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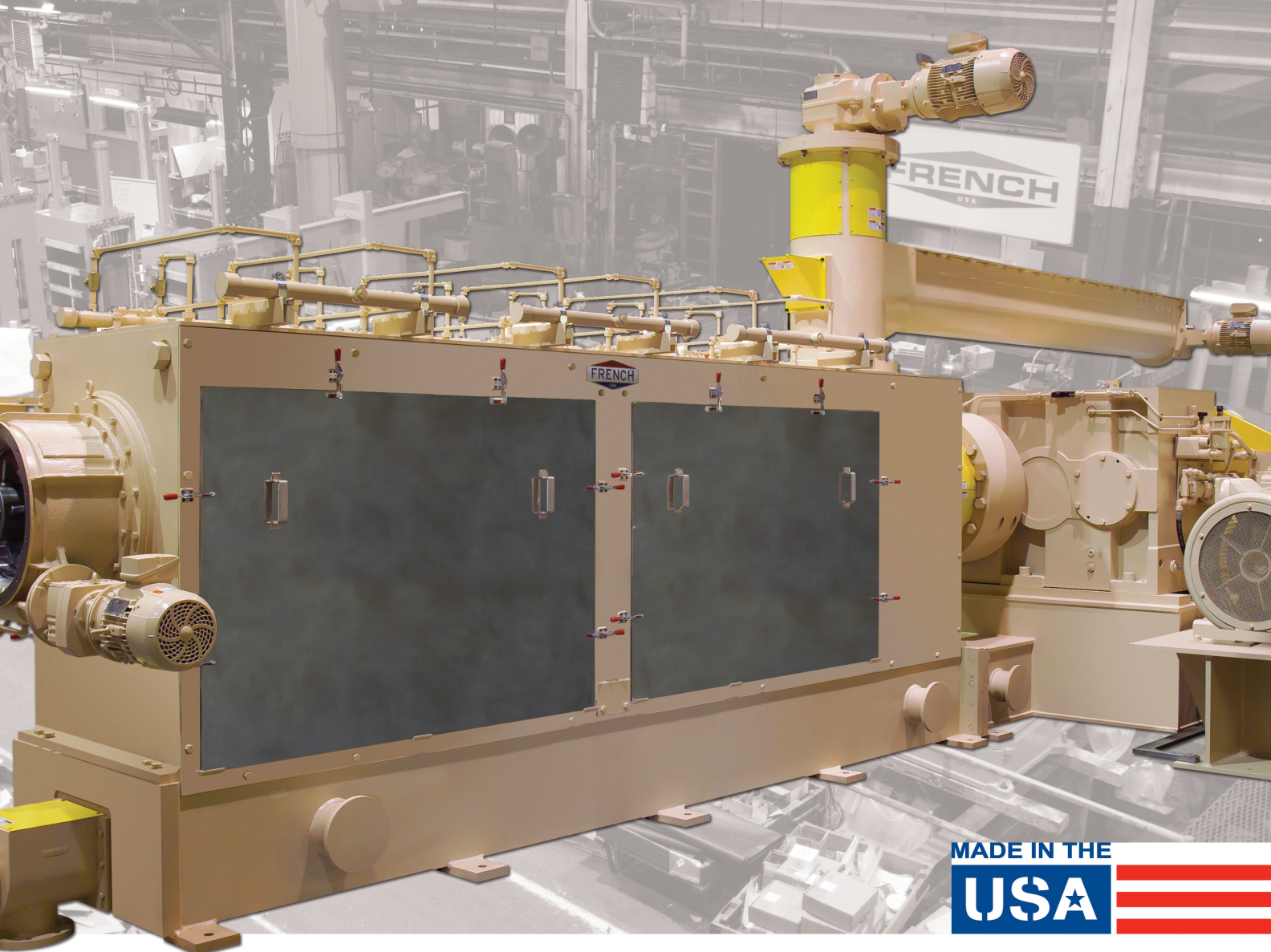


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July/August 2022

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# CONTENTS

## 6 Fish heads, fish heads: For biosurfactant production

When fed the proper nutrient-rich medium, microbes can convert long-chain carbon compounds into a chemical consortium that includes biosurfactants. Researchers are exploring the potential for fish peptones to provide the nourishment needed to achieve industrial scale fermentation.





## 12 Switching to sustainable surfactants

As the personal care industry races to drop petroleum-based ingredients, chemical companies are rolling out a dizzying array of biobased surfactants. Choosing from among them is not easy.

## 20 Testing enzymes: When everything is good, nothing is good

To assess the effect of enzymes on detergent performance, researchers must be conscious of their testing parameters. This story describes the criteria for useful stains that help laundry detergent formulators differentiate between enzyme activity.

## 26 Formulation engineering 2.0

Read about the theoretical process that combines a surfactant's middle phase microemulsion properties with its end-use conditions to predict formulation performance.

# DEPARTMENTS

5 Index to Advertisers  
25 AOCS Meeting Watch

Analysis/commentary  
30 Olio  
33 Regulatory Review  
39 Member Spotlight

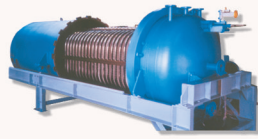
Publications and more  
40 Patents  
42 AOCS Journals  
44 Extracts & Distillates



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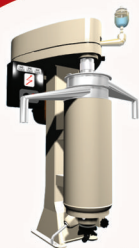
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## INDEX TO ADVERTISERS

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French Oil Mill Machinery Co. ....	1
Harburg-Freudenberger Maschinenbau GmbH .....	11
Koerting Hannover AG .....	9
Sharplex Filters (India) Pvt. Ltd. ....	4

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# Fish heads, fish heads: For biosurfactant production

Stacy Kish

- Biosurfactants are compounds produced by some bacteria that replicate the properties of petroleum-based surfactants.
- While many research studies have explored different bacteria grown on different medium, fish waste is proving to be a viable option to grow microbes for industrial-scale biosurfactant production.
- The Scottish start-up company, Eco Clean Team, has partnered with a researcher at the University of St. Andrews to develop a pilot project for scaling biosurfactant production with fish oil from the local aquaculture industry.
- While many hurdles remain, the biosurfactant industry and fish peptone industry are both on the upswing.

In 2010, Deep Water Horizon released over 130 million gallons (<https://tinyurl.com/3brsh5u8>) of oil, the equivalent to 200 Olympic-sized swimming pools, into the Gulf of Mexico. Twenty years earlier, the *Exxon Valdez* oil tanker ran aground in Alaska, releasing 11 million gallons of oil into Prince William Sound.

Oil spills harm marine plants and animals, and render seafood unsafe to eat. Environmental remediators apply different techniques, including skimming and burning, to remove oil pollution from the water's surface; however, synthetic dispersants created using petrochemicals, paradoxically, are the best means of clean-up. They reduce the opportunity for the contaminant to reach the shoreline by dispersing the oil and breaking it up into smaller droplets that are easier for microbes to consume. However, research shows synthetic surfactants are often toxic to marine organisms, changing their behavior, physiology, and reproduction patterns. These adverse effects raise concerns about which is worse for the environment—the dispersants applied to break up an oil slick or the oil itself.

Synthetic surfactants are a common class of molecules found in laundry and dishwashing detergents, among other household products. They are ubiquitous in our lives, yet they rarely garner the negative attention of an oil spill, because they are typically unseen. Although, in many instances synthetic surfactants are more pervasive and insidious to the environment.

“When people think about petroleum-based problems, they may think about the plastic trash pile floating in the middle of the Pacific Ocean, but other molecules, [like synthetic surfactants] that are not so readily evident, are also harming the planet,” said Rick Ashby, research scientist with the United States Department of Agriculture, Agriculture Research Service. “We are looking at biosurfactants as a means to avoid ecological contamination by petroleum-based products. The challenge is to make them at low-enough cost so they are competitive and compelling for industry to adopt.”



## MICROBES TO THE RESCUE

During different stages of growth, many microorganisms produce secondary metabolites in the form of surfactants. In nature, these microbes enzymatically convert the long-chain carbon compounds in oils into a chemical consortium that includes biosurfactants. The compounds vary in molecule length and structure, including glycolipids, lipopeptides, fatty acids, polysaccharide–protein complexes, peptides, phospholipids, and neutral lipids. As with any surfactant, biosurfactants have a polar, hydrophilic head group and a hydrophobic tail, that in this case is typically composed of one or more long chain fatty acids (<https://tinyurl.com/35tappu2>).

“The benefit of biosurfactants is that they are biodegradable and renewable,” said Ashby. “They will degrade into nontoxic materials in nature and can also be produced using renewable materials, like fats, oils, and plant biomass.”

When compared to synthetic counterparts, biosurfactants have a similar chemical composition while being just as effective at breaking up oil and creating lather for personal care products. They function over a wide-range of temperature, pH, and salinity conditions. They interact with a diverse variety of functional groups, which is beneficial when cleaning heavy minerals during environmental remediation. When produced within a renewable framework, biosurfactants also have a smaller carbon footprint than their synthetic equivalents.

Common strains of bacteria and fungi that produce biosurfactants include, but are not limited to, *Pseudomonas aeruginosa*, *Starmerella bombicola*, *Bacillus subtilis*, and *Ustilago maydis*. Scientists still speculate on the reason microbes produce biosurfactants. Incorporating hydrophobic carbon sources into their cells may somehow aid in microbe survival. Biosurfactants are known to protect against gram positive bacteria, like *Staphylococcus*, *Streptococcus*, *Listeria*, and *Bacillus*.

Previous studies have gathered these oil-munching organisms from contaminated water and soil environments. A 2021 study combined a lactonic sophorolipid biosurfactant, produced by *Starmerella bombicola*, with choline myristate and choline oleate ionic liquid surfactants to create a greener remediation product (<https://doi.org/10.1016/j.envpol.2021.117119>). The study examined two mixtures of different concentration of these compounds and found each to be thermodynamically stable and effective at dispersing crude oils. The maximum dispersion effectiveness was 78.23% for the 80:20 lactonic sophorolipid-choline myristate blend and 81.15% for the 70:30 lactonic sophorolipid-choline oleate blend. The high dispersion rates for these two mixtures are attributed to the hydrophobic tail and unsaturation of the additional surfactants, which improved the interactions between compounds in the mixture.

## NATURALLY LESS TOXIC

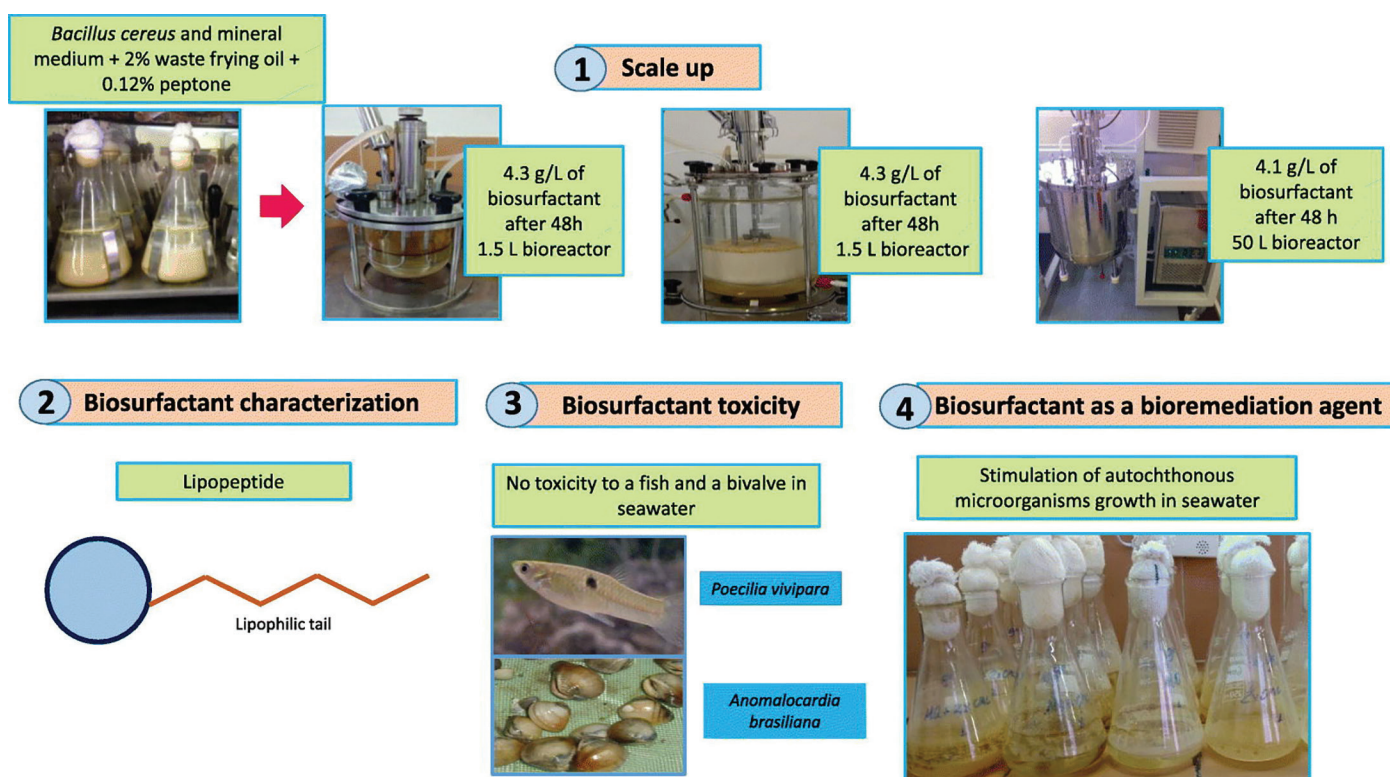
A series of toxicity studies compared the effect of various biosurfactants and synthetic surfactants on marine organisms. Indicator species provide a way to evaluate how the different compounds affect the organism’s physiological, nutritional, structural, and morphological characteristics.



A 2003 study in the journal *Marine Pollution Bulletin*, compared three synthetic surfactants (Corexit 9500, PES-61, and Triton X-100) with three biogenic surfactants (Bio-EM, Emulsan, and PES-51) to evaluate their toxicity on marine organisms and their effectiveness for dispersing oil ([https://doi.org/10.1016/S0025-326X\(03\)00238-8](https://doi.org/10.1016/S0025-326X(03)00238-8)). The study focused on two indicator species: *Mysidopsis bahia*, an epibenthic mysid shrimp living in estuarine waters spanning the Gulf of Mexico to Florida, and *Menidia beryllina*, an inland minnow living in waters from Mexico to Massachusetts. The study found that biosurfactants were intermediate to those of the synthetic surfactants in toxicity. *M. bahia* was generally more sensitive to the synthetic surfactants. The authors stress that application of the results requires balancing site-specific considerations of dilution, biodegradation, and exposure duration and depth when selecting a surfactant for remediation.

Almost two decades later, a 2020 study in the same journal obtained the biosurfactant, lipoprotein, from cultivated *Bacillus cereus* to evaluate the toxicity on two indicator species (<https://doi.org/10.1016/j.marpolbul.2020.111357>). The study also evaluated the hurdles to scaling-up production from the bench to industrial applications (fig.1). The toxicity portion of the study focused on the fish *Poecilia vivipara*, which is sensitive to potassium dichromate, sodium dodecyl sulfate, copper, and zinc. The second indicator species was the bivalve *Anomalocardia brasiliana*, which lives along the Brazilian coast. The results suggested that biosurfactants are safe and effective to remediate marine environments. The study also concluded that this biosurfactant was biocompatible for industrial-scale production, producing 4 g/L in only 48 h using a low-cost renewable raw material.





**FIG. 1: Studies demonstrate the potential for industrial-scale production biosurfactants and their biocompatibility in the remediation of marine environments polluted by oil spills.** Source: Duval, et al., *Marine Pollution Bulletin*, 157,111357, 2020.

## WITH AQUACULTURE WASTE, SCALE-UP MAY BE POSSIBLE

Despite these advantages, scaling the production of biosurfactants remains cost prohibitive, and hindered by the availability of suitable oils to grow the microbes. To compete in the global market, low-cost production is essential.

Using industrial wastes as feedstocks for biosurfactant production reduces the impact of pollution on the environment and offers a nutrient-rich medium for growing microbes. When processing fish, up to 60% of the total weight of the product ends-up as waste and a troubling source of local environmental pollution. Fish waste consists of a slurry of fish heads, fish skin, fish bones, meat, and viscera. Microbes can transform this rich source of suspended solids, organic carbon, and nitrogen into surfactants. Several studies have evaluated a series of microbes grown on fish waste, compared to other feedstocks, to produce biosurfactants for industrial-scale applications.

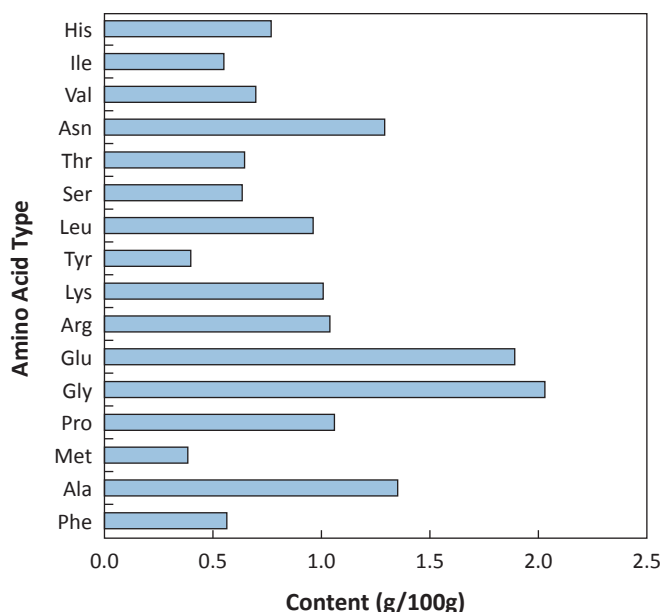
Some early work investigated the use of glycolipids as biopesticides to thwart fungal infections and kill mosquito larvae. These compounds are now being explored as biosurfactants. In 2011, researchers grew the phytopathogenic smut fungus, *Ustilago maydis*, to produce glycolipids using soy- and fish-based oils. The study found fish oil with the addition of lipase, an enzyme to break down fats, produced the highest yield (16.8 g/L of biomass), especially after seven days of incubation (<https://doi.org/10.5897/AJMR10.814>).

