent fatty acids of the composition is 50 area % or more and wherein the concentration of 3-MCPD as found upon analyzing the composition by American Oil Chemists' Society official method Cd 29b-13 assay A is less than 1.80 ppm.

# Method of refining a grain oil composition to make one or more grain oil products, and related systems

Lamprecht, B., *et al.*, Poet Research, Inc., US10711221, July 14, 2020

The present disclosure is related to refining one or more grain oil composition streams (e.g., distillers corn oil or syrup) in a biorefinery to provide one or more refined grain oil products, where each grain oil product has targeted amounts of a free fatty acid component and the fatty acid alkyl ester component.

# Lipid extraction processes

Home, N, *et al.*, Aker Biomarine Antarctic AS, US10704011, July 7, 2020

The present invention provides improved processes for extracting and preparing lipids from biological sources for use in pharmaceuticals, nutraceuticals, and functional foods.



# Storage-stable enzyme granule

Hede, P., et al., Novozymes A/S, US10711262, July 14, 2020

The storage stability of enzyme granules for detergents can be improved by incorporating a polyamine having a molecule with at least 10% w/w of nitrogen wherein at least 50% of the N atoms are present as amines.

# Rubber composition for tire tread, method for producing the composition, and tire manufactured using the composition

Lee, S. K., et al., Hankook Tire Co., Ltd., US10711122, July 14, 2020

Provided are a rubber composition for a tire tread having improved braking performance, abrasion performance, and anti-aging performance, and a tire manufactured using this rubber composition. Disclosed is a rubber composition for a tire tread, the composition including 100 parts by weight of raw material rubber, 60 to 130 parts by weight of a reinforcing filler, and 10 to 60 parts by weight of modified natural oil, wherein the modified natural oil includes 50% to 90% by weight of a first natural oil; and 10% to 50% by weight of a second natural oil having a weight average molecular weight that is 2 to 10 times the weight average molecular weight of the first natural oil, with respect to the total weight of the modified natural oil.

# Increasing the bioavailability of flavan-3-ols by polyphenols

Actis Goretta, L., et al., Nestec S.A., US10722584, July 28, 2020

The present invention relates generally to the field of flavan-3ols. In particular, the present invention provides a way to increase the bioavailability of flavan-3-ols. Embodiments of the present invention relate to the use of at least one polyphenolic compound in a composition comprising at least one flavan-3-ol for increasing the bioavailability of said flavan-3-ol, wherein the at least one polyphenolic compound is selected from a group consisting of flavonols, flavones, isoflavones, flavanones, or combinations thereof.

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.



# The AOCS Foundation gratefully acknowledges the following Century Club members!



# \$1000+

**Brian Nutter** William Hendrix



**Douglas Bibus** Clifford Hall **Timothy Kemper** Phillip Kerr Pennv Kris-Etherton Hongkong Lin **Deland Myers** 



# \$200

**Gregory Hatfield Douglas Hayes** Steven Hill Michael Maibach Mark Matlock Thomas McKeon Oil-Dri Corp of America Anthony O'Lenick Perry Videx LLC Anders Thomsen Jill Winkler-Moser Masaki Tsumadori



CENTURY

\$100

AAK Alfa Laval Inc Rotimi Aluko Yasuhiro Ando

Archer Daniels Midland Co **Bioriginal Food &** Science Corp Michael Boyer Morgan Brown Dario Cabezas Caldic Canada Inc Mike Clayton Colonial Chemical Inc Complejo Agroindustrial Angostura SA Croll Reynolds Co Inc Lorin De Bonte **DuPont Nutrition &** Health

Yasushi Endo Evonik Corp - Household Ċare Fuji Vegetable Oil Inc Lucky Inturrisi ITS Testing Services (M) Sdn Bhd David Johnson Elaine Krul Kuala Lumpur Kepong Bhd Liberty Vegetable Oil Co Keshun Liu Fung Sieng Lu Edmund Lusas

Dave McCall **Robert Moreau** Kuwano Noriyuki Mark Nugent **Renato Ramos** James Smith **Dennis Strayer** Thanakorn Vegetable Oil Prods Co Ltd Xin (Cindy) Tian Bhima Vijayendran Wilmar International Ltd Michael Woolsey Xiaoqing Yang Jennifer Young

Donations made between January 1, 2020, and November 3, 2020.