Another study examined two strains of *Pseudomonas aeruginosa* (H1 and SY1) that were collected from soils sur-

rounding olive oil and fish oil factories across Turkey (<http://doi.org/10.1590/S1517-838246320140727>). This microbe produces rhamnolipid biosurfactants. The study examined the potential of industrial production of rhamnolipid for bioremediation applications. The researchers grew the two environmental strains as well as a control strain (ATCC 9027) on two carbon feedstocks: kefir, a fermented milk drink, and fish meal. They found that the quality and quantity of biosurfactant produced was influenced by carbon and nitrogen substrates, the concentration of nutrients in the growth medium, culture conditions,—such as pH, temperature, and agitation—and culture dilution rate. The three microbial strains grown in the fish oil medium produced more rhamnolipid than those grown on the kefir medium. The results suggest that fish waste can be an important additive when exploring ways to ramp-up production of rhamnolipid biosurfactants.

In 2021, Memorial University of Newfoundland, Canada, civil engineering professor Bing Chen collaborated on a project with Chinese researchers to study generating fish peptones from the enzymatic hydrolysis of tuna fish waste (<https://doi.org/10.3390/catal11040456>). Fish peptones are the protein decomposition product obtained from different marine fish species. They are used as a nitrogen source for bacteria (fig. 2). Within their fish slurry, the researchers grew the bacterium, *Bacillus subtilis* (ATCC® 21332™). Then, they added a small amount of manganese to ensure the microbes produced sufficient lipopeptide biosurfactant.



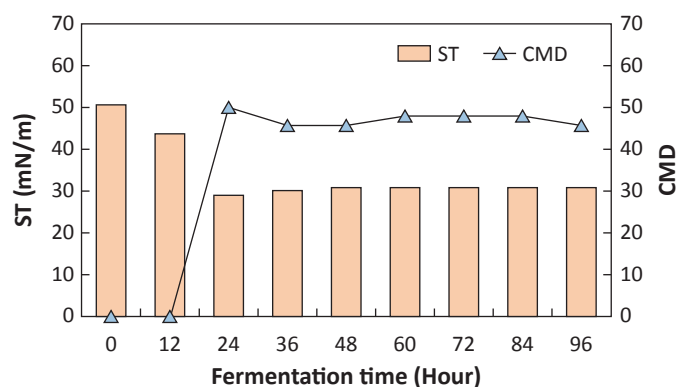


**FIG. 2:** Using the trichloroacetic acid (TCA) method, researchers achieved 44.2% enzymatic hydrolysis of fish waste. This is the amino acid composition in tuna waste-based peptone that resulted. Source: Hu, J. et al., *Catalysts*,11(4), 456, 2021.

The resulting biosurfactant reduced surface tension, as well as exhibited emulsifying, foaming, and biocatalytic activity. During the study, the highest biosurfactant production (274 mg/L) occurred between 24 to 36 hours of the fermentation process. The study authors are optimistic that it may be possible to scale-up surfactant production cost-effectively, noting that carbon and nitrogen supplements may be necessary to optimize the substrate (fig 3).

## REAL WORLD INDUSTRIAL-SCALE FERMENTATION

Alfredo Bonaccorso, senior research fellow at the Institute of Behavioral and Neural Sciences at the University of St.



**FIG. 3:** The results from batch-scale experiments showing biosurfactant production between 12-24 hours, as indicated by a lower surface tension (ST) and a higher critical micelle dilution (CMD).

Source: Hu, J. et al., *Catalysts*,11(4), 456, 2021.

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## Bitterness blocker

While cleaning products may be the focus of industry today, Ashby believes that biosurfactants could one day be found in a host of other products. He explained that some glycolipid biosurfactants (sophorolipids) have been shown to block the perception of bitterness (<https://doi.org/10.1002/jsde.12526>). This characteristic could garner interest from the food industry for hygiene products, like mouthwash and toothpaste. Pharmaceutical companies could also benefit from this characteristic to remove the bitter taste or aftertaste of some medications.

Andrews, has partnered with a Scottish start-up company, Eco Clean Team, to overcome the challenges of scaling biosurfactant production by tapping into a local resource—fish oil waste from Scottish aquaculture. According to an article in *Fish Focus*, the Scottish aquaculture sector processed 192,000 tons of Atlantic salmon in 2020. This market is anticipated to grow, with the potential to net up to \$4.45 billion by 2030 (<https://tinyurl.com/4znzmpxr>).

Bonaccorso and the Eco Clean Team have not revealed details, but say they aim to identify new approaches to scale production of biosurfactants using fish waste, which offers a near continuous and homogenous supply of oil that does not fluctuate in price like fossil fuels. It also provides added value to the aquaculture sector by creating jobs, and reducing and reusing the waste stream.

Mark Hamilton, co-founder and director at Eco Clean Team, told *Fish Focus*, “We have already proved the feasibility in a previous study and hope that, by the end of this project, we will find ourselves closer to full-scale commercialization and seeing the surfactant used in a range of industrial and selected household products.”

Bonaccorso says, he and his team have devised a more cost-effective, efficient, and eco-friendly process than traditional methods for producing biosurfactants. Bonaccorso anticipates it will be possible to create a pilot project in the next few years to begin the process of scaling production for industrial applications.



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## THE FUTURE OF THE BIOSURFACTANT MARKET

The ability to scale biosurfactants production will be critical for adoption by industry for a variety of applications. Fish waste may be the key to these future production goals. According to a March 2022 *Future Market Insights* article, the fish peptone market is on the rise (<https://tinyurl.com/2x3rphd7>). In the United States and Canada, fish peptones obtained from fish heads and fish livers are used as nitrogen and carbon sources for microbial growth and biosurfactant production. The new process to convert fish peptones has reduced the cost for lipo-peptide production on an industrial scale.

The rise in fish peptones parallels the rise in the biosurfactant market. A January 2022 *Kingpin Market Research* article reports the biosurfactant industry is anticipated to grow at a steady rate due to consumer demand for bio-based, eco-friendly biosurfactants (<https://tinyurl.com/2p87wre9>). In 2021, the global market for microbial biosurfactants was valued at \$19.41 million and is anticipated to reach \$38 million by 2027 (<https://tinyurl.com/fb9sfpgn>). The report focuses on regions of primary biosurfactant production, including China, the United States, Europe, and Japan where the market is dominated by five companies — Evonik Industries AG, BASF SE, Ecover, Jeneil, and Givaudan — that hold more than 80% of the market share (<https://tinyurl.com/2s3dbe7s>).

Evonik Industries AG has developed a line of products under the REWOFORM® trademark. These products leverage two biosurfactants: sophorolipids and rhamnolipids. Compared to traditional surfactants used in laundry formulations, rhamnolipids allow excellent cleaning using a lower surfactant concentration, thereby reducing the overall carbon footprint of the formulation.

“It is ok for a new, green product to be more expensive than the conventional product on the market if you do not need to use as much of it in a formulated product,” said Derek Dagostino, director Global Marketing Cleaning Solutions at Evonik Corporation. “It really depends on the market application and the formulation for customers to better understand the value that can be created for them by biosurfactants.”

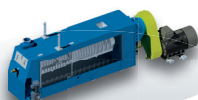
According to Dagostino, the rising consumer demand for green products motivates companies to formulate products that are safe, effective, and have lower volatile organic compounds, common to petroleum-based products. While the Evonik Industries AG currently uses a vegetable feedstock in the production of their two biosurfactant lines, they are constantly innovating and evaluating new feedstocks. Maybe, their next innovation will be fish waste.

*Stacy Kish is a freelance science writer. She has worked for 15 years to bring engaging stories about an array of science topics to a general audience. She can be contacted at [earthspin.science@gmail.com](mailto:earthspin.science@gmail.com).*

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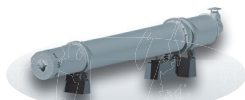
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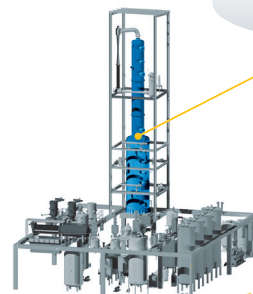
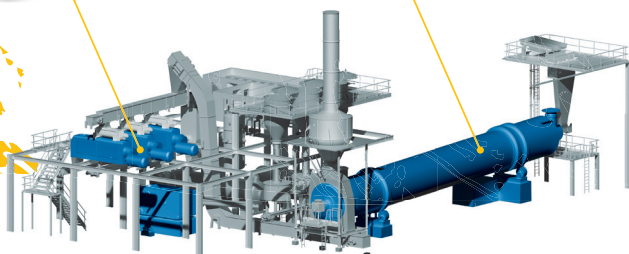
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# Switching to sustainable surfactants

Craig A. Bettenhausen

Personal care is going green at a breakneck pace. While most industries, including the chemical industry, are targeting net-zero greenhouse gas emissions by 2050, the consumer product brands that make soap, shampoo, lotion, and cosmetics are setting their sustainability goals at 2030, just 8 years away.

- Cleansers, lotions, and cosmetics all need surfactants, but many surfactants today are synthetic or semisynthetic.
- As personal care product makers go green, surfactants are at the center of their efforts.
- Chemical firms are responding with biobased surfactants and ways to make existing products from biomass feedstocks.

Those goals are also broader than just carbon dioxide emissions. When people buy personal care products, brand owners say, they are also looking for biodegradability, low environmental impact, and supply chains that are sustainable and ethical. Biobased is the keyword that consumers want to see on a product to know it meets those standards.

“What we are seeing, and have seen in the last few years, is that natural components are becoming a bigger driver of consumer behavior within the beauty and personal care category,” says Andrew McDougall, director of beauty and personal care for the market research firm Mintel. He says 98% of UK consumers bought “ecofriendly” beauty and personal care products in 2021, and natural ingredients are the top priority for 82% of Chinese beauty and personal care shoppers.

Surfactants are a prime target ingredient for making products more natural. These molecules play a central role in all types of personal care products, providing the grime-and-grease-removing power of face and body washes and holding disparate chemical phases together in lotions and makeup. Many surfactants also help moisturize and smooth skin and hair.

“It is a good time to be selling new ingredients,” says Neil Burns, a surfactant consultant and CEO of the personal care ingredient maker P2 Science. Personal care brands are “more receptive than I’ve seen them in a long time,” he says.

That atmosphere comes largely from the 2030 greenhouse gas emission targets set by many major consumer product companies, Burns says. “Big, big household names—the Procter & Gambles, Unilevers, L’Oréals—it’s all over their websites very publicly committing to some pretty ambitious goals regarding sustainability,” he says. “Given the scale of the commitments and given today’s slate of readily available raw materials and ingredients, they can’t get there from here. They need new stuff.”

But what are their options for that stuff, and which are the best for the planet? As they seek to replace synthetic surfactants with greener, low-carbon-footprint alternatives, consumer product makers have three main categories to choose from: microbial biosurfactants, inherently



biobased surfactants, and biobased versions of conventional surfactants. The decisions they make will create winners and losers in the chemical industry and could have a lasting impact on our environment.

## MICROBIAL BIOSURFACTANTS

Chemically speaking, surfactants are molecules that have both hydrophilic and hydrophobic sections. Soap, the old standby, has a carboxylic acid head attracted to water and a long hydrocarbon tail attracted to oil. Many other natural, synthetic, and semisynthetic molecules can do the same tricks with different combinations of polar and fatty molecular motifs.

The term *biosurfactant* refers to glycolipids produced by certain microorganisms. The water-soluble head in a biosurfactant is a sugar group, and the oil-soluble tail is a long, mostly saturated hydrocarbon chain. In nature, microbes use glycolipids for quorum sensing, adhesion, lubrication, and competition with other microorganisms.

The two most commercially advanced biosurfactants are rhamnolipids and sophorolipids, which feature rhamnose and sophorose, respectively, in their sugar heads. Within each of those families, structural variations can alter the surfactant's properties.



Booni Doon founder Pooja Ganesan says rhamnolipids give her powdered facial cleanser a gentle, effective foam.



The hydrocarbon tail of sophorolipids, for example, can either flop around loosely and terminate in a carboxylic acid or loop around and attach to the sugar head, creating a lactone ring. The lactone form is less foamy, explains Lawrence Clarke, a technical sales manager at Holiferm, a British glycolipid fermentation company.

Biosurfactants aren't new; references in the chemical literature date back to the 1950s. But they're newly available to the commercial market. A few home and personal cleansers, such as Booni Doon's powdered facial cleanser, already feature biosurfactants.

The strongest indicator of the interest in biosurfactants is the pace of dealmaking. A few days before In-cosmetics Global, a trade show for personal care ingredients held in Paris during the first week of April, Holiferm signed a deal in which the chemical company Sasol will buy most of the sophorolipids produced at a plant Holiferm plans to open in 2023 in the UK.

According to Louis Snyders, Sasol's global director of fabric, home care, and institutional and industrial cleaning, the deal brings products that complement Sasol's existing portfolio of surfactants, and it is a step in transforming that portfolio from its petrochemical heritage to a sustainable future.

Holiferm uses a strain of yeast, which it isolated from honey, that consumes sugar and sunflower oil to make the target surfactant. Clarke says the firm's semicontinuous process, which extracts sophorolipids throughout weeks-long fermentation runs, sets it apart from companies that use batch production methods.

BASF, a Sasol competitor, also has a deal with Holiferm and a controlling interest in the Japanese sophorolipid maker Allied Carbon Solutions. Nader Mahmoud, vice president of North American personal care business management at BASF, says the firm "intends to lead on biosurfactants." He says the Holiferm collaboration targets process development and manufacturing, with a focus on rhamnolipids and mannosylerythritol lipids (MELs), an emerging category of glycolipid surfactants.

Earlier this year, the surfactant fermentation specialist Locus Performance Ingredients signed a similar agreement to supply Dow with sophorolipids for the home and personal care markets. Isabel Almiro do Vale, Dow's global marketing and strategy director for personal care, says the scale-up and distribution incorporated into the deal will "democratize access to sophorolipids."

And in January, Evonik Industries announced plans to build a rhamnolipid plant in Slovakia at a "three-digit-million-euro" cost. Unilever is already on board as a customer, and Evonik representatives at In-cosmetics said buyers at the meeting were asking for the ingredient. Evonik had rhamnolipids on hand in a sample formulation of a facial cleanser cream.

The activity by chemical majors like Evonik and Dow has brought biosurfactants to the cusp of the personal care mainstream, according to Burns. "Those companies have sort of lent the field a little bit of commercial credibility," he says, and they "bring some scale that I think ultimately will help bring costs down."

Beyond being biobased, biosurfactants have a lower carbon footprint than conventional surfactants such as sodium lauryl ether sulfate (SLES), a common ingredient in personal care products, according to Dan Derr, a bioprocessing expert who helped develop the rhamnolipid technology Stepan acquired in 2020. That CO<sub>2</sub> advantage comes mostly from the mild conditions of fermentation, which is carried out at ambient temperature and pressure.

SLES is usually made by reacting fatty alcohols derived from palm oil with ethylene oxide and sulfur trioxide. Those steps consume a lot of energy because they take place at elevated temperatures and pressures. And though the oil component is biobased, palm oil is fraught with sustainability concerns of its own, including the deforestation required to build palm farms and greenhouse gas emissions from the leftover woody plant matter.

According to Clarke, a life-cycle analysis commissioned by Holiferm concluded that replacing 1 metric ton (t) of a typical ethoxylated surfactant with 1 t of sophorolipids would reduce greenhouse gas emissions by 1.5 t of CO<sub>2</sub>. Biosurfactants are also more potent by some measures than SLES and most other options, making it possible to use less in a final formulation.

"Fermentation becomes a really interesting process versus synthetic chemistry just because of the energy consumption," Almiro do Vale says. But because the feedstocks for biosurfactants today are sugars and oils, agricultural practices have a huge impact on sustainability. "You need to make sure that you're tracking really well your supply chain for the fermentation," she says.

Biosurfactant fermentation expert Sophie Roelants takes that concern a step further. She says a cradle-to-grave environmental analysis reveals that this first generation of biosurfactants is only marginally better than other types of surfactants. "The main reason is that you are dependent on agriculture to produce your feedstocks, and agriculture is really bad for the environment." Her conclusion is different from Holiferm's, Roelants says, because of how far back in the production process she starts counting carbon.

Roelants estimates that 80% of the net carbon emissions from biosurfactants happen while growing the feedstock. Seeing an opportunity, she recently cofounded the company Amphi-Star to commercialize biosurfactants made from industrial and food waste. The firm, a spin-off from Ghent University and the nonprofit Bio Base Europe Pilot Plant, is looking for partners to help it scale up and improve process efficiency.

## INHERENTLY BIOBASED

Other biobased surfactants—made by chemically modifying and combining molecules extracted from plants—have been available for years but are now enjoying increased interest from personal care brands.

The most popular class in this category is alkyl polyglucosides, or APGs. Chemically similar to microbial glycolipids, APGs are made by combining glucose or other sugars with fatty alcohols derived from plant oils. The reaction is driven by inorganic catalysts or enzymes. Like biosurfactants, APGs are milder and



Personal care brands are leaning heavily on natural and biobased claims, as seen in this display at a Sephora store in Paris.

generally have a lower carbon footprint than conventional surfactants.

APGs are not a slam dunk for personal care products, however, because they cost more and aren't as foamy. According to sales representatives from Colonial Chemical who were at In-cosmetics, APGs cost two and a half to three times as much as SLES. Foaming doesn't actually improve personal care cleansers' effectiveness much, but people see bubbles as a sign of efficacy.

Though not as popular as SLES and related ethoxylated ingredients, APGs are already widespread in personal care. Marcelo Lu, BASF's senior vice president for care chemicals in North America, says BASF is working with some global brands on "chassis change"—reformulating products around APGs and other inherently biobased surfactants. "We're talking about major volumes," he says.

"In a way, we were a bit ahead of our time," Lu says, noting that BASF has produced APGs for decades. "But because of regulatory forces and also the focus on biobased content, you see a lot of brands now paying more attention."

"We've flipped quite a few formulations already. And I think we are in the beginning stage of this," Lu says. "We may get into a supply constraint if everybody starts switching."