For the complete list of Century Club members, visit aocsfoundation.org/CenturyClub.

# Your support enables the AOCS Foundation to fund the development of new products and services for AOCS.



www.aocsfoundation.org | patrick.donnelly@aocs.org |

+1217 - 693 - 4838

# **AOCS JOURNALS**

# The best way to improve peer review: Be a reviewer.

In November 2018, *The New York Times* ran an article with the title, "Peer review: the worst way to judge research, except for all the others."

The process by which scientific results are vetted by academic peers has its flaws, but it remains the central pillar of trust for researchers. 84% of researchers believe that without peer review there would be no control in scientific communication, and 9 out of 10 researchers feel that peer review improves the quality of their published paper.

Efforts to make peer review more open and transparent; less subjective; easier and faster; and more reliable, consistent, and inclusive are ongoing. But, ultimately, the success of those efforts rests on increasing the number and diversity of qualified reviewers. As a result, more students and professors are volunteering as reviewers.

Getting involved in the peer review process can be a highly rewarding experience that can also improve your own research and help to further your career, but how do you know if you are qualified to review an article?

# **WHO CAN REVIEW**

Anyone who is an expert in the article's research field can serve as a reviewer. All a potential reviewer needs is enough knowledge in a specific area to evaluate the manuscript and provide constructive criticism to editors and authors. A good reviewer can be at any stage of his or her career.

Occasionally, an editor might ask a reviewer to look at a specific aspect of an article, even if the overall topic is outside of the reviewer's specialist knowledge. In that case, the editor should outline just what it is they would like you to assess.

# AOCS MEETING WATCH

May 3–14, 2021. AOCS Annual Meeting & Expo, annualmeeting.aocs.org.

**October 5–7, 2021.** Plant Protein Science and Technology Forum, Millennium Knickerbocker, Chicago, Illinois, USA.

May 1–4, 2022. AOCS Annual Meeting & Expo, Hyatt Regency Atlanta, Atlanta, Georgia, USA.

**April 30–May 3, 2023.** AOCS Annual Meeting & Expo, Colorado Convention Center, Denver, Colorado, USA.

For in-depth details on these and other upcoming meetings, visit http://aocs.org/meetings or contact the AOCS Meetings Department (email: meetings@aocs.org; phone: +1 217-693-4831).

# HOW DO YOU BECOME A REVIEWER?

There is no one way to become a reviewer, but there are some common routes. These include:

- asking a colleague who already reviews for a journal to recommend you;
- networking with editors at professional conferences;
- becoming a member of a learned society and then networking with other members in your area;
- contacting journals directly to inquire if they are seeking new reviewers;
- seeking mentorship from senior colleagues; and
- working for senior researchers who may then delegate peer review duties to you.

# **TIPS FOR NEW REVIEWERS**

When you are invited to review an article, the editor should send you a copy of the abstract to help you decide whether you wish to do the review. Try to respond to invitations promptly; it will prevent delays. It is also important at this stage to declare any potential Conflict of Interest.

If you are new to peer review and feeling unsure of yourself, don't worry. Confidence will come with experience—but how do you get the right kind of experience?

One way is to seek guidance from more experienced colleagues. Gaining the support of an experienced mentor and familiarizing yourself with the process of peer review should help you build your confidence and track record.

You can also find many helpful tips and recommended best practices on the internet. Wiley, the publisher of AOCS Journals, offers a step-by-step guide to reviewing a manuscript at https://tinyurl.com/qkffh6r, and the Wiley Author Learning and Training Channel features expert advice on a broad range of publishing topics at https://tinyurl.com/yxoddpgd.

If you're just starting out as a reviewer, don't be deterred. Journal editors are often looking to expand their pool of reviewers, which means there will be a demand for your particular area of expertise.

Finally, keep in mind that reviewing is more than rejecting or accepting a manuscript. Good reviews should be a dialogue between author and reviewer that improves the integrity and scientific merit of the paper. Toward that end, reviewers may suggest additional experiments that authors have overlooked, or challenge the interpretation of data. That takes time—sometimes as much as a full day per paper. Shortcuts can result in the loss of scientific rigor and create cracks in the pillar of trust that authors and other scientists depend upon, causing reviewers to miss one of the most satisfying aspects of reviewing.