BASF has APG plants in the US, Asia, and Europe and is looking to expand capacity, according to Lu. Other suppliers, such as Solvay, Dow, and Colonial, also offer APGs at commercial scale. For delivering low-CO<sub>2</sub>, biobased surfactants at the scale needed to support the green ambitions of global brands, "APGs are the closest," Lu says.

## CONVENTIONAL, BUT BIOBASED

Even with help from suppliers, reformulation to accommodate new ingredients is not trivial; it's expensive and risky for a brand owner to change a product line that already works. Avoiding reformulation is the value proposition that some major chemical companies are bringing with biobased versions of conventional surfactants.

Especially in personal care, many workhorse surfactants, such as SLES, are already partially biobased. Roughly half the carbons in an ethoxylated, vegetable oil-based surfactant have biomass origins.

Two chemical makers, Croda International and Clariant, have made a change allowing them to get to 100% biobased: they are deriving their ethylene oxide from plants instead of fossil resources. Croda launched its Eco range of surfactants in 2018, and Clariant's Vita-branded surfactants and polyethylene glycols came out in February of this year.

The chemistry used by both was developed primarily by the New Jersey-based engineering firm Scientific Design. The process starts by dehydrating plant-derived ethanol into ethylene. The subsequent steps of oxidizing ethylene to ethylene oxide and then creating ethoxylated surfactants are the same as in the synthetic route, though Scientific Design offers systems that integrate all three steps.

Both Croda and Clariant say the production lines for their biobased ethoxylated surfactants are fully segregated from those of their conventional counterparts, and they offer customers carbon isotope data to prove that no petroleum carbon is mixed in. Fossil carbon doesn't contain the heavy carbon isotope <sup>14</sup>C, whereas biobased carbon does, so the amount of <sup>14</sup>C in a sample can indicate where its carbons came from.

Although consumers value the labels that such biobased products permit, for environmental advocates, being plant derived is not as central a goal as it once was. A lot of the talk around sustainability in the chemical industry today is about carbon emissions, and the CO<sub>2</sub> benefits of biobased ethylene oxide are not cut and dried.

According to a life-cycle analysis published by Clariant, making a kilogram of ethylene oxide from fossil fuels emits the equivalent of 1.5 kg of CO<sub>2</sub>. Making that same kilogram from ethanol emits slightly less, 1.4 kg of CO<sub>2</sub>. But the biomass that's fermented into ethanol absorbs 2.0 kg of CO<sub>2</sub> from the air as it grows, Clariant says. So from corn or sugarcane to ethylene oxide, the process is carbon negative to the tune of 0.6 kg of CO<sub>2</sub> per kilogram of ethylene oxide.

But that number does not count the greenhouse gas emissions associated with growing the biomass. And in the case of corn and sugarcane, those emissions can be substantial. Debates on the environmental impact of producing ethanol from edible sugars have raged since Brazil began blending ethanol into gasoline in the 1970s.

David Schwalje, head of emerging market development for the fuel and chemical engineering firm Axens, says the provenance of the ethanol feedstock makes all the difference when it comes to measuring the carbon intensity of the resulting products. Ethylene oxide made from conventionally grown corn or sugarcane alcohol—often called first-generation ethanol—isn't reliably better from an emission standpoint than ethylene oxide made from petroleum, he says.

A look at some publicly available numbers shows how widely the carbon footprint can range for the compound. The carbon emissions of growing the corn needed to produce a kilogram of biobased ethylene oxide—including from fertilizer production, truck and tractor fuel, and other emission sources—



## Surfactants used in personal care products vary in properties and applications.

### Rhamnolipids

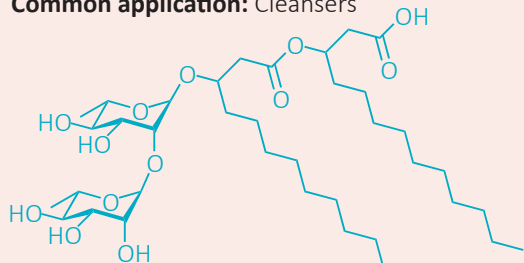
**Type:** Biosurfactant

**Typical concentration in a shampoo:** 2–10%

**Foam amount:** High

**Mildness:** Very mild

**Common application:** Cleansers



Rhamnolipid

### Alkyl polyethylene glycol ethers

**Type:** Synthetic

**Typical concentration in a shampoo:** 15–25%

**Foam amount:** Moderate

**Mildness:** Irritating

**Common application:** Creams and gels



$n = 3-9$

Alkyl polyethylene glycol ether

### Alkyl polyglucosides (APGs)

**Type:** Inherently biobased

**Typical concentration in a shampoo:** 10–40%

**Foam amount:** Moderate

**Mildness:** Mild

**Common application:** Solid shampoos, skin-care products



Lauryl glucoside, an APG

### Sophorolipids

**Type:** Biosurfactant

**Typical concentration in a shampoo:** 0.5–10%

**Foam amount:** Low

**Mildness:** Very mild

**Common application:** Micellar-water makeup removers



Straight-chain sophorolipid

### Sodium lauryl ether sulfate (SLES)

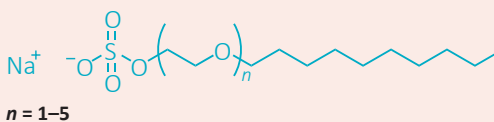
**Type:** Semisynthetic

**Typical concentration in a shampoo:** 40%

**Foam amount:** High

**Mildness:** Moderate

**Common application:** Cleansers



$n = 1-5$

Sodium lauryl ether sulfate

### Sodium lauryl sulfate (SLS)

**Type:** Semisynthetic

**Typical concentration in a shampoo:** 30%

**Foam amount:** High

**Mildness:** Irritating

**Common application:** Cleansers



Sodium lauryl sulfate

**Sources:** Patents, SpecialChem.com, Croda International, Clariant, Stepan, Colonial Chemical, Dow, BASF.

were between 0.8 and 2.8 kg of CO<sub>2</sub>, according to C&EN calculations based on estimates from Argonne National Laboratory and the University of Minnesota (*Biofuels, Bioprod. Biorefin.* 2021, DOI: 10.1002/bbb.2225; *Proc. Natl. Acad. Sci. U.S.A.* 2017, DOI: 10.1073/pnas.1703793114). Factor in Clariant's numbers on ethylene oxide production, and the biobased route offers anywhere from an 86% reduction to a 46% increase in CO<sub>2</sub> emissions relative to the petrochemical route.

However, Schwalje says, second-generation ethanol made from waste or sustainably grown cellulose where the fermentation and distillation equipment uses carbon capture can be massively carbon negative and carry that CO<sub>2</sub> advantage downstream to the products made from it.

Both Croda and Clariant are using first-generation ethanol, for now at least. Croda makes its Eco products from corn-based ethanol at a \$170 million, purpose-built plant in

Delaware that has a capacity of roughly 30,000 t per year. The plant is powered by methane captured from a nearby landfill, reducing the carbon footprint of the ingredients produced there, the firm says.

Clariant's sugarcane- and corn-based plant is in Uttarakhand, India, part of a joint venture with India Glycols. Fabio Caravieri, head of global marketing for Clariant's industrial and consumer specialties division, says the firm has "double-digit kilotons" of its Vita products available. "It's not something where we are doing an investment today that in 2 to 3 years will be available to the market. It's now," he says.

Just using biobased ethylene oxide is not going to make a shampoo or body wash carbon negative, Caravieri acknowledges, but it does offer an improvement. Clariant says that because of the specific feedstock and equipment at the plant in India, a consumer product maker can claim a car-

bon footprint reduction of up to 2 kg of CO<sub>2</sub> for every 1 kg of surfactant.

And Clariant is in a position to do more. The firm operates one of the world's only second-generation ethanol plants, a 50,000-t-per-year facility in Romania that started making ethanol from straw in 2021. That cellulosic ethanol could become a feedstock for Vita products in the future, Caravieri says, if such a combination looks profitable once the firm has more experience with both products.

## THE MASS-BALANCE WAY

Other major chemical firms active in surfactants are meeting the demand for renewable carbon content through an accounting approach known as mass balance. As with the ethanol-based ethoxylates, the surfactants made through this method are chemically identical to what is already on the market. But the mass-balance approach introduces the biomass further upstream. It is blended with fossil-carbon feedstocks like naphtha or natural gas as those substances are fed into the crackers that make ethylene and other building-block chemicals.

Mass-balance accounting methods vary, but the basic idea is that an operator gets credits for each biobased carbon atom fed into its cracker. It can assign those credits to a portion of the plant's output containing the same number of carbon atoms. Customers that want to buy from the biobased portion pay the market rate for the conventional chemical plus a supplement for the biomass, or renewable, carbon.

For example, if a company makes 100 kg of a surfactant using a raw material with 10% renewable content, it can claim that 10 kg of the surfactant is 100% renewable under the mass-balance concept, even though really all 100 kg have 10% renewable content.

As of January, Sasol is offering mass-balance surfactants and other chemicals to its customers in Europe by buying ethylene made with enough renewable carbon to meet its mass-balance orders from customers. Operating that way offers nimbleness and scalability that new ingredients and segregated facilities do not, Snyders says. Sasol makes a wide range of chemicals from the same handful of building blocks, so changing which products get credited with the renewable carbon is just a matter of bookkeeping.

Sasol started with thousands of metric tons of mass-balance SLES and plans to quickly expand to tens of thousands, Snyders says, though Russia's invasion of Ukraine has slowed the timeline. He says mass-balance offerings are a first step and one Sasol can take now. Long term, he says, the hope is to create feedstock-segregated offerings by converting entire units to recycled and biomass-carbon sources.

The biomass supply chain needs work before that's even feasible, however. For example, Sasol has capacity for around 400,000 t per year of ethylene oxide in Lake Charles, Louisiana. Snyders says it's impossible right now to supply a whole facility of that size with biomass feedstocks suitable for use in petrochemical equipment.

Sasol isn't the only fan of mass balance. At the end of March, BASF and the consumer product maker Henkel signed a deal in which 110,000 t per year of the ingredients BASF makes for Henkel in Europe will be manufactured with renewable feedstocks on a mass-balance basis. The firms say the move will lighten the carbon footprint of Henkel brands, including Persil, Pril, and Fa by 200,000 t per year.

Despite the logistic advantages of mass balance, the approach doesn't convince all end users. Mass-balance certification abbreviations such as ISCC and REDcert<sup>2</sup> don't mean much to the lay consumer. And even if the mass-balance bookkeeping is legitimate, the carbons in the final product are a mix of plant based and fossil sourced, and that's not what many shoppers want, Intel's McDougall says.

Ivo Grgic, Henkel's global purchasing category manager for surfactants, acknowledges the concerns, but he says mass balance is the fastest next step the firm can take to make its products more sustainable. "We clearly decided we want to deliver a contribution today, now. That doesn't mean that we are going to close the door for these new technologies in the next years. Not at all. But we need to start."

Grgic says the 110,000 t from BASF is a big chunk of Henkel's overall annual ingredient volume for consumer brands in Europe, and surfactants are the biggest portion of the deal. Adding mass-balance purchases in other parts of the world will be next.

"We are a company producing big, big, big volumes," Grgic says. "From our point of view, we need to make an impact, and we decided that biomass balance is the approach where we can replace fossil carbon on a big scale in the fastest way. Other technologies, like CO<sub>2</sub> capture and biosurfactants—they will follow in the next coming years."

Consumer product brands looking to go biobased have options: new ingredients like glycolipids, expanded use of specialty ingredients like APGs, and newly biobased versions of the ingredients they're used to. But the choices involve a complex balance of sustainability, efficacy, availability, and of course, cost. Caravieri says ecoconscious consumers are willing to tolerate a 25–40% price premium before they are turned off.

The chemistry behind biobased surfactants has been known for years. The shift toward them is happening now, Roelants says, because consumers are more conscious of what they're using to clean their faces and shampoo their hair. In ever-increasing numbers, they want biobased, sustainable products.

Buyers like Henkel are taking an all-of-the-above approach to meeting that demand, even as they look down the road for better approaches. "Folks are looking for sustainability," Burns says, "and want to try and access it from any and all areas they can."

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Recognizes an individual who has made significant contributions to the Division's area of interest, or whose work has resulted in major advances in health and nutrition.

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Recognizes a young scientist who is making significant and substantial research contributions in one of the areas represented by the Health and Nutrition Division of AOCS.

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 The award recipient must agree to attend the AOCS Annual Meeting & Expo and present an award lecture. The 2023 AOCS Annual Meeting & Expo will be held in Denver, Colorado, USA, from April 30–May 3, 2023.

\*As of May 26, 2022. Award details subject to change.



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# Testing enzymes:

Arjan Siebum and David Hong

## When everything is good, nothing is good

The days of boiling the laundry with some soap in a kettle are long past. Technologies like bleach, surfactants and enzymes have delivered enormous performance improvements to the cleaning power of detergents over time. Nowadays, detergents deliver cold-water wash performance that 20 years ago would have been unimaginable. Enzymes, produced in an industrial fermentation process, are the key ingredients that contribute to this performance enhancement. Walk down the laundry aisle in your supermarket and you will see that over three-quarters of the detergents are enzymatic – most of these with multiple enzyme functions. In fact, nearly all mid-to-upper tier detergents contain enzymes. But how exactly do we know to what extent enzymes contribute to the overall cleaning process?

- Enzymes are powerful contributors to detergent cleaning performance.
- Faulty experimental design can lead to poor decision-making for enzyme selection or dosage during formulation development.
- We address common pitfalls and provide guidelines for optimization in test design.

Detergent performance is often measured using regional industry test protocols focused on stain removal using test monitors or artificial stains. However, these tests do not always properly assess the benefits of enzymes in detergents. Inadequate choice of test materials or test conditions can cause a loss of discriminative power in tests. This leads to poor decisions when choosing enzyme function or dosage when formulating a detergent. This article will explain some common pitfalls in setting up cleaning performance tests and will provide guidelines to avoid them.

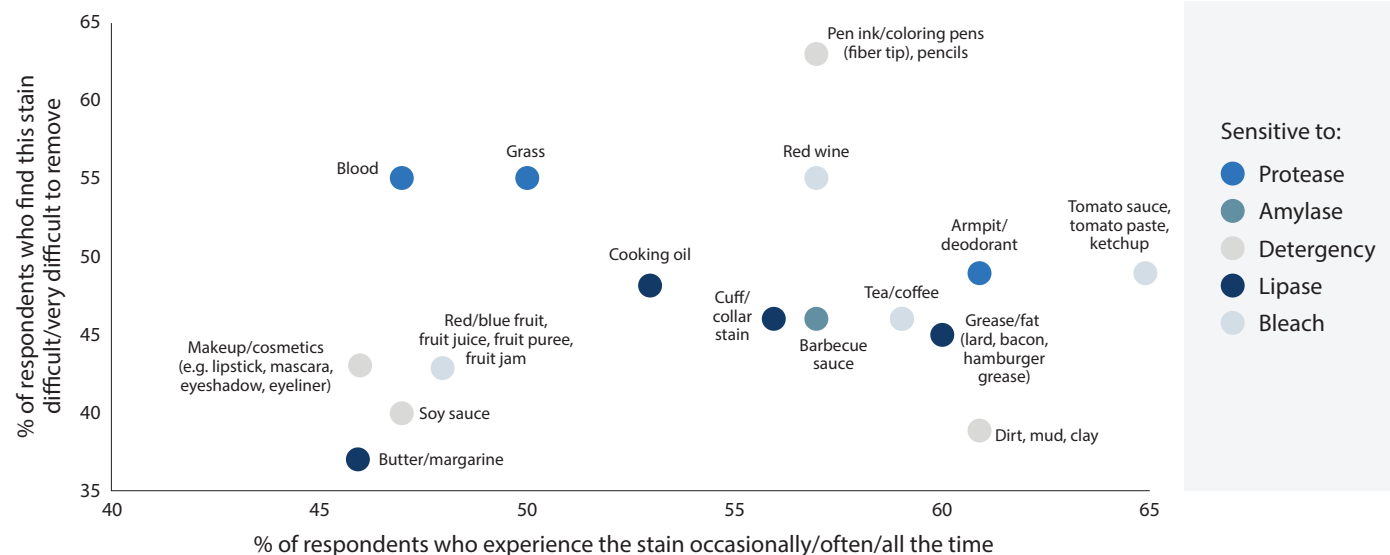
### PERFORMANCE AS A MAIN DRIVER FOR CONSUMER SATISFACTION

Stain removal remains one of the most important attributes of a detergent. An inability to provide satisfactory cleaning perfor-

## Many stains are responsive to enzymes

How often do you experience a stain caused by each of the following items?

How hard are the following stains to remove?



**FIG. 1: Upper quadrant of the IFF stain hierarchy, showing prevalence of stains (x-axis) vs their ease of removal (y-axis).** Source: IFF consumer survey, 2017. Base: North America (702).

mance will drive customers away from a brand. The value of enzymes lies in the fact that they are an important ingredient for stain removal; and therefore, a key contributor to meeting consumers' expected performance level. This is demonstrated in the picture below.

International Flavors & Fragrances, Inc (IFF) surveyed a large group of consumers on their food consumption (and spilling) habits, asking them how often they encountered stains and how hard those stains were to remove. We used the results to build a stain hierarchy. The graph shows how prevalent stains are (along the x-axis) and how hard they are to remove (height on the y-axis). Many of the stains that are both common and hard to remove, like blood, BBQ sauce or hamburger grease, are enzyme sensitive. We conclude that enzymes remain essential for an optimal cleaning performance and that detergent companies can still improve on stain removal. A detergent can be formulated to stand out by investing the right type and dosage of enzymes.

## CHALLENGE OF DEFINING A DISCRIMINATIVE TEST

Detergent manufacturers invest in enzymes to deliver satisfactory cleaning performance to the end user. However, if the cleaning performance cannot be properly assessed, a formulator cannot determine benefits versus investment. As a class of ingredients in detergents, enzymes can take up to 25% of total formulation cost. This is a significant portion. If test wash results cannot properly measure the cleaning contribution of enzymes, the expenditure on enzymes becomes a risk. A poorly set-up performance trial can underestimate the enzyme cleaning contribution, potentially leading to over-

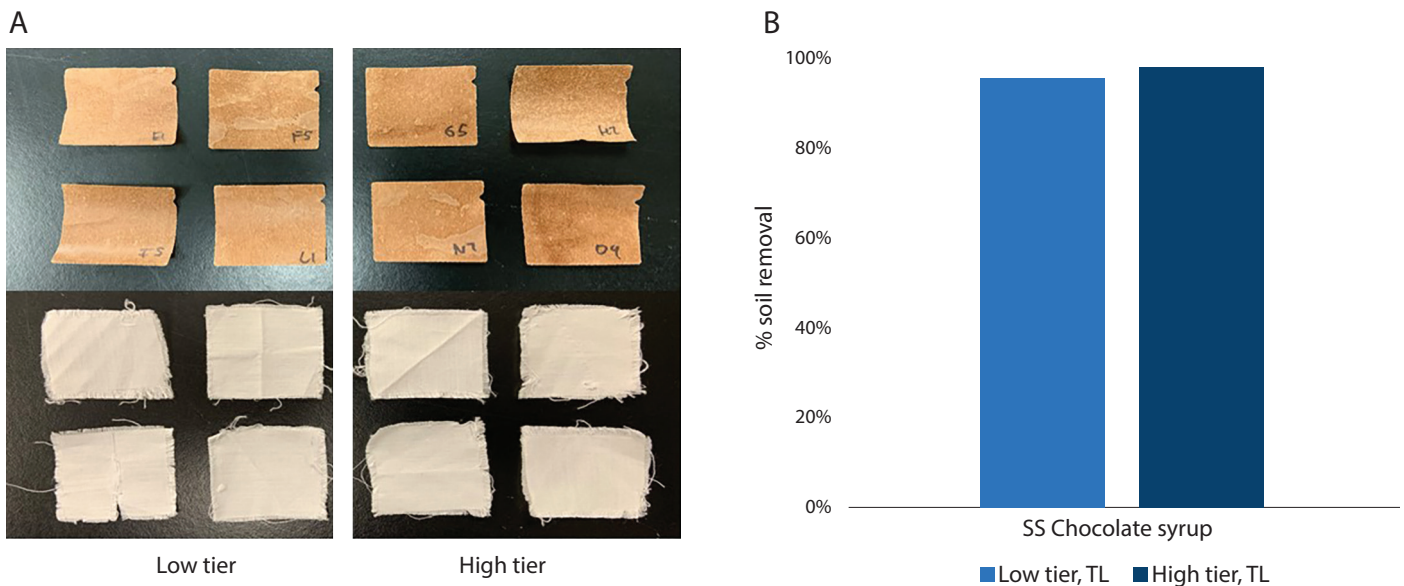
dosing of enzymes. On the contrary, an overestimation of the enzyme contribution can potentially lead to underdosing and subsequent customer dissatisfaction with the detergent's performance.

## STAIN SELECTION IS KEY

One of the largest factors for a proper measurement of enzymatic performance is the choice of the stain. Poor stain selection during performance assessments cause pitfalls such as, complete wash out, no dynamic range, and inconsistent results. Figure 2 shows the impact of improper stain selection on test outcomes. Here you see the wash results for a commonly encountered real life stain, chocolate syrup, converted to a commercial test stain. The commercial stains are cotton or polycotton swatches that are impregnated with syrup. The reflectance of the stains is measured before and after washing. When using a standard 30°C wash program, the stains are totally cleaned. It is not possible to show the difference between a low-tier and a high-tier detergent using this stain, even though the high-tier detergent contains enzymes and a significantly higher level of surfactants. Running this experiment when formulating a detergent would have been a waste of time, as the results tell you nothing. Using test materials like these when testing new detergent compositions may lead to a wrong decision with regards to the effectiveness of enzymatic detergents.

Another problem that is often encountered is the variation in soil removal between washes, or poor reproducibility of results. Also, stains can exhibit uneven soil removal across the surface of the swatch, resulting in high variability. Both will give results with large error margins, which make it impossible





**FIG. 2. (A)** Example of a stain with (too) little dynamic range. Unwashed stains depicted top left, washed stains bottom-left. The stains are almost completely clean after a regular main wash and no real difference can be seen between a low tier and a high tier detergent. **(B)** Photo-spectrometric soil removal measurements confirm that there is no real performance difference discernible between a low and high tier detergent using these test materials.

to reliably compare the performance of different detergents or how different enzymes stack up against each other (figure 3). This is an issue that affects both detergent manufacturers and enzyme developers.

At IFF, we use the latest biotechnology tools to alter the structure of enzymes and improve their properties to achieve specific performance targets. Enzymes have become more effective cleaners, are becoming more concentrated, and they are being dosed at increasingly higher levels. To perceive this evolution, we need stain cleaning tests that can keep pace with enzyme changes. If we see complete washout like shown

previously, we will not be able to quantify the benefits of protein engineering. By combining market research and expertise in application testing developed over the years, IFF works with suppliers and customers to further develop differentiating stain sets for assessing enzymatic performance in detergents.

## INDUSTRY GUIDANCE CAN HELP

Recently, there have been efforts in Europe to update and improve the stain set used in the AISE (International Association for Soaps, Detergents and Maintenance Products) detergent test protocol. In North America, the ASTM



**FIG. 3.** Example of a swatch with inconsistent cleaning (SS Blood). The pictures show uneven cleaning across the surface of one swatch (A), between swatches (B), and the resulting effects when comparing wash performance (C). No conclusions can be drawn from differences in performance between detergents due to the large experimental error.

(American Society for Testing and Materials) method currently specifies a broad stain set and allows flexibility in choosing stains made by stain manufacturers or stains made by hand. Their guideline provides a good starting point.

In particular, they have selected a list of stains that occur often and are challenging to remove. Most of these appear in the often/difficult to remove quadrant of our “pain of the stain” graph shown earlier (figure 1). However, ASTM’s guidelines suggest testers prepare this category of stains themselves. This is time consuming and introduces a lot of measurement error due to stain-to-stain, batch-to-batch, and lab-to-lab variability. This can lead to large standard deviations, inconsistent results and makes it hard to compare results over time. Fortunately, specialized suppliers exist who create consistent quality stains and provide suitable test materials for these types of experiments.

There are several criteria that make a good stain:

- Stains need to be sensitive to the enzymes of interest. This would seem obvious, but too often decisions on enzyme incorporation are made on the cleaning results of stains that react mostly to the presence of surfactant or bleach.
- Stains need good reproducibility to give confidence that your results are real. It is essential that stains give consistent results that do not vary due to washes being

carried out on a different day or by a different operator. Moreover, the stain must clean evenly, without darker and lighter regions. Otherwise, this would leave you guessing which region represented the real cleaning performance.

- A good stain has a wide dynamic range and shows a response that depends on the enzyme dosage. This means that the difference in soil removal between a non-enzymatic detergent and the same detergent dosed with a high amount of enzyme should be large. Incremental improvements in cleaning should be visible with increasing levels of enzyme dosage in the detergent. A stain that is either completely removed or shows only a small amount of soil removal between no enzyme and a high enzyme dose will not be able to differentiate between enzyme levels or low-tier and high-tier detergents.

## AN IFF CASE STUDY

To take a closer look at this challenge, IFF conducted a benchmark study. We carefully selected stains based on the criteria outlined above and took consumer relevance into account. We started with a set of 19 commercially available soiled swatches from several vendors that covered a broad range of commonly encountered stains. We performed an initial wash trial and

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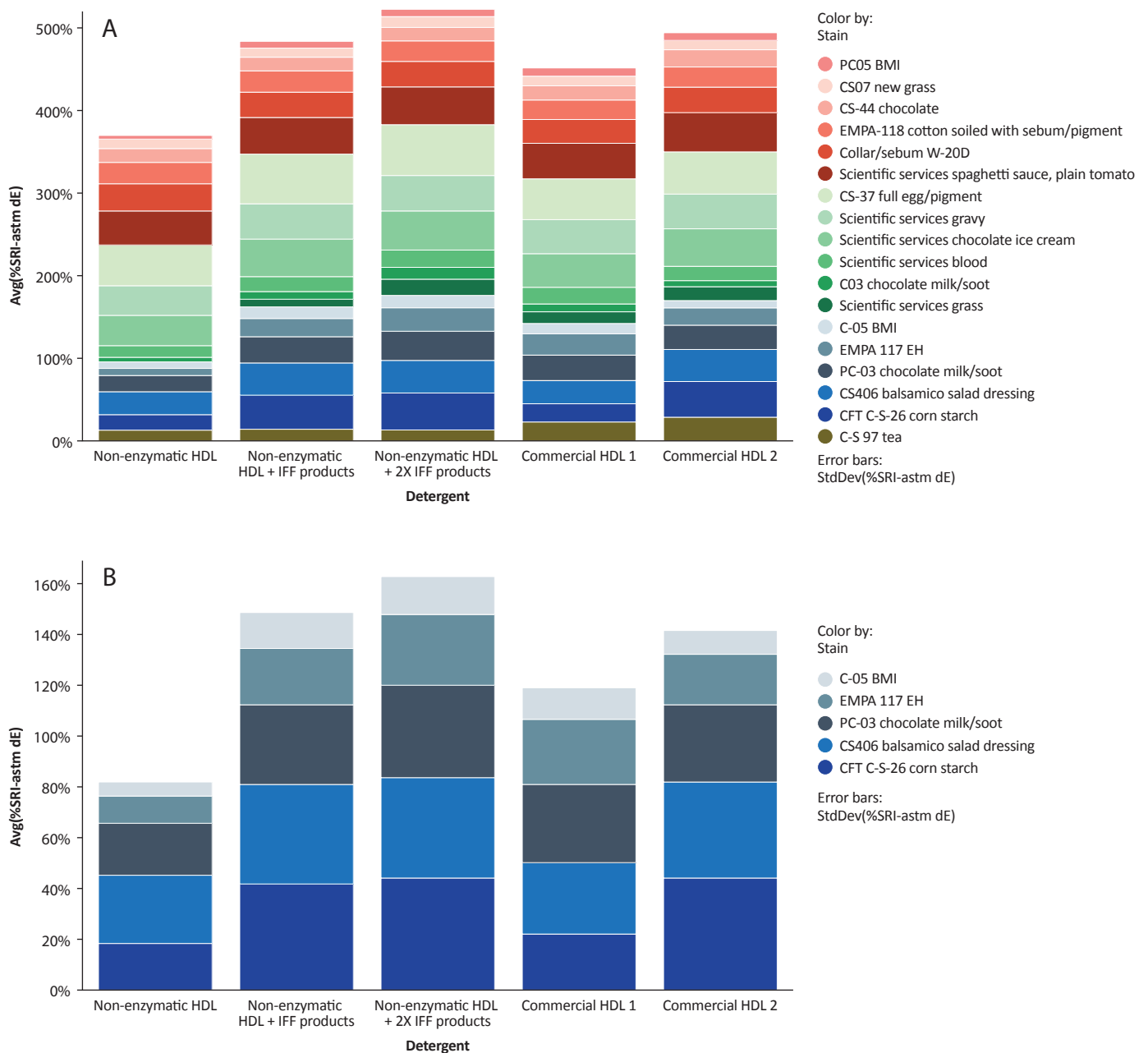
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**FIG 4. Comparison of the cumulative performance over a full stain set (A) vs the cumulative performance on a sub-set of responsive, reproducible, enzyme sensitive stains (B).**

removed the stains that had no dynamic range. They either showed nearly complete wash-out or little response. We then removed the stains that showed inconsistent results between swatches in a wash. We were left with a set of six stains that gave reproducible results, with a good dynamic range and a good dose-response relationship for the enzyme-sensitive stains.

If we compare the performance profiles before and after removing the unresponsive (red) and inconsistent (green) and non-enzymatic stains (brown), a clear difference in soil removal between the different detergents is visible in figure 4. The non-enzymatic detergent now has a much lower soil removal level than the others. Looking at the two off-the-

shelf, commercial detergents it is immediately visible which is the premium and which is the value detergent. Finally, this stain set allows us to see what the effects are of adding enzymes to a detergent. The dramatic increase in performance is apparent and the data indicate what enzymes should be included to match or exceed the performance of commercial formulations.

We learned that selecting the right stain set for your experiments is key to optimizing the enzymes for a detergent formulation. It is essential that the test yields accurate and useful information. This means that some groundwork is required, like screening the stain set to assess its relevance, sensitivity, reproducibility, and dynamic range.

In the end, you want to be able to see which detergent performs better and which worse. You want to be able to see the effect of additional investments on performance or other formulation changes. If all detergents, regardless of quality, pass the test, then none do. That is why: 'When everything is good, nothing is good.'

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*David Hong, currently employed at Culture Biosciences, worked as a senior application associate at IFF for 10 years in Home and Personal Care, designing performance experiments and market studies for detergents and enzymes.*

*This article is based on IFF's AOCS 2021 Annual Meeting presentation.*

## AOCS MEETING WATCH

**July 31–August 3, 2022.** Edible Oil Products Processing Course, Fats and Oils R&D Center LLC, College Station, Texas, USA. (<https://fatsandoilrnd.com/annual-courses/>)

**August 27–September 1, 2022.** World Congress on Oleo Science, hosted by the Japan Oil Chemists' Society, Online. (<https://jocs.jp/en/conference-meeting/>)

**October 4–6, 2022.** Sustainable Protein Forum, Millennium Knickerbocker Hotel, Chicago, Illinois, USA and Online.

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

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# Formulation engineering 2.0

Edgar J. Acosta

- The HLD is a number that indicates the approach to the phase inversion point of surfactant-oil-water (SOW) systems and is obtained from empirical correlations that consider formulation conditions such as oil and surfactant hydrophobicity, salinity, and temperature.
- While the HLD number is a great formulation tool, it does not produce quantitative values of SOW system properties such as solubilization and interfacial tension (IFT).
- The NAC uses critical scaling theory to predict oil and water solubilization radii ( $R_o$ ,  $R_w$ ), employing the HLD as the scaling distance to the critical point (HLD=0, or net-zero curvature), and later introducing the concept of interfacial rigidity to predict IFT.
- When NAC-predicted properties are combined with transport concepts such as the Weber, Bond, and Capillary numbers, one can predict formulation performance and understand the connection between formulation and end-use conditions, accelerating the progress towards optimized SOW-based products and processes.

In 2010, an *INFORM* article highlighted the use of the hydrophilic-lipophilic-difference (HLD) and net-average curvature (NAC) to formulate surfactant-oil-water (SOW) systems. There are now plenty of industrial and academic examples of HLD-guided formulations in oil extraction, environmental remediation, detergency, drug delivery, agrochemicals, cosmetics, and others. However, there are only a few reports on NAC-guided formulations. This article introduces Formulation Engineering 2.0, where NAC is combined with transport equations to predict the performance of products and processes.

The HLD can be traced to the 1977 dissertation of Jean-Louis Salager, a retired professor at University of the Andes in Mérida, Venezuela. The model he produced was an empirical correlation for the phase inversion of ionic surfactants in SOW systems as a function of salinity ( $S$ , in g NaCl/100 mL), oil hydrophobicity (equivalent n-alkane carbon number or EACN), surfactant hydrophobicity (surfactant parameter,  $\sigma$  for ionics,  $\beta$  for nonionics), and the concentration of medium-chain alcohols or cosurfactants ( $f(A)$ ). Eventually, a term for temperature ( $T$ ) was included, as well as the development of the correlation for nonionic surfactants, leading to the following HLD equations:

$$\ln(S) - k \cdot \text{EACN} - \alpha_T \cdot (T - 25^\circ\text{C}) + \sigma = \text{HLD}_{\text{ionic}} \propto (\mu_{s,w} - \mu_{s,o}) / (R \cdot T) \quad \text{Eq. (1)}$$

$$b \cdot S - k \cdot \text{EACN} + c_T \cdot (T - 25^\circ\text{C}) + \beta = \text{HLD}_{\text{nonionic}} \propto (\mu_{s,w} - \mu_{s,o}) / (R \cdot T) \quad \text{Eq. (2)}$$

where  $k$ ,  $b$ ,  $\alpha_T$ ,  $c_T$  are surfactant-dependent constants and  $R$  is the gas constant. In a 2000 paper, the Salager group introduced the HLD denomination and the HLD validation as representing the difference in surfactant chemical potential in water and oil ( $\mu_{s,w}$ ,  $\mu_{s,o}$ ).

My first exposure to the HLD correlations was in 1999, when I was a Master's student. I used the HLD as a guide to formulate systems that could promote solubilization of chlorinated solvents while maintaining high interfacial tension (IFT or  $\gamma$ ) to prevent oil mobilization. At the time, Salager and others introduced qualitative relationships between HLD, solubilization and IFT. However,

even using HLD, it took more than one thousand test tubes and 1.5 years of work to get a quantitative answer. It became clear that the point was to find the appropriate surfactant tail length ( $L_T$ ) and the appropriate negative HLD to achieve the desired effect. This experience highlighted the need for an equation of state for SOW systems that could predict SOW properties starting from formulation conditions.

The quantitative prediction of solubilization capacity and IFT from HLD became a central question for my doctorate degree. Jeff Harwell, professor at the University of Oklahoma in Norman, pointed me to critical scaling theory, where the property of a system in a given state is a function of the chemical potential difference (or distance) between the state and the critical state. The critical state is the transition point between two extremes. For SOW systems, the critical state has oil-like and water-like properties, corresponding to middle phase microemulsions ( $\mu E$ ) at  $HLD=0$ . Other research groups have previously scaled the curvature of  $\mu E$  to the temperature approach to the phase inversion temperature (PIT) as the distance scale. The NAC starts here with a few modifications.

The first modification was that instead of curvature—which requires expensive facilities to be assessed—the inverse of a sphere-equivalent solubilization radius of oil ( $R_o$ ) or water ( $R_w$ ) was the scaled property. The second is that instead of temperature, the value of the HLD was used as the distance to the critical point ( $HLD=0$ ), leading to the equation  $1/R_o = -HLD/L$ , where  $L$  is a scaling length constant. While this equation is adequate at  $HLD < -1$ , as  $HLD$  approaches 0,  $R_o$  tends to infinite, which is not realistic. Bicontinuous  $\mu E$ s achieve net-zero curvature via coexisting regions of positive and negative curvature.