Make your plans to attend the 2021 AOCS Annual Meeting & Expo, a preeminent scientific event and an indispensable resource for comprehensive technical content and up-to-date industry information. Join AOCS members and colleagues from around the globe this May to discover the latest research and innovations in oils, fats, proteins, surfactants and bio-sourced materials.

Learn what is new and what is next for processing, foods, personal and home care, and sustainable materials industries through over 190 hours of live oral presentations.

FOR A SINGLE INTEREST AREA US \$225 FOR THE ENTIRE MEETING

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

BIO Biotechnology LOQ Lipid Oxidation and Quality IOP Industrial Oil Products PCP Protein and Co-Products

S&D Surfactants and Detergents

# **Review Articles**

# The potential of genome editing for improving seed oil content and fatty acid composition in oilseed crops

Subedi, U., *et al., Lipids* 55: 495–512, 2020, https://doi.org/10.1002/lipd.12249.

A continuous rise in demand for vegetable oils, which comprise mainly the storage lipid triacylglycerol, is fueling a surge in research efforts to increase seed oil content and improve fatty acid composition in oilseed crops. Progress in this area has been achieved using both conventional breeding and transgenic approaches to date. However, further advancements using traditional breeding methods will be complicated by the polyploid nature of many oilseed crops and associated time constraints, while public perception and the prohibitive cost of regulatory processes hinders the commercialization of transgenic oilseed crops. As such, genome editing using CRISPR/Cas is emerging as a breakthrough breeding tool that could provide a platform to keep pace with escalating demand while potentially minimizing regulatory burden. In this review, we discuss the technology itself and progress that has been made thus far with respect to its use in oilseed crops to improve seed oil content and quality. Furthermore, we examine a number of genes that may provide ideal targets for genome editing in this context, 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.

# LOQ HEN Polyphenols and their applications: an approach in food chemistry and innovation potential

Fernandes de Araújo, F., *et al., Food Chem.* 338: 127535, 2021, https://doi.org/10.1016/j.foodchem.2020.127535.

Polyphenols are compounds naturally present in fruits and vegetables that are gaining more and more attention due to their therapeutic effects and their potential technological applications. In this review, we intend to demonstrate the importance of some phenolic compounds, addressing their biological effects and potential for applications in various industrial fields. The intake of these compounds in appropriate concentrations can present promising effects in the prevention of diseases such as diabetes, obesity, Parkinson's, Alzheimer's, and others. They can also be used to improve the physicochemical properties of starch, in the preservation of foods, as natural dyes, prebiotic ingredients, hydrogels, and nanocomplexes. In addition, these compounds have potential for innovation in the most diverse technological fields, including organic fine chemistry, basic materials chemistry, pharmaceuticals, food chemistry, chemical engineering, among others.

# EAT LOO Microencapsulation of natural dyes with biopolymers for application in food: a review

Ribeiro, J.S. and C.M.Veloso, *Food Hydrocoll*. 112: 106374, 2021, https://doi.org/10.1016/j.foodhyd.2020.106374.

Microencapsulation is a technique increasingly studied in food science aimed to increase stability and control the release of aromas, dyes, antioxidants, nutrients, enzymes, preservatives, and microorganisms. The most commonly used wall materials include hydrocolloids, proteins, starches, dextrins, lipids, various emulsifiers, and fibers, alone or associated with other compounds. Encapsulation techniques are diverse, and the choice of the most appropriate technique depends on the material to be encapsulated, the purpose of use, and the availability of equipment. In the case of the core material, several studies have focused on the encapsulation of natural dyes, due to the increasing market demand for this type of food additive. This review focuses on the microencapsulation of natural food dyes, presenting the main encapsulated compounds and wall materials, as well as the techniques used for this purpose.

# **Original Articles**

# ANA EAT Novel cocoa butter equivalent from microalgal butters

Ghazani, S.M. and A.G. Marangoni, *J. Am. Oil Chem.' Soc.* 97: 1095–1104, 2020, https://doi.org/10.1002/aocs.12408.