The NAC represents this two-curvature space assuming the coexistence of two alternate hypothetical states, one where oil is solubilized in water ( $R_o$ ) and another where water is solubilized in oil ( $R_w$ ). Based on this approximation, the net curvature ( $H_n$ ) is:

$$H_n = (1/R_o - 1/R_w) = -HLD/L \quad \text{Eq. (3)}$$

At  $HLD=0$ , one obtains equal and finite water and oil solubilization such that  $R_o = R_w$ . The scaling length  $L$  is approximately 1.2 to 1.4 times  $L_T$ , estimated using cheminformatic tools or from Tanford's length correlation for hydrocarbon tails. Nevertheless, Eq. 3 does not give  $R_o$  at  $HLD=0$ . Finding  $R_o$  and  $R_w$  at  $HLD$  near-zero requires the concept of characteristic length ( $\xi$ ), which is the maximum  $\mu E$  solubilization radius. The average curvature ( $H_a$ ) represents the inverse of the  $\mu E$  solubilization radius:

$$H_a = (1/2)(1/R_o + 1/R_w) \geq 1/\xi \quad \text{Eq. (4)}$$

At  $HLD=0$ , then  $R_o = R_w = \xi$ . Currently,  $\xi$  is one of the NAC inputs that cannot be predicted from the surfactant/oil structure. However,  $\xi$  can be fit using  $\mu E$  phase scans.

One advantage of NAC over previous models is that it can be implemented with limited resources. One can use phase volumes or liquid chromatography to obtain the volume frac-

tion of oil ( $\phi_o$ ) or water ( $\phi_w$ ) solubilized in a solution containing a given volume fraction of surfactant ( $\phi_s$ ). These values give  $Ro = 3(\phi_o/\phi_s) \cdot (v_s/a_s)$  and  $Rw = 3(\phi_w/\phi_s) \cdot (v_s/a_s)$ , where  $(v_s/a_s)$  is the volume to area ratio of the surfactant molecule at the interface;  $v_s$  can be obtained from the molecular weight and density of the surfactant;  $a_s$  can be estimated from surface tension isotherms.  $v_s$  and  $a_s$  can also be estimated using cheminformatic software that calculates the minimum and polar surface area of molecules starting from their SMILES structure.

The  $\mu E$ -oil IFT can be predicted considering that the interfacial free energy of a sphere with radius  $R_o$  is  $4\pi R_o^2 \cdot \gamma_{\mu E-oil}$ .  $\mu E$  are in thermodynamic equilibrium; therefore, this interfacial free energy must be compensated by the surfactant-surfactant interaction, represented by the interfacial rigidity,  $E_r$ , such that  $\Delta G = 4\pi R_o^2 \gamma_{\mu E-oil} - E_r = 0$ , then:

$$IFT_{\mu E-oil} = \gamma_{\mu E-oil} = E_r/(4\pi R_o^2) \quad \text{Eq. (5)}$$

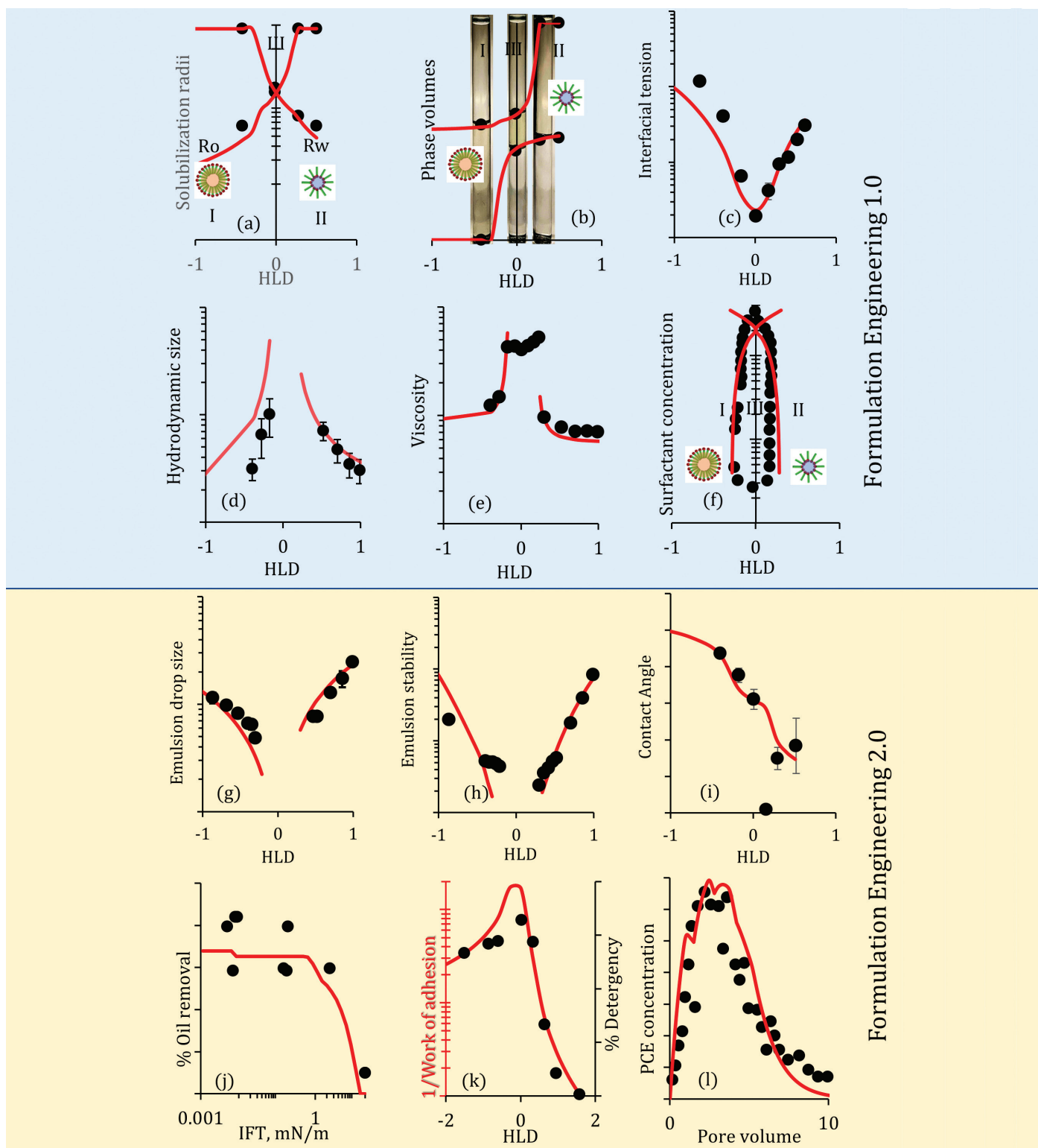
$E_r$  often ranges from  $1K_B T$  for flexible  $\mu E$ s to  $10 K_B T$  for rigid  $\mu E$ s.  $K_B$  is Boltzmann's constant. Eq. (5) can be used to find  $\gamma_{\mu E-water}$  using  $R_w$  instead of  $R_o$ .

Equations 1 thru 5 represent the core of the HLD-NAC. Figures 1a-c represent the basic HLD-NAC predictions ( $R_o$ ,  $R_w$ , IFT), shown as solid red lines. The HLD,  $L$  and  $v_s/a_s$  parameters for sodium dihexylsulfosuccinate (SDHS)-toluene in Figures 1a-i were obtained from the literature, and  $\xi = 70\text{\AA}$  and  $E_r = 1.5 K_B T$  were fitted. To calculate the hydrodynamic radius in Figure 1d, one must assume that  $\mu E$  drops are cylinders of length  $L_c$  with hemispherical caps with radius  $R_d$ . To obtain  $L_c$ ,  $R_d$ , one interprets  $H_n$  as the area-averaged interfacial curvature and  $H_a$  as the cylinders' sphere-equivalent area/volume ratio. These  $\mu E$  dimensions are then incorporated into a model for the viscosity of rigid rod suspensions, obtaining the viscosity in Figure 1e.  $R_o$  and  $R_w$  at different surfactant concentrations are used to obtain the Type I ( $\mu E$  + excess oil) – III (middle phase  $\mu E$ ) – Type II ( $\mu E$  + excess water) boundaries of the fish diagram in Figure 1f.

Formulation Engineering 2.0 combines  $\mu E$  properties with end-use conditions to predict formulation performance. Figure 1g uses the Weber number (inertial/interfacial forces), involving mixing power dissipation (end-use condition) and IFT to predict emulsion drop size. Figure 1h starts from the emulsion drop size, the volume fraction of the emulsified phase, the dimensions of the settling unit (end-use conditions), and  $R_d$ , IFT, viscosity and  $E_r$  to predict emulsion stability. Figure 1i predicts the contact angle of oil drops submerged in the aqueous phase considering the surface energy of the solid (end-use condition), IFT and Neumann's equation of state for wettability.

Figure 1h presents the relationship between % oil removed from drill cuttings vs. IFT. The inputs include the soil particle size, mixing power dissipation, initial oil content (end-use conditions), Weber number applied to the emulsification of oil films, and IFT. Please note that the predicted line in Figure 1h is nearly flat from  $\sim 1$  to  $1E-3$  mN/m, meaning that one can reduce the IFT in that region without substantially improving oil removal. This film emulsification model would





**FIG. 1: HLD-NAC predicted properties (Formulation 1.0) and performance (Formulation 2.0) are shown as solid red lines vs. experimental data (black points).** Source: Figures 1c,d,e,g,h,i were adapted from Acosta, E. (2020). Engineering cosmetics using the Net-Average-Curvature (NAC) model. *Curr. Opin. Colloid Interface Sci.* 48, 149–167 with permission from Elsevier. Figure 1j was adapted from Quraishi, S. et al. (2015), Capillary Curves for Ex-situ Washing of Oil-Coated Particles. *J. Surfact Deterg* 18, 811-823 with permission from Wiley. All other figures and accompanying algorithms are included in an upcoming book on Surfactant Formulation Engineering using HLD and NAC.

indicate that one needs to reduce the IFT to about  $1\text{E-}4$  mN/m to observe oil removal (not shown in Figure 1h) from micron-sized clay particles. The same model predicted near-complete bitumen removal (confirmed experimentally) from bitumen-coated sand if the sand particle was large enough, the oil

viscosity low enough, and the mixing power high enough to work with IFTs of  $\sim 1\text{E-}2$  mN/m. HLD-NAC 2.0 predictions like this can tell you how hard your formulation needs to work, and you can decide if it is worth the cost and effort to reach certain performance targets.

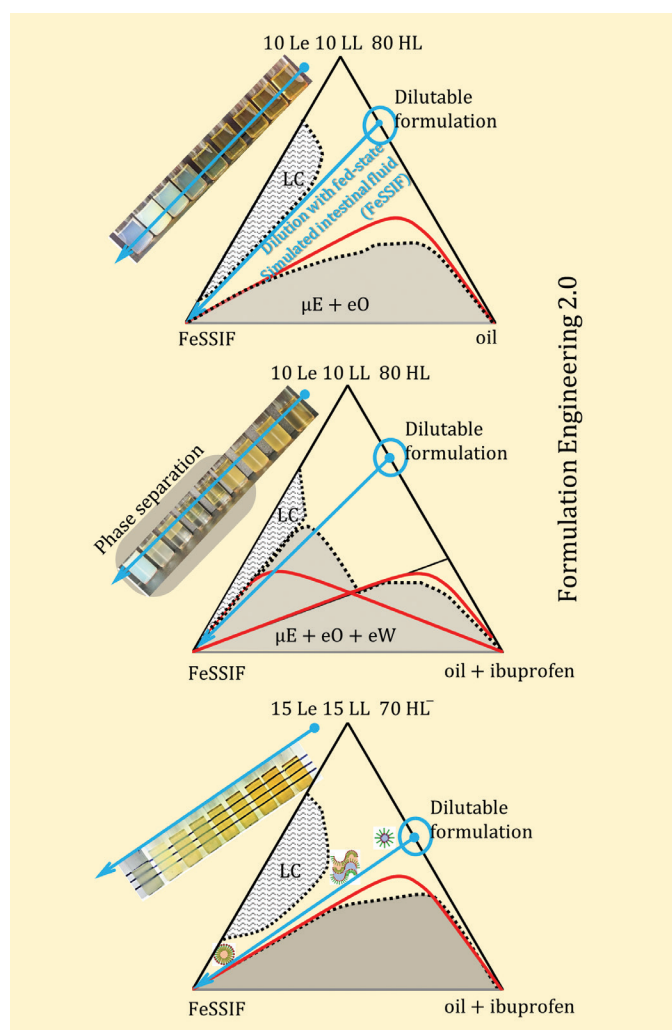
Figure 1i presents a partial connection between detergency and formulation conditions via the work of adhesion ( $IFT \cdot (1 + \cos(\text{contact angle}))$ ). Here the transport equations are yet to be fully developed, but there is a good empirical correlation between detergency and the NAC-calculated IFT and contact angle.

Figure 1h presents an example of the simulation of the concentration of perchloroethylene (PCE, a contaminant found as oil trapped in some aquifers) extracted from a sand-packed column. This PCE work, along with the drill cuttings work were undertaken in the laboratory of Professor David Sabatini of the University of Oklahoma. The PCE work requires a two-part simulation; the first part uses Capillary (shear/interfacial forces) and Bond (Buoyancy/interfacial forces) numbers to find the minimum IFT that would not cause oil mobilization. The second part uses this minimum IFT and NAC to determine the required HLD and Ro, setting the maximum PCE solubilization ( $C_{\text{sat}} = \rho_{\text{PCE}} \cdot R_o \cdot \phi_s / (3 \cdot v_s / a_s)$ ). Considering a mass transfer coefficient ( $K_i$ , the only fitted value) and the PCE solubilized concentration at any position and time,  $C(z,t)$ , one sets a transport equation that generates the solid line. Researchers at Pennsylvania State University, the University of Oklahoma, the University of Texas, and Chevron have incorporated more accurate HLD-NAC variants into their reservoir simulators to track crude oil mobilization and solubilization for enhanced oil recovery applications.

Figure 2 presents ternary phase diagrams (TPD) generated by the HLD-NAC. The calculation algorithm uses modified versions of Ro and Rw that consider the contribution of the surfactant to the volume of the continuous phase. Figure 2a shows a fully-dilutable, pharma-grade formulation developed as a drug delivery platform. However, incorporating ibuprofen (a polar oil) produced a phase change illustrated in Figure 2b. TPD predicted via HLD-NAC led us to identify that hydrophilic linkers with  $\sigma$  of -5 or more negative ( $HL^-$  in Figure 2c) could compensate for the effects of ibuprofen. This prediction was confirmed days later, as shown in Figure 2c, leading to a formulation that improved the oral bioavailability of ibuprofen by more than 3-fold over ibuprofen suspension.

The examples in this article illustrate the connections between HLD, NAC, and process or product performance. Databases, group contribution models, and correlations for HLD and NAC parameters continue to grow. We are approaching the point where we could undertake pre-lab formulations, meaning that we could select candidate surfactants and oils (pre-screened for safety, sustainability, and cost) and use HLD-NAC to predict performance. In practice, making the transition to the pre-lab concept is not easy. We often start new projects with HLD (and our formulation experience) and use NAC when we get in trouble. However, using HLD-NAC in the earlier stages of the research (even if not at a pre-lab stage) is helping us accelerate our work and determine when certain formulation targets are not feasible.

If you want to start your journey into HLD-NAC, new colleagues have indicated that Steven Abbott's website (<https://www.stevenabbott.co.uk/>) and e-book on practical surfactants have served as an effective introduction. The *Journal of Surfactants and Detergents* has numerous research and review articles on HLD, and a few on HLD-NAC, including in a recent



**FIG. 2: Ternary phase diagrams (TPDs) predicted with HLD-NAC for lecithin (Le)-lipophilic linker (LL, glycerol monooleate) – hydrophilic linker (HL, polyglycerol caprylate) produced with ethyl caprate as solvent oil (a), and systems containing dissolved ibuprofen (b, c).** Source: The graphs were adapted from Nouraei, M. *et al.* (2021), HLD-NAC design and evaluation of a fully dilutable lecithin-linker SMEDDS for ibuprofen. *Int. J. Pharm.* 610, 121237 with permission from Elsevier.

special edition. A book project on Surfactant Formulation Engineering using HLD and NAC is currently ongoing and expected to be released in 2023. The book covers a detailed description of the concepts along with academic and industrial example applications (including those in Figures 1 and 2) and a database of HLD and NAC parameters and methods to assess them when unavailable.

Edgar Acosta obtained a bachelor's degree in chemical engineering from Universidad del Zulia (Venezuela, 1995), and M.A.Sc. and Ph.D. degrees in chemical engineering from the University of Oklahoma in 2000 and 2004, respectively. He is currently a professor at the University of Toronto where he leads the Laboratory of Colloids and Formulation Engineering (LCFE) group. His research interests include microemulsion phase behavior, cleaning technologies, drug delivery, cosmetic formulations, lung surfactants, bio-based surfactants, recovery of value-added products from waste, surfactant-based separations, and environmental remediation technologies. He can be reached at [edgar.acosta@utoronto.com](mailto:edgar.acosta@utoronto.com).



# Detecting cancer from inside

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

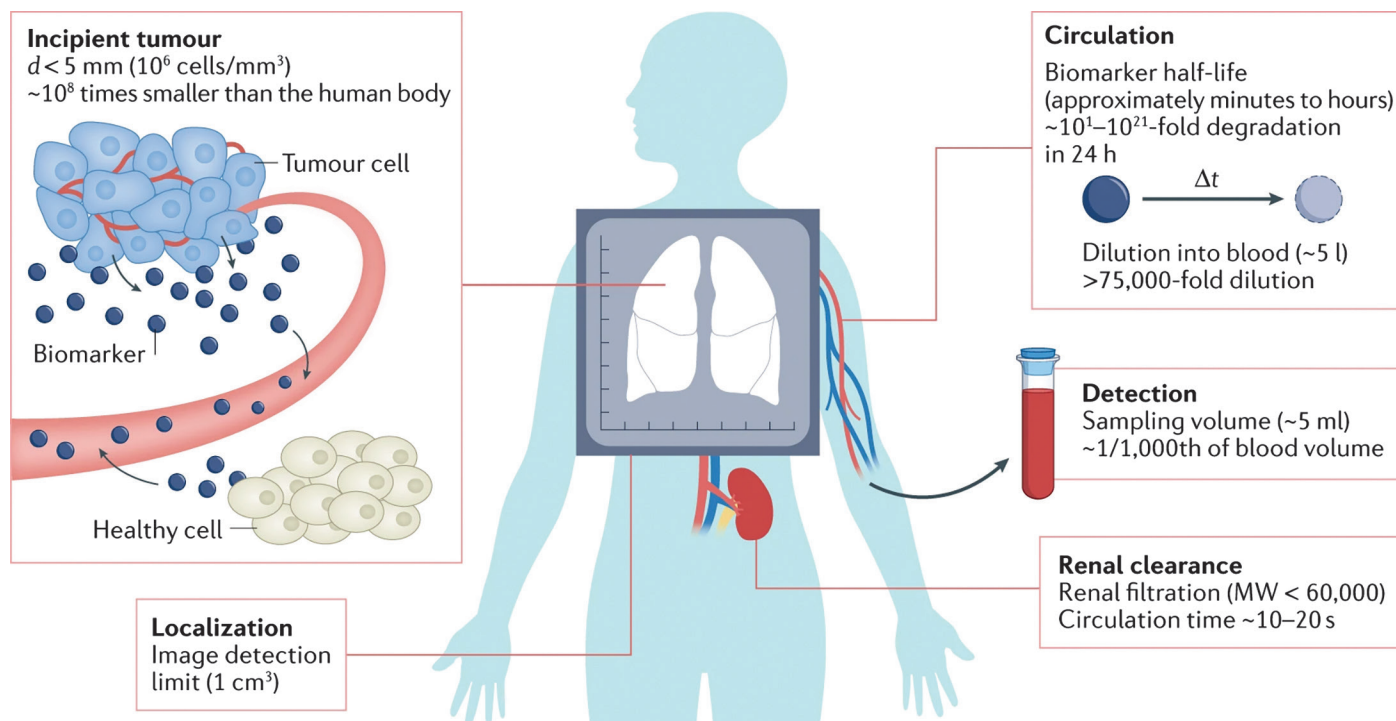
Alexa Tascher

A surprising debate in the medical community is whether or not certain cancer screenings should be carried out universally for asymptomatic patients. Surely, the more information one has about their potential risks, the better, right? However, some argue against tests such as prostate-specific antigen (PSA) screening. (Researchers debate whether there is a significant difference between mortality rates in PSA-tested groups versus non-tested groups.) In particular, false positives can lead to unnecessary biopsies and immune system-weakening treatment for cancers that are absent or indolent (<https://doi.org/10.1056/NEJMs1616281>).

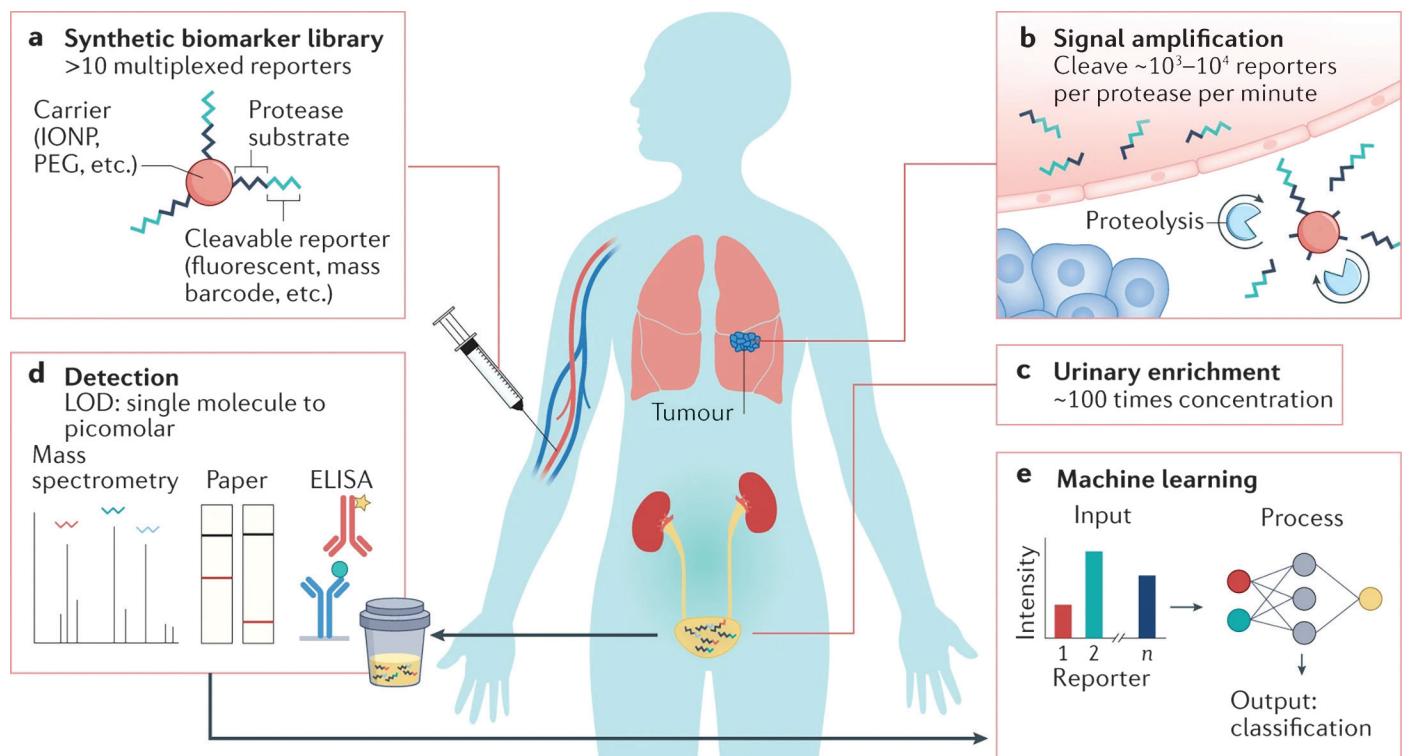
The inefficiency of some asymptomatic cancer screenings, though, cannot lead to simply rejecting all such tests. In fact, quite the opposite is true: detecting tumors at an early stage is often crucial to full and effective treatment, especially in cases of aggressive cancer. However, current methods are not sensitive enough and also do not extend to a wide variety of cancer types. For example, one study analyzing the detec-

tion of mutated DNA released by tumors found that in a typical ten-milliliter blood draw containing four milliliters of plasma, there are on average only six mutated molecules per tube (<https://doi.org/10.1038/s41576-018-0071-5>).

Complicating the matter is tumor heterogeneity: each tumor acts differently, and a molecule known to be a clear marker of a certain type of cancer may not be secreted at a



**FIG. 1. Challenges associated with detecting early-stage tumors.** Source: Kwong, G., et al., *Nat. Rev. Cancer*, 2021



**FIG. 2. Activity-based synthetic biomarkers enrich tumor protease signatures.** Source: Kwong, G., et al., *Nat. Rev. Cancer*, 2021

detectable amount by every tumor. Similarly, each patient's body is different; background "noise" from other organs may obfuscate a positive test result.

To address these unresolved issues, researchers have recently focused on synthetic biomarkers for the detection of cancers. Historically, the administration of outside agents to detect cancer has had strong footing in the medical world—consider the infusion of inulin to measure kidney function—as the body's reaction to these agents can produce more easily detectable signals of wellness and function.

Synthetic biomarkers, though, add a new layer to the diagnostics question. Activity-based synthetic biomarkers include sensor components that, when activated by enzymes in a tumor, kickstart a mechanism which amplifies the number of tumor biomarker molecules.

One main area of interest is protease-activated synthetic biomarkers. There are over 550 proteases encoded in the human genome, and their dysregulation is a common trait of many cancers. For example, the vast majority of cancers lead to the overexpression of matrix metalloproteinases (MMP), which can increase nutrient-rich blood flow to nascent tumors (<https://doi.org/10.1038/sj.leu.2404402>). By examining the activity of proteases like MMP, clinicians can discern between anomalies in regulation, leading to the discovery of the culprit tumor.

The synthetic biomarkers used to detect protease activity are made of peptide substrates conjugated to the surface of an inert carrier (often propylene glycol, or PEG). Very recently, lipid nanoparticles have been used as carriers, specifically in mRNA-based vaccines for COVID-19; such lipid carriers could prove useful in the synthetic biomarker field given their sta-

ble nanostructure and resulting protective nature (<https://doi.org/10.1038/s41578-021-00358-0>). When these biomarkers come into contact with tumor proteases, they are cleaved, and molecular reporters are released into the blood or urine for detection (<https://doi.org/10.1038/nbt.2464>). Because peptide bonds break up irreversibly and proteases do that job thousands of times, these enzymes are incredibly potent molecular amplifiers. A single tumor protease can churn out reporter molecules in quantities that are orders of magnitudes higher than any other biomarker (<https://doi.org/10.1146/annurev-cancerbio-030617-050549>).

Additionally, these methods harness specific aspects of the human body to increase the number of cancer-indicating molecules to a detectable level in biofluids. For example, patients can avoid the discomfort of clearing surface-conjugated peptides as waste through the kidneys if a large enough biomarker carrier is used instead (<https://doi.org/10.1038/nbt1340>). The biomarkers will passively travel in the blood to potential tumor sites.

Since proteases are usually multifunctional and capable of cleaving a wide range of substrate peptide sequences, a single sensor variety is not usually enough to definitively detect a cancer. Many different proteases in the body that are not acting as a result of mutation may be able to interact with a biomarker the way a tumor protease does, so a larger library of sensor molecules is needed to ensure high specificity in tumor detection.

To detect each specific type of cancer, the most effective route can be a compilation of a "cocktail" of synthetic biomarkers, each individually labeled with a unique molecular barcode. There are many methods for stamping each biomarker,



including enriching reporters with stable isotopes like carbon-13, labeling reporters with unique DNA sequences, using ligand-encoded reporters with antibody-detectable small molecules, and including volatile organic compounds which are emitted as gases after cleavage by a protease (<https://doi.org/10.1073/pnas.1805337115>).

Such densely multiplexed cocktails of sensors are not only useful for increasing the specificity of a cancer screening; they can also provide invaluable information regarding the nature of a detected tumor. By using machine learning to analyze biomarker data, extremely accurate predictions can be made about the stage, speed of progression, and benignity of tumors (<https://doi.org/10.1038/s41568-021-00389-3>).

In one study, a cocktail of 14 different sensors using an eight-arm PEG carrier was delivered to mice in order to test for early-stage lung tumors. By using a type of machine learning called a random forest classifier, Kirkpatrick *et al.* were able to predict lung cancer progression and the presence of distinguished lung cancer versus benign lung cancer with very high accuracy (<https://doi.org/10.1126/scitranslmed.aaw0262>). The AUROC (a variable quantifying the accurate performance of machine learning systems) values were 0.90 and 0.97, respectively.

Although the budding field of synthetic biomarkers shows great promise for improving our current methods of screening for cancer, many challenges stand in the way of its implementation. Primarily, the separation of legitimate indications of tumors from biological background noise is paramount. Many

activity-based synthetic biomarkers studied thus far were not selected against background arising from circulating blood, so further research is needed in order for screening strategies to be developed that account for naturally-occurring activity from healthy bodily enzymes.

Additionally, there needs to be further characterization of all early-stage cancers. Most immortalized cancer cell lines used for study today were derived from patients with advanced metastatic cancers. Hence, the mechanisms and biomarkers present in early-stage tumors may not be fully represented.

It has been less than a decade since the first clinical trials using synthetic biomarkers in humans were conducted and questions of scale remain (<https://doi.org/10.1245/s10434-017-5991-3>). When moving from a mouse model to a human model, although most human proteases mirror mouse proteases, physiological differences between the species—like varying kidney activity—make allometric scaling difficult.

Lastly, the specific location of a detected tumor can be difficult to discern. When a biomarker carrier travels throughout the entire body via the blood and leaves reporter molecules in the urine, the location of dysregulated tumor proteases is not immediately clear. The answer to this problem may lie in the machine learning systems that can analyze huge sets of data from biomarker cocktails and predict tumor details.

While synthetic biomarkers make up an exciting new field with ample room for innovation, a great deal of research is needed before these concepts can be put into practice for better cancer screening.



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# Approval of an extraction agent with a 100% certified plant origin

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

**Laurence Jacques**

The European Food Safety Authority (EFSA) just approved the use of a new biobased solvent for food extraction, paving the way for the substitution of the petrochemical solvents widely used for the food and feed production.

On March 14th, 2022, The Minafin Group eagerly announced that the EFSA has released a positive opinion for the use of methyloxolane as extraction agent for food processing. Thus, the use of methyloxolane under the approved production conditions has been identified as safe for consumers and the environment. We see this as a positive step for the safety and sustainability of the food production in Europe. By unlocking the opportunity to substitute petrochemicals for a plant-based extraction agent in food processes, petroleum residues in the whole food chain can be eliminated.

This technology brings to the food and feed market a 100% renewable and safe extraction solution for oils, plant proteins and natural ingredients. This breakthrough and award-winning innovation is biobased, safer for human health, and has a ten times lower CO<sub>2</sub> impact than the currently used petrochemical solvents.

## HOW DID IT START?

The method for producing the molecule, methyloxolane, was developed in the 2000s. Our product has been used in the pharmaceutical industry since 2007. However, the study of its application as an extraction agent began in 2012. At the time a thesis conducted in France, named Anne-Gaelle Sicaire, was part of a collaboration between a laboratory in Bordeaux, the Institut des Corps Gras and the Eco-Extraction laboratory of the University of Avignon. Sicaire showed that among the 10 bio-solvents that had been studied in comparison with hexane



(such as ethanol, ethyl acetate, and limonene) methyloxolane was the only one that produced equivalent yields. In 2017, we verified that the extraction worked on a wide variety of seeds and substrates. We invested in additional studies to determine that our solution was innocuous on humans and animals.

It would take us 10 years, from 2012 to 2022, to develop the extraction application. Finally, we filed our application for food approval in December 2019 with the European Commission. With the positive opinion in March, it will now take 6 months to have the European law updated and our product fully approved for food.

## PERMITTED EXTRACTION AGENTS IN EUROPE

Twenty solvents are currently authorized in the European Parliament's Directive 2009/32/CE for processing foodstuff (Table 1). Most of these chemicals are derived from petroleum. Methyloxolane is the only extraction agent permitted in food applications that is 100% certified carbon from plant origin, a first since the Directive was first issued in 2009. Except for the addition of dimethyl ether (a dossier reviewed by EFSA in 2015), the toxicological database of approved extraction agents has



**TABLE 1. The 20 solvents which are currently approved for use in the Directive 2009/32/CE.**

Name	Origin	At 20°C	Comments	Note in the 2009/32/CE
Propane	Petrochemical	Gas		
Butane	Petrochemical	Gas		
Ethyl acetate	Petrochemical or biobased	Liquid		
Ethanol	Petrochemical or biobased	Liquid		
Carbon dioxide	Petrochemical	Gas	Greenhouse Gas	
Acetone	Petrochemical	Liquid		The use of acetone in the refining of olive-pomace oil is forbidden.
Nitrous oxide	Mineral or biobased	Gas	Greenhouse Gas	
Hexane	Petrochemical	Liquid		The term 'hexane' means a commercial product consisting essentially of acyclic saturated hydrocarbons containing six carbon atoms and distilling between 64°C and 70°C. The combined use of hexane and ethyl methyl ketone is forbidden.
Methyl acetate	Petrochemical or biobased	Liquid		
Ethyl methyl ketone	Petrochemical	Liquid		The level of n-hexane in this solvent should not exceed 50 mg/kg. The combined use of hexane and ethyl methyl ketone is forbidden.
Dimethyl ether	Petrochemical	Gas		
Dichloromethane	Petrochemical	Liquid		
Methanol	Petrochemical	Liquid		
2-propanol	Petrochemical	Liquid		
Diethyl ether	Petrochemical	Gas		
Cyclohexane	Petrochemical	Liquid		
1-butanol	Petrochemical	Liquid		
2-butanol	Petrochemical	Liquid		
1-propanol		Liquid		
1,1,1,2-tetrafluoroethane		Gas		
Methyloxolane	Biobased only	Liquid		

not been reviewed or updated by EFSA since the Directive was issued in 2009. We hope that this review will occur soon as the review of the food additives authorized prior to 2009 has just been completed. We think it is important for public authorities to regularly ask the manufacturers of the substances used in food processing to update their toxicological database and bibliography to consider the latest inputs of science as is regularly done for pesticides, and food and feed additives.

## WHAT IS METHYLOXOLANE?

Methyloxolane is a bio-based product upcycled from sugar cane bagasse, the fibrous by-product remaining after the sugar has been extracted from the cane. It can be fully mixed with lipids. It is particularly well-adapted for vegetable oil extraction and other defatting processes. This property is rare for a bio-

based solvent. Ethanol for instance is not naturally miscible with lipids.

## HOW METHYLOXOLANE IMPROVE FOOD PROCESSING?

Oil and protein-rich food make up half of our diet. We consume an average of 130 grams of lipids and 50 to 70 grams of protein every day. Oil in food production is essential and present in many everyday foods, spreads, chocolate bars, chips, sweet and savory cakes, meats, cold meats, and ready meals.

Today, the production of vegetable oil and protein-rich plant products relies on hexane extraction, a technique which was optimized in the 1950s in large industrial processing facilities. Its use results in the presence of hexane residues in our food. However, with one exception, hexane is forbidden in organic food.



Hexane is indispensable today. About 20% of vegetable oil production depends on hexane-based solvent extraction. The production of long-life, high-protein feed depends on solvent-aided extraction of the oil. Besides the extraction of major oilseed crops such as soya bean, sunflower, rapeseed, cotton seed, and palm kernels, hexane is also used for the production of natural extracts such as hop extract, annatto extract, and omega 3 rich oils.

The use of large quantities of hexane has a negative impact on the environment, because of the large amount of emissions into the atmosphere. Each year, food extraction plants release 700,000 tons of hexane in the air, which contributes air pollution (hexane is classified as hazardous pollutant in the US) and to the transfer of non-renewable carbon to the atmosphere. The replacement of hexane using methyloxolane will significantly reduce the environmental impact of these parts of the food and feed industries.