An acetone solvent fractionation procedure was used to obtain two algal butter fractions, an algal stearin containing high amounts of 1,3-stearoyl-2-oleoyl-glycerol (SOS), and an algal mid-fraction containing high amounts of 1,3-dipalmitoyl-2-oleoyl-glyc"The AOCS Annual Meeting & Expo's online format allowed me to access more presentations than ever before. I can now broaden my knowledge by viewing a wide array of presentations on-demand on my own schedule."

**Tong (Toni) Wang** Professor, University of Tennessee AOCS Member since 1995



Our virtual 2020 event attracted researchers and innovators from 80 countries and almost double the attendance of past in-person meetings. We anticipate an even larger and more inclusive event in 2021 with 80+ interactive live broadcast sessions and 600+ technical presentations.

# annualmeeting.aocs.org

# Make the most of the meeting with AOCS membership

Membership means optimized savings and an enriched experience with member only registration discounts and extended on-demand access to presentations through April 2022.



erol (POP), and 1-palmitoyl-2-oleoyl, 3-stearoyl-glycerol (POS). Algal stearin and algal mid-fraction were blended (1:9 w/w) to yield a potential cocoa butter equivalent (CBE). The amount of POP, POS, and SOS, in algal CBE was 15.8%, 32.0%, and 24.6%, respectively, compared to 15.4%, 38.8%, and 27.7% in Cocoa butter (CB). However, a higher amount of POO and SOO in the algal CBE caused a lower solid fat content at all temperatures compared to CB. This study demonstrates the potential for producing a new algal CBE using solvent fractionation and blending techniques that can be used in chocolate and other confectionery products.

# ANA EAT PRO Potential effect of cavitation on the physical properties of interesterified soybean oil using high-intensity ultrasound: a long-term storage study

Lee, J., et al., J. Am. Oil Chem.' Soc. 97: 1105–1117, 2020, https://doi.org/10.1002/aocs.12414.

The objective of this research was to evaluate if cavitation events generated during sonication (20kHz, 216 micrometer amplitude, 10 s) are responsible for changes in physical properties of a fat with low levels of saturated fatty acids, and if these changes are maintained during storage. The fat was crystallized at 24 and 34°C and stored at 25°C for up to 24 weeks. An increase in solid fat content and melting enthalpy was observed for sonicated samples crystallized at 34°C, and an increase in elasticity was observed for sonicated samples crystallized at  $24^{\circ}C$  (*P* < 0.05). Hardness increased in sonicated samples crystallized at 24 and  $34^{\circ}C$  (*P* < 0.05) after 60 min of crystallization and after 24 weeks of storage. Elasticity of non-sonicated samples crystallized at 24°C decreased (P < 0.05) after storage at 25°C for 48 h, while it remained constant in sonicated samples. Sonicated samples had more and smaller crystals compared to the non-sonicated ones. No significant change was observed in physical properties of sonicated samples crystallized at 24°C and 34°C during the 24 weeks of storage. Sonication at 24°C was less efficient at changing the physical properties of the fat compared to 34°C; however, the number of subharmonic components generated during sonication at these two temperatures was not affected by crystallization temperature. These results suggest that changes in physical properties are associated with secondary effects of sonication, such as bubble streamers, rather than changes in cluster dynamics.

# ANA **IOP** The friction and wear behaviors of vegetable oil-based waxes, natural beeswax, and petroleum paraffin wax

Tao Fei, *et al., J. Am. Oil Chem.' Soc.* 97: Pages 1141–1150, 2020, https://doi.org/10.1002/aocs.12398.

Quantitative analyses on the coefficient of friction of common coating waxes are necessary and essential for designing systems for coating, conveying, packaging operations, transporting, and storing of papers and paperboards, while analyses on wear behavior can be helpful for predicting performance durability of the coating surface. In this study, we investigated the friction and wear behaviors of six waxes, including four commercial waxes and two soybean oil-based wax developed in our lab, for bulk corrugated coating. The effect of normal load, sliding velocity, and environmental temperature was evaluated. The friction coefficient of different waxes varies with sliding conditions. Higher normal load, sliding velocity, and environmental temperature resulted in significantly greater wear loss. Crystalline morphology and crystallinity of waxes were affected by the environmental temperature, and they correlate to the variations in friction coefficient and wear loss of these materials.