## WHAT ARE THE MAIN USES IN FOOD APPLICATIONS?

Methyloxolane dissolves lipids and so the main application is in the processing of oilseeds and grains. In addition to the extraction of valuable lipophilic natural pigments, flavors, fragrances, and bioactives from a wide range of natural sources. The Minafin Group is currently scaling-up production of EcoXtract®, an innovative and patented extraction system using methyloxolane in such food-based extraction applications.

## WHAT ARE THE MAIN BENEFITS OF METHYLOXOLANE FROM A FOOD SAFETY PERSPECTIVE?

Consumers are generally unaware that hydrocarbon solvents are used in food processing as they are categorized as “processing aids” and are exempt from labelling. However, residues are unavoidable and widespread, particularly in foods that contain vegetable oils or plant protein concentrates. At Minafin Group, we advocate for transparent labelling about the use of safe extraction agents in food applications. EFSA’s positive opinion on methyloxolane relates to a full application dossier including state-of-the-art scientific studies performed under OECD standards. The opinion confirms the safety of the product for food applications. These studies combined with a 15-year background of methyloxolane use to manufacture pharmaceutical actives ensures a high level of protection for the consumer. In addition, methyloxolane is a naturally occurring molecule found in our close environment. For instance, it is naturally produced by a yeast living on human skin, known as *Malassezia furfur*. It was also identified in mother’s milk samples in 1982, long before any commercial production of product started at the beginning of the 21st century.

## ORGANIC LABELING?

The EFSA positive opinion will support the methyloxolane dossier for an organic certification. The use of the product is already approved by COSMOS label to produce organic cos-



metic ingredients. Minafin Group will apply for this recognition in the coming weeks for food and feed applications.

## WHAT ARE THE MAIN BENEFITS OF METHYLOXOLANE FROM AN ENVIRONMENTAL PERSPECTIVE?

As a science-led company dedicated to developing and commercializing breakthrough bio-based solutions, we are committed to building a fossil carbon-free world through the replacement of petrochemicals used in the food value chain. Reducing our dependence on petroleum derivatives is our day-to-day mission. Due to its plant origin, the carbon emissions from the production of methyloxolane is less than 200 g CO<sub>2</sub> per kg, 90% less than an average petrochemical solvent.

Because it is upcycled from agricultural byproducts, the production of methyloxolane does not require additional land. Land use being the foremost direct cause of biodiversity loss with the largest relative global impact. The superior performance of EcoXtract® solution as a lipophilic solvent, enables high extraction yields which maximizes oil production from a given quantity of oilseed and reduces the protein food waste.

## WHAT IS THE IMPACT ON THE GLOBAL FOOD SUPPLY?

All cumulated food waste represents roughly one-third of the food production in the world. In the oilseeds industry, food waste represents 20% of the total production. We must act upon the current global situation where one out of nine people are hungry or undernourished and 2,37 billion people did not have access to enough safe and nutritious food in 2020.

According to Wageningen University, the limited application of oilseed residues, like rapeseed press cake, results in 26 Megatons of global protein losses annually. Implementation of this protein source is limited because conventional oilseed processing by mechanical press is optimized for oil extraction only and often reduces protein functionality.

Using methyloxolane in an extraction process, results in a 33% increase of the oil yield compared with mechanical processing alone and renders a high quality well-defatted long-lasting protein-rich residue which can be used for feed or for food application. Reducing food losses and waste is essential in a world where the number of people affected by hunger has been slowly on the rise since 2014, and multiple tons of edible food are lost and/or wasted every day. Globally, around 14% of food produced is lost between harvest and retail, while an estimated 17% of total global food production is wasted (11% in households, 5% in the food service and 2% in retail).



Methyloxolane offers a bio-based option to food ingredient producers committed to limiting the use of processing aids derived from petroleum sources, particularly those that use vegetable oil and high-protein co-products of oilseeds. Therefore, methyloxolane eliminates petroleum residues across the food chain.

The European approval of this extraction agent is a major boost to the safety and sustainability of food processing in Europe. Thanks to EFSA's positive opinion, issued on March 15th this year, methyloxolane will be admitted soon to the list of permitted food extraction agents described in the Directive 2009/32/CE. You can access the full EFSA's opinion here: <https://efsa.onlinelibrary.wiley.com/doi/full/10.2903/j.efsa.2022.7138>.

## WHAT'S NEXT?

We have prepared the patent filing for the US, Canada and Australia; other countries will follow. In Europe, the Directive 2009/32/EC be amended by the end of the year. We have already been working with our partners and customers to prepare the adaptation of their process to methyloxolane in their industrial plants. Our aim is to bring our extraction solution to the market to allow ingredient producers to offer a safer and more sustainable products to their customers. We will also apply in the coming months for recognition for use in organic processing.

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*Laurence Jacques is the managing director of the EcoXtract® program. This article is an excerpt from an interview with her. For more information contact [gabriel.dufour@ecoxtract.com](mailto:gabriel.dufour@ecoxtract.com).*



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# Meet Liyun Ye

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



Liyun Ye on a hiking/biking holiday in Île de Groix, France.

## PROFESSIONAL

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

I start my day with a short exercise session, a big breakfast, and a coffee. Usually, there are a few Zoom meetings, some spontaneous discussions with colleagues, and some lab work. I chat with my parents in Lianyungang, Jiangsu Province, China, in the evening, usually during my drive home or during a walk in the park.

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

Getting to work with everyone on the research and development team. They are all experts in their own areas of specialization, and they complement and support each other. They are also very fun people, making me want to go to work and see them.

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

I wanted to be a biologist or naturalist. Books about Charles Darwin, his theories of evolution, and all the amazing creatures he described had a major impact on me.

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

The burgeoning cellular agriculture area has a great mission that matches well with my personal vision and goals.

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

Sean O'Keefe, my master's and Ph.D. mentor at Virginia Tech, has had the most influence on my life. I would not be here

## Fast facts

<b>Name</b>	Liyun Ye
<b>Joined AOCS</b>	2015
<b>Education</b>	Ph.D. in 2015 from Virginia Tech (Blacksburg, Virginia, USA)
<b>Job title</b>	Senior scientist
<b>Employer</b>	Finless Foods, Inc. (Emeryville, California, USA)
<b>Current AOCS role</b>	Chaired three sessions on lipid oxidation and quality in 2021; organized one session in 2022

without him. He is always supportive and inspiring to me any place, any time. He is an amazing person and wonderful mentor who is generous, forgiving, fair, knowledgeable, and light-hearted. Without him, I would not be where I am in either my personal life or career life.

## PERSONAL

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

I like the Les Mills Bodypump workout.

*What is the most impressive thing you know how to do?*

I know how to ride a motorcycle.

*What skill would you like to master?*

Communication skills, to be able to express myself better.

*What are some small things that make your day better?*

Grabbing a coffee or having a walk with a colleague as a work break; spontaneous new adventures (e.g., suddenly decide to explore a new dance bar with colleagues).

*What are you looking forward to in the coming months or years?*

A good recovery from the pandemic with a safe opening of borders so that families and friends can reunite, in addition to increased growth of the cellular agriculture sector as well as public awareness of the impact humans have on the earth.



# PATENTS

## Caramel, confection comprising the caramel and method of making the confection

Rodrigues, T., *et al.*, Mars Inc, US11134702, October 5, 2021

A caramel is provided comprising a solid plant fat and an amount of water less than 10 weight percent. The caramel exhibits a chewy texture when consumed but is yet depositable. The caramel is thus advantageously used in a confection. In addition to the caramel, the confection may comprise a crisp component. Methods of making the confection are also provided.

## Lipid compositions with high DHA content

Hallaraker, Hogne, *et al.*, Arctic Nutrition A/S, US11135230, October 5, 2021

The invention provides lipid compositions comprising phospholipids having a high docosahexaenoic acid (DHA) content, which compositions are preferably extracted from natural sources. The lipid compositions are excellent sources of highly bioavailable DHA and they can be used in oral delivery vehicles, dietary supplements, functional foods, and the like.

## Solvent extraction of oil from distillers dried grains and methods of using extraction products

Bruinsma, K., *et al.*, Novita Nutrition LLC, US11136508, October 5, 2021

A process for extraction of crude oil from distillers dried grain solubles and/or distillers dried grains and producing corn distillers meal that may be used as a livestock supplement is disclosed. For example, the corn distillers meal may be used as a crude protein supplement for use in a livestock feed diet or a poultry feed diet. The solvent extracted crude oil may be suitable for oleochemical processing for personal care and home care products, biodiesel production, and/or renewable diesel production from hydro-treating the extracted oil to make green diesel fuel.

## Lubricating oil compositions

Hartley, J.P., *et al.*, Infineum Ltd, US11136523, October 5, 2021

A lubricating oil composition which contains, or is made by admixing: (A) an oil of lubricating viscosity, in a major amount; (B) at least one oil-soluble or oil-dispersible sulfurized fatty acid ester, as an additive in an amount providing the lubricating oil composition with greater than or equal to 0.02 mass percent sulfur; and, (C)

at least one oil-soluble or oil-dispersible alkali metal or alkaline earth metal salicylate detergent, as an additive in an effective minor amount, providing greater than or equal to 5 mmol of salicylate soap per kilogram of the lubricating oil composition, wherein the lubricating oil composition does not contain a phosphorus ester additive.

## Methods of making a squeezable spread containing butter

Paddock, J.K.H. and Hohn, A., Land O'Lakes, US11140910, October 12, 2021

Butter spreads include at least butter, canola oil, palm oil, cream, and buttermilk powder. The spreads are squeezable from a package and maintain their consistency over repeated package handling and repeated cycling between refrigeration and ambient temperatures.

## Phospholipid concentrate manufacturing method

Yuasa, K., *et al.*, Marudai Food Co Ltd, US11142537, October 12, 2021

This invention provides a technique that is capable of suppressing variation in the amount of phospholipids obtained in each operation when a phospholipid concentrate is obtained by subjecting an ethanol extract concentrate of livestock or poultry tissue to a degumming step and collecting gum. More specifically, the invention provides a method for producing a phospholipid concentrate from livestock or poultry tissue, comprising step (A) of mixing an ethanol extract concentrate of livestock or poultry tissue with water, the water being in an amount of less than 7 parts by mass per 100 parts by mass of the concentrate, and step (B) of centrifuging the obtained liquid mixture at 2 degrees centigrade or lower.

## Emulsions

McKee, M.A., *et al.*, Givaudan S/A, US11147280, October 19, 2021

A topical flavor composition comprising at least one hydrophilic flavor compound and at least one lipophilic flavor compound, the flavor composition being a water-in-oil emulsion having a continuous non-aqueous phase and a disperse aqueous phase, the at least one lipophilic flavor compound being dissolved or dispersed in the continuous phase and the at least one hydrophilic flavor compound dissolved or dispersed in the disperse phase. The composition is useful for topical application to a wide variety of comestible products and allows highly versatile flavoring possibilities.

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](mailto:scott.bloomer@aocs.org).



## 2022–2023 Student Common Interest Group Leaders

Thank you to our student leadership for all they do to support AOCS throughout the year.



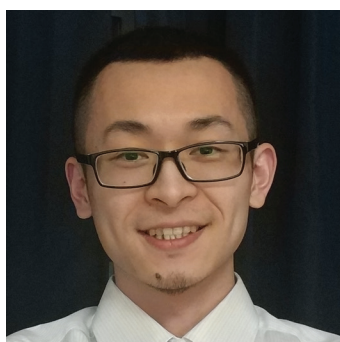
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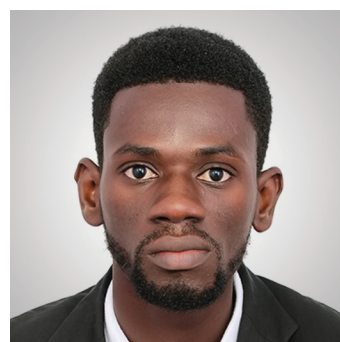
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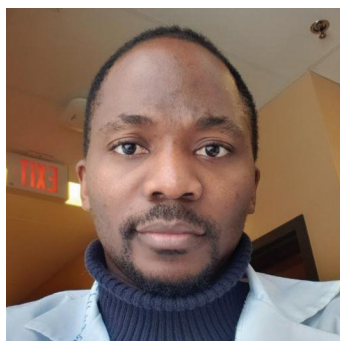
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Massachusetts Amherst, USA

Membership in the Student CIG provides numerous opportunities for you to get further involved in AOCS.  
Contact Bill Stanton at [william.stanton@aocs.org](mailto:william.stanton@aocs.org) for information on how to get involved with this student-led group.



# Celebrating 25 years of publishing the *Journal of Surfactants and Detergents*

This year's AOCS Annual Meeting and Expo pulsed with a unique energy. After two years of seeing our colleagues through screens over shoddy Wi-Fi, being in-person together felt exuberant. Despite ending a few hours early, as COVID raised its ugly spikes, the three days of informative technical sessions, networking receptions by the pool, and a continual supply of chocolate (thanks to Utah State University's Auggie Chocolate Factory) were rejuvenating.

The Monday morning session held particular importance as an opportunity to acknowledge the 25 years that the *Journal of Surfactants and Detergents* has been an invaluable scientific resource. In the 1990s, industrial researchers working in the field of surfactants had no place to submit their findings for publication. Michael Cox organized a team consisting of Mark Nace, Lisa Quencer, and Arno Cahn to establish the journal for publishing peer-reviewed articles on the science and application of surfactants and detergents. This past May, George Smith compiled a virtual issue of previously published articles describing the hydrophilic-lipophilic deviation method, showing the theory's evolution and application over the journal's lifespan ([https://aocs.onlinelibrary.wiley.com/doi/toc/10.1002/\(ISSN\)1558-9293.HLD](https://aocs.onlinelibrary.wiley.com/doi/toc/10.1002/(ISSN)1558-9293.HLD)). The 25<sup>th</sup> Anniversary session further highlighted innovative surfactants and detergents research.

Doug Hayes, *JSD* editor-in-chief and session organizer, gathered a diverse group of speakers. Attendees heard talks on viscoelastic surfactants, sustainable emulsions for cosmetics and lubrication, how additives affect micelle structure, and a simplified HLDN equation. If you were unable to attend this year's annual meeting, AOCS members receive extended access to on-demand content through December 31, 2022. Use your AOCS credentials to login to the platform and watch talks from the 25th Anniversary session (<https://tinyurl.com/AM22OnDemand>).





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| <b>PRO</b> Processing                     | <b>PCP</b> Protein and Co-Products     |
| <b>S&amp;D</b> Surfactants and Detergents |  |

## Original Articles

### **BIO** **PCP** **IOP** Co-Utilization of amino acid-rich wastes and glycerol for microbial lipid production

Kamal, R., *et al.*, *Biofuel Bioprod Biorefin*, 16, 1, 142-154, 2022.  
<https://doi.org/10.1002/bbb.2265>

Microbial lipids are considered to be potential raw materials for biodiesel production. However, the costs of such a process remain high due to the use of expensive feedstocks for microbial lipid production. The meat industry and biodiesel refineries generate amino acid (AA) wastes and crude glycerol, respectively, which could be explored as relatively cheap feedstocks for oleaginous yeasts. Unfortunately, AA wastes have a high nitrogen content and crude glycerol contains several impurities, discouraging microbial lipid production. Here, glycerol was co-used with AA, or with AA blends with compositional profiles matching those of different meats, so that the media had favorable carbon-to-nitrogen (C/N) ratios to promote AA conversion into lipids using the oleaginous yeast *Cryptococcus curvatus* ATCC 20509 under two-stage culture conditions. Co-utilization of glycerol and AA resulted in improved lipid production. The highest lipid yield of 20.9 g 100 g<sup>-1</sup> was achieved with lysine and glycerol, where the lipid yield was 13.9 g 100 g<sup>-1</sup>, using an AA blend of similar composition to sheep viscera and glycerol. The lipid products comprised over 93% of 16 and 18 carbon-containing fatty acids, showing high potential for biodiesel production. The co-use of glycerol with AA improved AA conversion into lipids while decreasing the inhibitory effects at relatively high AA concentrations. The utilization of crude glycerol and AA-rich wastes from the meat industry could help both the development of biorefineries and waste minimization.

### **PCP** **IOP** **PRO** Sustainable production of jojobyl alcohols and cell viability study

Acherki, H., *et al.*, *Biofuel Bioprod Biorefin*, 16, 2, 451-459, 2022.  
<https://doi.org/10.1002/bbb.2292>

An innovative biorefinery approach has been applied to evaluate jojoba oil (JO) in high value-added products. It consists of enzymatic transesterification using short (1-propanol, 1-butanol), medium linear (1-octanol), and branched (2-ethylhexanol) chain alcohols. The proposed biorefinery approach uses an integrated process for the production of jojobyl alcohol (JA) mixtures (11-eicosenol, 13-docosenol and 15-tetracosenol) as products that can be used in pharmaceutical applications. The remaining fraction of fatty acid alkyl esters (FAAE) has a wide range of industrial uses and could be used as a promising alternative to conventional fuels, as it complies with EN14214, the European Biodiesel Norm. The separation of JA from FAAE was carried out by crystallization using a binary mixture of hexane and diethyl ether. After the separation step, the main properties of FAAE were determined. The results showed that the cold flow properties and oxidation stability of the FAAE fraction, obtained as co-product during the process, have been improved with respect to transesterified JO. The use of 2-ethylhexanol as an alcohol in the transesterification reaction increases the amount of the most valuable fraction containing JAs. *In vitro* cell viability was measured in HEK293T cells using the tetrazolium dye reduction (MTT) assay. The results showed that this oily liquid mixture of JA components (*cis*-11-eicosenol, *cis*-13-docosenol, and *cis*-15-tetracosenol) had a cytotoxic effect at concentrations of 10 and 100 µmol L<sup>-1</sup> and no cytotoxic activity at 1 µmol L<sup>-1</sup>. The concentration of 1 µmol L<sup>-1</sup> does not, therefore, modify the cell viability, does not produce toxic effects in the tested cells and could be used as a therapeutic compound.