# BO Generation and characterization of a soybean line with a *Vernonia* galamensis Diacylglycerol acyltransferase-1 gene and a myo-inositol 1-phosphate synthase knockout mutation

AL-Amery, M., *et al.*, *Lipids* 55: 469–477, 2020, https://doi.org/10.1002/lipd.12253.

Soybean (Glycine max) meal is an important protein source. Soybean meal with lower phytate and oligosaccharides improves meal quality. A single recessive mutation in soybean myo-inositol 1-phosphate synthase (Gm-lpa-TW75-1) confers a seed phenotype with low phytate and increased inorganic phosphate. The mutant was crossed with high oil lines expressing a diacylglycerol acyltransferase1 (DGAT) gene from Vernonia galamensis (VgD). Gmlpa-TW75-1 X VgD, designated GV, has 21%, and 22% oil and 41% and 43% protein from field and greenhouse seed production, respectively. No significant differences were found in mineral concentrations, except for Fe which was 229 microgram/g dry mass for GV followed by 174.3 for VgD and 162 for Gm-lpa-TW75-1. Phosphate (Pi) is higher in Gm-lpa-TW75-1 as expected at 5 mg/g, followed by GV at 1.6 mg/g whereas Jack, VgD, and Taiwan75 have about 0.3 mg/g. The Gm-lpa-TW75-1 line has the lowest phytate concentration at 1.4 mg/g followed by GV with 1.8 mg/g compared to Taiwan75, VgD, and Jack with 2.5 mg/g. This work describes a high oil and protein soybean line, GV, with increased Pi and lower phytate which will increase the nutritional value for human and animal feed.

# **EAT** LOO Ferulic acid production from brewery spent grains, an agro-industrial waste

Sibhatu, H.K., *et al*, *LWT*: 135: 110009, 2021, https://doi.org/10.1016/j.lwt.2020.110009.

This research looks at the use of brewery spent grains (BSG) for the extraction of a natural antioxidant, ferulic acid. Brewery spent grain was initially treated with dilute sulfuric acid, then subjected to alkaline hydrolysis using NaOH, prior to ferulic acid extraction and purification. X-ray diffraction analysis of the raw and hydrolyzed BSG showed evidence for a possible release of phenolic extractives.

# **LOQ** HEN Prebiotic effects of olive pomace powders in the gut: *in vitro* evaluation of the inhibition of adhesion of pathogens, prebiotic and antioxidant effects

Ribeiro, T.B., *et al., Food Hydrocoll. 112*: 106312, 2021, https://doi.org/10.1016/j.foodhyd.2020.106312.

Olive pomace is a biowaste rich in polyphenols and insoluble dietary fiber, with high potential to develop new value chains that support a sustainable and circular bioeconomy. Regarding gut health, olive pomace phenolics and insoluble dietary fiber (after possible fermentation) could act as antioxidants, antimicrobial, and prebiotic agents. Two powders from olive pomace: liquid-enriched powder (LOPP)—a source of phenolics—and pulp-enriched powder (POPP)—a major source of insoluble dietary fiber. LOPP and POPP were subjected to *in vitro*-simulated gastrointestinal digestion followed by *in vitro* faecal fermentation. The undigested fraction retained in the colon was analyzed for potential antioxidant, antimicrobial, and prebiotic effects. LOPP and POPP did not impact the gut microbiota diversity negatively, showing a similar ratio of Firmicutes/Bacteroidetes compared to a positive control (FOS). LOPP exhibited a positive (similar to FOS) effect on the *Prevotella* spp./Bacteroides spp. ratio. Both powders promoted more production of short-chain fatty acids (mainly acetate > butyrate > propionate) than FOS and demonstrated significant total phenolic content and oxygen radical absorbance capacity during faecal fermentation up to 48 h. The powders also showed mucin-adhesion inhibition ability against pathogens, principally POPP against *Bacillus cereus* (22.03 ± 2.45%) and *Listeria monocytogenes* (20.01 ± 1.93%). These results demonstrate that olive pomace powders have prebiotic effects on microbiota, including the stimulation of short-chain fatty acids production and antioxidant and antimicrobial activity which could potentially improve human gut health.