### **IOP** **PRO** Dual-stage biorefinery to convert spentwash hydrolysate into oleochemicals using *Trichosporon cutaneum* and *Yarrowia lipolytica*

Sreeharsha, R.V., *et al.*, *Bioresour. Technol.*, 354, 127146, 2022.  
<https://doi.org/10.1016/j.biortech.2022.127146>

Yeast lipids from low-cost renewable feedstock are valuable resources for oleochemicals thus enabling circular chemistry. Current study focuses on lipid and volatile fatty acid (VFA) production through dual-stage fermentation of spentwash in a biorefinery framework with *Trichosporon cutaneum* (*Tc*) and *Yarrowia lipolytica* (*Yl*). During cell proliferation phase, *Tc* and *Yl* accumulated 2.9 and 2.5 g/L of dry biomass respectively in acid-hydrolysed spentwash (AHSW) and produced 16 and 5.5 g/L of total VFA respectively. Lipid yields (29.8%) and lipid titres (0.89 g/L) were higher in *Tc*/AHSW, when compared to *Yl* indicating the efficacy of *Tc* in spentwash bioremediation. Lipid accumulation was enhanced to 35% in *Tc*/AHSW, in presence of 0.05% NH<sub>4</sub>Cl due to oxidative stress of ammonium ions. Analysis of fatty acid composition revealed the presence of higher oleic acid, which is ideal for biodiesel production. The results demonstrate a sustainable biorefinery model for bioremediation of spentwash and its value addition.

## PCP BIO Sesquiterpene synthase engineering and targeted engineering of $\alpha$ -santalene overproduction in *Escherichia coli*

Zhang, J., et al., *J. Agric. Food Chem.*, 70, 17, 5377-5385, 2022.  
<https://doi.org/10.1021/acs.jafc.2c00754>

As a natural sesquiterpene compound with numerous biological activities,  $\alpha$ -santalene has extensive applications in the cosmetic and pharmaceutical industries. Although several  $\alpha$ -santalene-producing microbial strains have been constructed, low productivity still hampers large-scale fermentation. Herein, we present a case of engineered sesquiterpene biosynthesis where the insufficient downstream pathway capacity limited high-level  $\alpha$ -santalene production in *Escherichia coli*. The initial strain was constructed, and it produced 6.4 mg/L  $\alpha$ -santalene. To increase  $\alpha$ -santalene biosynthesis, we amplified the flux toward farnesyl diphosphate (FPP) precursor by screening and choosing the right FPP synthase and reprogrammed the rate-limiting downstream pathway by generating mutations in santalene synthase (*Clausena lansium*; CISS). Santalene synthase was engineered by site-directed mutagenesis, resulting in the improved soluble expression of CISS and an  $\alpha$ -santalene titer of 887.5 mg/L; the  $\alpha$ -santalene titer reached 1078.8 mg/L after adding a fusion tag to CISS. The most productive pathway, which included combining precursor flux amplification and mutant synthases, conferred an approximate 169-fold increase in  $\alpha$ -santalene levels. Maximum titers of 1272 and 2916 mg/L were achieved under shake flask and fed-batch fermentation, respectively, and were among the highest levels reported using *E. coli* as the host.

## PCP BIO Supplementation with *rac*-GR24 facilitates the accumulation of biomass and astaxanthin in two successive stages of *Haematococcus pluvialis* cultivation

Wang, X., et al., *J. Agric. Food Chem.*, 70, 15, 4677-4689, 2022.  
<https://doi.org/10.1021/acs.jafc.2c00479>

The unicellular freshwater green alga *Haematococcus pluvialis* has attracted much research attention due to its biosynthetic ability for large amounts of astaxanthin, a blood-red ketocarotenoid that is used in cosmetics, nutraceuticals, and pharmaceuticals. Recently, numerous studies have investigated the functions of natural astaxanthin; however, the high cost of the production of astaxanthin from *H. pluvialis* cultures restricts its commercial viability. There is an urgent need to fulfill commercial demands by increasing astaxanthin accumulation from *H. pluvialis* cultures. In this study, we discovered that treatment of *H. pluvialis* cultures at the beginning of the macrozooid stage (day 0) with 1  $\mu$ M *rac*-GR24, a synthetic analogue of strigolactones (a class of phytohormones), led to significant increases in biomass [up to a maximum dry cell weight (DCW) of 0.53 g/L] during the macrozooid stage and astaxanthin (from 0.63 to 5.32% of DCW) during

the hematocyst stage. We elucidated that this enhancement of biomass accumulation during the macrozooid stage by *rac*-GR24 is due to its increasing CO<sub>2</sub> utilization efficiency in photosynthesis and carbohydrate biosynthesis. We also found that *rac*-GR24 stimulated the overproduction of nicotinamide adenine dinucleotide phosphate (NADPH) and antioxidant enzymes in *H. pluvialis* cultures, which alleviated the oxidative damage caused by reactive oxygen species generated during the hematocyst stage due to the exhaustion of nitrogen supplies. Moreover, *rac*-GR24 treatment of *H. pluvialis* synergistically altered the activity of the pathways of fatty acid biosynthesis and astaxanthin esterification, which resulted in larger amounts of astaxanthin being generated by *rac*-GR24-treated cultures than by controls. In summary, we have developed a feasible and economic *rac*-GR24-assisted strategy that increases the amounts of biomass and astaxanthin generated by *H. pluvialis* cultures and have provided novel insights into the mechanistic roles of *rac*-GR24 to achieve these effects.

## EAT LOQ A novel strategy to simultaneously enhance bioaccessible lipids and antioxidants in hetero/mixotrophic *Chlorella vulgaris* as functional ingredient

Canelli, G., et al., *Bioresour. Technol.*, 347, 126744, 2022.  
<https://doi.org/10.1016/j.biortech.2022.126744>

Microalgae are a promising source of polyunsaturated fatty acids as well as bioactive antioxidant compounds such as carotenoids, phenolics and tocopherols. However, the accumulation of these biomolecules is often promoted by conflicting growth conditions. In this study, a phased bioprocessing strategy was developed to simultaneously enhance the lipid and antioxidant amounts by tailoring nitrogen content in the cultivation medium and applying light stress. This approach increased the overall contents of total fatty acids, carotenoids, phenolics, and  $\alpha$ -tocopherol in *Chlorella vulgaris* by 2.2-, 2.2-, 1.5-, and 2.1-fold, respectively. Additionally, the bioaccessibility of the lipids and bioactives from the obtained biomasses improved after pulsed electric field (5  $\mu$ s, 20 kV cm<sup>-1</sup>, 31.8 kJ kg<sup>-1</sup> sus) treatment (up to +12%) and high-pressure homogenization (100 MPa, 5–6 passes) (+41–76%). This work represents a step towards the generation of more efficient algae biorefineries, thus expanding the alternative resources available for essential nutrients.

## H&N Hypoglycaemic and anti-ageing activities of green alga *Ulva lactuca* polysaccharide via gut microbiota in ageing-associated diabetic mice

Chen, Y., et al., *Int. J. Biol. Macromol.*, 212, 97-110, 2022.  
<https://doi.org/10.1016/j.ijbiomac.2022.05.109>

Ageing-related type 2 diabetes is a significant public health problem. Particularly, the number of cases and fatality rates of ageing-associated diabetes increase with population ageing. This



study aimed to investigate the structural characterisation of *Ulp* lactuca polysaccharide (ULP) and the hypoglycaemic effect on ageing-associated diabetic mice using gut microbiota variation. Sugar residuals analysis showed that the purified ULP (ULP-1) comprised  $\beta$ -D-Xylp-(1 $\rightarrow$ 3)- $\beta$ -D-Arap-(1 $\rightarrow$ 6)- $\beta$ -D-Galp-(1 $\rightarrow$ 6)- $\beta$ -D-Glcp linked to  $[\rightarrow\alpha$ -L-Rhap-(1 $\rightarrow$ 4)- $\beta$ -D-GlcpA $\rightarrow$ ] $_n$  and  $\alpha$ -D-Manp-(1 $\rightarrow$ 4)- $\alpha$ -L-Rhap(2SO $_3^-$ )-(1 $\rightarrow$ 2)- $\alpha$ -L-Rhap(4SO $_3^-$ )-(1 $\rightarrow$ 2)- $\alpha$ -L-Arap-(1 $\rightarrow$ 2)- $\alpha$ -L-Rhap-(1 $\rightarrow$  as its side chains at  $\beta$ -D-Glcp. Moreover, ULP modulated the expression levels of p16<sup>Ink4a</sup>, MMP2, FoxO1, GLP-1/GLP-1R, STAT3, and GLUT4 to improve the status of ageing and diabetes, which was concurrent with the increased abundance of *Dubosiella*, *Enterococcus*, *Romboutsia*, *Bifidobacterium*, *Kurthia*, *Clostridium\_sensu\_stricto\_1*, *Corynebacterium*, *Faecalibaculum*, *Aerococcus* and *Vibrio*. Notably, *Dubosiella*, *Romboutsia*, *Bifidobacterium*, *Turicibacter* and *Clostridium\_sensu\_stricto\_1* could serve as important intermediates for delaying ageing and diabetes. Additionally, the ULP-1 structure is strongly binding interaction with the target protein through hydrogen bonding and Van der Waals force, especially for GLP-1 (−10.34 kcal/mol), p16<sup>Ink4a</sup> (−10.51 kcal/mol) and GLP-1R (−8.57 kcal/mol). Moreover, the average length of the hydrogen bond was observed to be 2.36 MPa, which is smaller than that of the traditional hydrogen bond. Therefore, ULP has the potential to function as a nutraceutical to delay or prevent the development of ageing-related type.

## LOQ H&N Purification, identification, and antioxidative mechanism of three novel selenium-enriched oyster antioxidant peptides

Xia, Z., et al., *Food Res. Int.*, 157, 111359, 2022.  
<https://doi.org/10.1016/j.foodres.2022.111359>

Natural organic selenium (Se) has multiple physiological health benefits and has become a hotspot of research in recent years. In this study, the Se-enriched antioxidant peptides were purified from Se-enriched oyster hydrolysate. Three novel Se-enriched antioxidant peptides LLVSeMY (685.2953 Da), MMDSemL (687.1875 Da) and VSeMDSemL (703.1599 Da) were identified from fraction F6-4, which all exhibited strong cellular antioxidant activity (CAA) with EC50 values of 0.739, 0.423, and 0.395  $\mu$ g/mL, respectively. These three Se-enriched antioxidant peptides (0.025 mg/mL) could significantly enhanced cell viability to  $84.60 \pm 3.32\% \sim 86.18 \pm 1.36\%$  compared with the AAPH injury group ( $75.99 \pm 0.79\%$ ), and the cytoprotective effects were even better than that of GSH ( $80.47 \pm 2.78\%$ ). Moreover, these three Se-enriched peptides also significantly protected HepG2 cells from AAPH-induced oxidative injury by inhibiting ROS production and enhancing the activities of antioxidant enzymes. The molecular docking results showed that these three Se-enriched peptides can form stable hydrogen and hydrophobic bonds with key amino acid residues of Keap1 protein, thereby potentially regulating the Keap1-Nrf2 pathway. In conclusion, the three novel Se-enriched oyster antioxidant peptides are expected to be used in medicine or functional food, providing a new theoretical basis for the high-value utilization of natural organic Se.

## LOQ ANA Identification of novel bioactive proanthocyanidins with potent antioxidant and anti-proliferative activities from kiwifruit leaves

Ly, Ji-Min, et al., *Food Biosci.*, 46, 101554, 2022.  
<https://doi.org/10.1016/j.fbio.2022.101554>

Agro-wastes contribute major social, economic, and environmental challenges for food production and circular economy systems. The current increasing demand for clean label food production and use of natural bioactive compounds could turn these challenges into opportunities providing avenues for proper utilization of agro-wastes to produce valuable products. This study aimed to investigate the potential use of kiwifruit (*Actinidia chinensis*) leaves as a source of proanthocyanidins (PAs) bioactive phenolic phytochemicals. Kiwifruit leaves PAs were extracted, purified, identified, and evaluated for their antioxidant and anti-proliferative activities. The structural composition of the purified PAs was characterized using HPLC-QTOF-MS/MS and MALDI-TOF-MS. The results showed that purified kiwifruit leaves PAs (PKLPs) comprised mainly procyanidins, propelargonidins, and prodelphinidins ranging from dimers to hexamers with (epi)catechin as terminal units and (epi)afzelechin or (epi)gallocatechin as dominant extension units. This study reports the structure of novel PKLPs monomer fractions was unique compared to the PAs that extracted from the other plant sources. The PKLPs exhibited higher phenolic content than the skin and flesh of several kiwifruit cultivars. Moreover, the PKLPs exhibited higher in vitro antioxidant activity in chemical-based (DPPH, ABTS, and FRAP) assays and H<sub>2</sub>O<sub>2</sub>-induced injury cell model than ascorbic, Trolox, and catechin ( $p < 0.01$ ). A remarkable dose-dependent anti-proliferation activity ( $IC_{50} = 186.04 \pm 2.61 \mu$ g/mL) against HepG2 cells was observed. In conclusion, this study demonstrated that kiwifruit leaves waste could serve as a sustainable and low-cost source of PAs, a group of multi-functional bioactive compounds that plays a key role in the food and pharmaceutical industries.

## LOQ ANA Isolation and characterization, antioxidant, and antihypertensive activity of novel bioactive peptides derived from hydrolysis of King Boletus mushroom

Kaprasob, R., et al., *LWT*, 160, 113287, 2022.  
<https://doi.org/10.1016/j.lwt.2022.113287>

King Boletus mushroom protein hydrolysate (KBMPH) was prepared using bromelain and isolated by membrane ultrafiltration into four molecular size fractions (>10, 3–10, 1–3, and <1 kDa). All fractions were assessed for their characteristics, antioxidant and angiotensin I-converting enzyme (ACE) inhibitory activities. KBMPHF4 (<1 kDa) showed the highest surface hydrophobicity (S<sub>0</sub>), and high levels of hydrophobic



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amino acid (HAA). KBMPHF4 at 1 mg/mL exhibited anti-oxidative against DPPH• and ABTS•+ and linoleic acid oxidation, and ACE inhibitory activity ( $IC_{50} = 0.12$  mg/mL). Peptide fraction-51 from ion-exchange-chromatography (IEC) showed high protein content, and biological properties. Three novel peptides (SETGGGHTTSCETDLGEFL, AAPLPGP, and GTDPTGEMLT) from F1 fraction had the highest antioxidant capacity and moderate ACE inhibitory activity. Six novel peptides (DLDLLEKGIRKT, NGGNAPI, VSWNVLQEP, DTGRGLGASH, IDDNLDNLIKL, and LIYAQGFSK) from F4 fraction had moderate antioxidant capacity and the highest ACE inhibitory activity. LIYAQGFSK peptide had the greatest ACE-binding energy ( $-9.2$  kcal/mol) through hydrogen bonds. Overall, novel peptides from KBMPHF4 could be potential use as natural antioxidants and antihypertensives in functional food and nutraceutical.

## H&N PRO Cold-pressed sesame seed meal as a protein source: Effect of processing on the protein digestibility, amino acid profile, and functional properties

Almeida Sá, A.S., *et al.*, *J. Food Compos. Anal.*, 111, 2022.  
<https://doi.org/10.1016/j.jfca.2022.104634>

Alternative protein sources for the human diet may help overcome the food security challenge for a growing population and its environmental impact. Edible oil extraction by-products are potential sources due to high protein content. This study characterized cold-pressed sesame seed meals about the proximate composition, antinutritional factors, amino acid profile and score, and in vitro protein digestibility (IVPD). A central composite experimental design supported evaluating the impact of cooking, microwave, and ultrasound on the nutritional quality of this meal. The raw sample presented 35% of protein and 89% of IVPD. Optimized conditions were 87.8 °C, pH 8.0, and 37 min for all processes studied, increasing IVPD up to 95%. The treatments effectively reduced up to 55% trypsin inhibitor activity and 81% phytic acid content. Lysine was the only limiting amino acid before or after processing. Sesame seed by-product has high-quality protein and can be a plant-based source for food formulations.

## BIO PRO Non-catalytic esterification of palm fatty acid distillate with 2-ethyl hexanol for high purity production of biolubricant ester

Pauzi, N.N.P.N., *et al.*, *Biofuel Bioprod Biorefin*, accepted.  
<https://doi.org/10.1002/bbb.2390>

Palm fatty acid distillate (PFAD), a byproduct of crude palm oil (CPO) refining, is an attractive feedstock for biolubricants due to its inedible nature and lower cost than palm oil. Ethylhexyl ester is a novel high-performance biolubricant synthesized via the non-catalytic esterification of PFAD with 2-ethylhexanol (2EH), normally the esterification of PFAD with methanol (MeOH). By substituting a longer carbon chain alcohol for MeOH, the cold flow characteristics, flash point, and oxidative stability are improved. The optimal esterification parameters were 1:2 (PFAD:2EH) mole ratio, 180 °C, 100 rpm, and 6 hours. The kinetic reaction fitted well the pseudo-first-order with an activation energy of 29.89 kJ/mol and a pre-exponential factor of  $15.05 \text{ min}^{-1}$ . The increase in ester purity and conversion from 44 to 95% and 97 to 99.5% were achieved by fractionation via vacuum distillation at 125–200 °C and 47–55 mbar. The physicochemical properties of the ester appear to be suitable as ISO VG 10 lubricant for hydraulic application in terms of kinematic viscosity at 100 °C of  $2.9 \text{ mm}^2/\text{s}$ , a viscosity index of 243, and a flash point of 180 °C. This study will assist in designing industrial biolubricant reactors.

## BIO PRO Microbial co-culturing of *Chlorella* sp. And *Rhodotorula toruloides* for the enhanced microbial lipid production

Wang, H., *et al.*, *Fuel*, 324, Part B, 2022.  
<https://doi.org/10.1016/j.fuel.2022.124609>

A microbial co-culture system composed of *Chlorella* sp. and *Rhodotorula toruloides* was established to improve the microbial lipid production in this study. Within this mixed culture, *Chlorella* sp. acts as oxygen emitters to provide a constant supply of oxygen and regulate the pH, leading to higher biomass and lipid production. Subsequently, a series of culture conditions were optimized to improve the suitability and stability of the cocultivation system, including the C/N ratio, temperature, inoculation time and inoculation ratio. Under the optimal conditions, the accumulation of biomass and lipid content was improved 26.9 g/L and 70.94%, which was 1.8-fold and 2.13-fold than that of pure culture, respectively. Interestingly, the composition of fatty acids was significantly changed under different temperatures in mixed culture systems. The enhancement of unsaturated fatty acids will contribute to high value-added applications. In addition, the highest biomass of 31.9 g/L was obtained from molasses, indicating the great potential for industrial application.

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