# EAT LOO Composition of bound polyphenols from carrot dietary fiber and its *in vivo* and *in vitro* antioxidant activity

Dong, R., et al., Food Chem. 339: 127879, 2021, https://doi.org/10.1016/j.foodchem.2020.127879.

Qualitative analysis of bound polyphenols from carrot dietary fiber (CDF-PP) was performed by ultra-performance liquid chromatography equipped with an electrospray ionization and quadrupole time-of-flight mass spectrometry (UPLC-ESI-QTOF-MS/MS).



# **Biobased Surfactants** Synthesis, Properties, and Applications Second Edition

Edited by Douglas G. Hayes, Daniel K. Solaiman and Richard D. Ashby May 2019 | 512 pages | ISBN: 9780128127056 Available in softcover and eBook

Biobased Surfactants: Synthesis, Properties, and Applications, Second Edition, covers biosurfactant synthesis and applications and demonstrates how to reduce manufacturing and purification costs, impurities and by-products. Fully updated, this book covers surfactants in biomedical applications, detergents, personal care, food, pharmaceuticals, cosmetics and nanotechnology. It reflects on the latest developments in biobased surfactant science and provides case scenarios to guide readers in efficient and effective biobased surfactant application, along with strategies for research into new applications. This book is written from a biorefinery-based perspective by an international team of experts and acts as a key text for researchers and practitioners involved in the synthesis, utilization and development of biobased surfactants.

### **Key Features:**

- Describes new and emerging biobased surfactants and their synthesis and development
- Showcases an interdisciplinary approach to the topic, featuring applications to chemistry, biotechnology, biomedicine and other areas
- Presents the entire lifecycle of biobased surfactants in detail

Available for purchase at store.elsevier.com/aocs \*AOCS Members use code AOCS30 at checkout to receive 30% discount and free shipping worldwide. Eleven organic acids, nine hydroxybenzoic acids and derivatives, six hydroxycinnamic acids and derivatives, four phenolic alcohols and derivatives, three flavonoids and derivatives, seven esters and derivatives, and two other compounds were detected by matching their retention times, secondary mass spectrometry fragment information with authentic standards, or literature data. Furthermore, *in vitro* antioxidant activity was determined by different kinds of assays, including DPPH, ORAC, and PSC, demonstrating that CDF-PP could scavenge radicals in a dose-dependent manner. Moreover, CDF-PP exhibited significantly reactive oxygen species (ROS) scavenging activity in living *Caenorhabditis elegans*. To our knowledge, this is the first comprehensive research to investigate composition and *in vitro/in vivo* antioxidant activity of bound polyphenols in CDF, which implies that CDF-PP could be a promising source of antioxidants.

# **PRO HEN EAT** Synthesis of mediumand long-chain triacylglycerols by enzymatic acidolysis of algal oil and lauric acid

Li, Y., *et al.*, *LWT* 136, Part 1, January 2021, https://doi.org/10.1016/j.lwt.2020.110309.

To obtain medium- and long-chain triacylglycerols (MLCTs) with high levels of lauric acid (LA) and docosahexaenoic acid (DHA), an acidolysis reaction was catalyzed by lipase in a sol-

![](_page_47_Picture_5.jpeg)

vent-free system to incorporate LA into algal oil (rich in DHA). Three regiospecific immobilized lipases were compared for their efficiency in the synthesis of MLCTs-riched structured lipids (SLs). The results showed that Lipozyme AOAB8 (from *Aspergillus oryzae*) displayed the top performance for LA incorporation of MLCTs-riched SLs. The optimum conditions of acidolysis reaction were: reaction temperature 65°C, substrate mole ratio 1:8 (algal oil to LA), enzyme load 12 g/100 g total substrates, and reaction time of 2.5 h. Under these conditions, LA and DHA of the MLCTsriched SLs production were 30.91% and 44.68%, respectively, and Lipozyme AOAB8 was reusable for at least 9 times. Melting and crystallization profiles revealed that the MLCTs-riched SLs production possessed preferable physical properties over the original algal oil. The MLCTs-riched SLs can be applied in human milk fat substitutes and dietary supplements as functional lipids.

# Extraction separation of rhamnolipids by n-hexane via forming reverse micelles

Chaojun Zhou, et al., J. Surfactants Deterg. 23: 883–889, 2020, https://doi.org/10.1002/jsde.12409.

Rhamnolipids (RL) have been regarded to be insoluble in nhexane. Unexpectedly, we noticed that RL could be extracted together with vegetable oil by n-hexane when analyzing oil content of fermentation broth. This paradoxical phenomenon was assumed to be due to the formation of reverse micelles. As found in this paper, the micelle size as well as conductivity increased due to water solubilization, illustrating the formation of reverse micelles of RL in n-hexane. In this reverse micellar system, the maximal water solubility was detected to be 6.26 mol H2O/mol RL while the reverse critical micelle concentration of RL was around 3% (w/w). Hence, it seems that the presence of water could increase the dissolution of RL in n-hexane via forming the reverse micelles of RL/n-hexane/water. Lastly, the formation of reverse micelles was further applied for extraction of RL by n-hexane. Using this method, over 99.0% of RL was extracted under the appropriate pH of 4.5. This extraction separation using n-hexane instead of chloroform proposed a much cleaner and safer strategy in RL manufacture. Moreover, the capability of RL in forming reverse micelle system could benefit future applications, such as those that improve enzymatic reactions, the stability of nanoparticles, environmental treatment, and protein folding.

# SED Application of pH-responsive viscoelastic surfactant as recyclable fluid at high temperature

Xuepeng Wu, et al., J. Surfactants Deterg. 23: 863–872, 2020, https://doi.org/10.1002/jsde.12418.

A pH-responsive amphiphilic surfactant stearic amide 3-(N,N-dimethylamino)propylamide (SAA) was synthesized and served as a thickener in aqueous solution to construct a switchable viscoelastic surfactant fluid (VES fluid). The structure of SAA was studied by 1H NMR, and the viscoelastic behavior of VES fluid was

![](_page_48_Picture_0.jpeg)

# **OPPORTUNITIES**

2020 HAS CHANGED OUR WORLD. AOCS IS HERE TO SUPPORT YOU WITH THE RESOURCES YOU NEED TO TURN YOUR CHALLENGES INTO OPPORTUNITIES.

# What will you do to excel this year?

- 📀 Attend an AOCS Webinar
- Continue the conversation with More Informed
- 📀 Network at a Midweek Mixer
- Read the latest research in AOCS journals
- Register for the 2021 AOCS Annual Meeting & Expo

AOCS.ORG/MEMBERSHIP

![](_page_48_Picture_10.jpeg)

studied in detail by rheological measurements. The viscosity of this VES fluid can be switched reversibly from low to high immediately by adjusting system pH value. Even at high shear rate (170s–1) and high temperature (90°C), excellent viscoelastic behavior of this VES fluid can be observed, which is a key performance for fracturing applications. Meanwhile, the recycled VES fluid can still maintain good pH-responsive behavior even after more than three cycles. The unique performances of this VES fluid not only enhanced our understanding of the transformation of wormlike micelles at high temperature, but also enriched a large potential of VES fracturing fluid in the development of oil and gas reservoirs.

# SND EAT Benzalkonium chloride alters phenotypic and genotypic antibiotic resistance profiles in a source water used for drinking water treatment

Harrison, K.R., et al., Environ. Pollut. 257: 113472, 2020, https://doi.org/10.1016/j.envpol.2019.113472.

Antibiotic resistance is a major public health concern. Triclosan is an antimicrobial compound with direct links to antibiotic resistance that was widely used in soaps in the United States until its ban by the US Food and Drug Administration. Benzalkonium chloride (BAC), a quaternary ammonium compound, has widely replaced triclosan in soaps marketed as an antibacterial. BAC has been detected in surface waters and its presence will likely increase following increased use in soap products. The objective of this study was to determine the effect of BAC on relative abundance of antibiotic resistance in a bacterial community from a surface water used as a source for drinking water treatment. Bench-scale microcosm experiments were conducted with microbial communities amended with BAC at concentrations ranging from 0.1 microgram L-1 to 500 microgram L-1. Phenotypic antibiotic resistance was quantified by culturing bacteria in the presence of different antibiotics, and genotypic resistance was determined using qPCR to quantify antibiotic resistance genes (ARGs). BAC at concentrations ranging from 0.1 microgram L-1 to 500 microgram L-1 was found to positively select for bacteria resistant to ciprofloxacin and sulfamethoxazole, and negatively select against bacteria with resistance to six other antibiotics. Exposure to BAC for 14 days increased the relative abundance of sul1 and blaTEM.

This study re-highlights the importance of employing both culture and non-culture-based techniques to identify selection for antibiotic resistance. The widespread use of BAC will likely impact antibiotic resistance profiles of bacteria in the environment, including in source waters used for drinking water, wastewater treatment plants, and natural waterways.

# SED Lipidomic analysis of epithelial corneal cells following hyperosmolarity and benzalkonium chloride exposure: new insights in dry eye disease

Magny, R., et al, Biochimica et Biophysica Acta–Molecular and Cell Biology of Lipids 1865: 158728, September 2020, https://doi.org/10.1016/j.bbalip.2020.158728.

Dry eye disease (DED) is a multifactorial chronic inflammatory disease of the ocular surface characterized by tear film instability, hyperosmolarity, cell damage and inflammation. Hyperosmolarity is strongly established as the core mechanism of the DED. Benzalkonium chloride (BAK)—a quaternary ammonium salt commonly used in eye drops for its microbicidal properties—is well known to favor the onset of DED. Currently, little data are available regarding lipid metabolism alteration in ocular surface epithelial cells in the course of DED. Our aim was to explore the effects of benzalkonium chloride or hyperosmolarity exposure on the human corneal epithelial (HCE) cell lipidome, two different conditions used as in vitro models of DED. For this purpose, we performed a lipidomic analysis using UPLC-HRMS-ESI+/-. Our results demonstrated that BAK or hyperosmolarity induced important modifications in HCE lipidome including major changes in sphingolipids, glycerolipids, and glycerophospholipids. For both exposures, an increase in ceramide was especially exhibited. Hyperosmolarity specifically induced triglyceride accumulation resulting in lipid droplet formation. Conversely, BAK induced an increase in lysophospholipids and a decrease in phospholipids. This lipidomic study highlights the lipid changes involved in inflammatory responses following BAK or hyperosmolarity exposures. Thereby, lipid research appears of great interest, as it could lead to the discovery of new biomarkers and therapeutic targets for the diagnosis and treatment of dry eye disease.

# Want to contribute to Extracts & Distillates?

Want an incentive to keep up with the latest research in your interest area? *Inform* magazine's Extracts & Distillates column is seeking professionals in surfactants and detergents, processing, health and nutrition, edible applications, or proteins and coproducts to select and contribute abstracts of interest from journals in their respective areas. Contributors to the column provide 3–5 abstracts of interest per issue on a regular schedule. If you are interested in contributing to Extracts & Distillates, please contact Kathy Heine at kheine@aocs.org.

# Full-scale aftermarket support.

![](_page_50_Picture_1.jpeg)

# Only Crown delivers problem-solving expertise, superior parts and on-site support.

Optimize your operation with Crown's Aftermarket Parts and Field Services. Available to all Crown customers, our comprehensive program includes field engineering support in 13 languages to assist with install, troubleshoot issues, train staff, realize cost savings and more. To minimize downtime, our superior replacement parts are available for immediate shipment anywhere in the world. We also work with you to refurbish or replace equipment nearing the end of its lifecycle. Our aftermarket commitment is another way Crown lowers total cost of ownership to customers around the globe.

Get the aftermarket attention you deserve. Choose Crown for the long run.

![](_page_50_Picture_5.jpeg)

Field Engineering | Replacement Parts | Replace or Refurbish Contact Crown today 1-651-639-8900 or visit our website at www.crowniron.com

# Consistency is key.

You can rely on Oil-Dri. With unrivaled customer service, dependable supply, and highly experienced technical service experts, our world-class team is tough to beat. We stay committed to enhancing your refinery processes each and every step of the way.

Our globally trusted bleaching earths for edible oils — Pure-Flo®, Supreme<sup>™</sup>, and Perform<sup>®</sup>— are the industry's reliable choice for performance in color reduction, metals removal, and filtration. Feel at ease with products you can trust.

> **OID-Dri** FLUIDS PURIFICATION

Choose reliability.

Visit **oildri.com/purify** to learn more about our quality products and services.

# Bleaching Earths

© 2020 Oil-Dri Corporation of America. All rights reserved.