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October 2015

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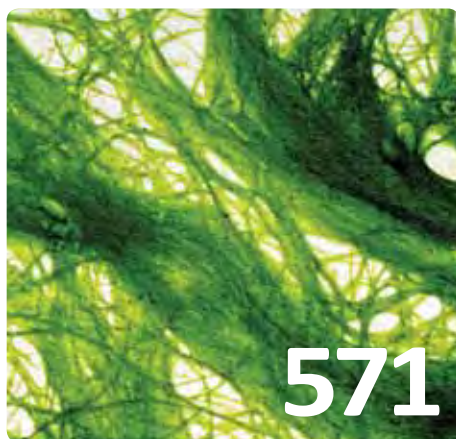
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Separation anxiety

Catherine Watkins

Right now, millions of industrial membranes are hard at work. They are separating macromolecules from seawater and wastewater. They are filtering out bacteria, concentrating proteins, and reclaiming process water in food production. They are removing particulates from flue gases in the petrochemical industry and accomplishing specific fractionations of high-value products in the biotech and pharmaceutical industries. In the process, they are all getting dirty.

- Membrane separation technology is a global industry that is expected to grow 9.2% annually through 2017, according to The Freedonia Group market research firm.
- The market for membrane cleaners formulated for specific types of foulants is also expected to grow.
- This represents a largely unexplored business opportunity for surfactant researchers, formulators, and manufacturers.

No matter the type of filtration being conducted—whether reverse osmosis, ultra-, nano-, or microfiltration—each of these millions of membranes is becoming fouled and losing efficiency. Small membrane assemblies, known as elements, generally are discarded once they are fouled, but it is more cost-effective to clean larger assemblies. Many have to be cleaned frequently, some every day, most often in place. This, as it turns out, represents a huge business opportunity for surfactant researchers, formulators, and manufacturers.

MARKET BACKGROUND

Membrane technology is a comparatively recent development. Created in the 1960s when Sidney Loeb and Srinivasa Sourirajan synthesized the first asymmetric membranes, the technology took off in the 1970s and 1980s. Currently, about three-quarters of commercially available membranes are made of organic polymers; the rest are made of ceramic, metallic, or other inorganic materials, according to consultant H.S. Muralidhara of INSEPPPCON, LLC in Plymouth, Minnesota, USA. Muralidhara, formerly with Cargill, is a co-editor (with Z.F. Cui) of *Membrane Technology* (<http://dx.doi.org/10.1016/B978-1-85617-632-3.00002-1>, 2010, Elsevier Ltd.) and a frequent presenter on the topic.

Despite being new to the scene, membrane technology is a large and robustly growing field. Market research conducted by The Freedonia Group in Cleveland, Ohio, USA, found that global demand for membranes likely will increase by a healthy 9.2% annually to \$25.7 billion in 2017. Rising environmental standards and regulations in many parts of the world as well as high population growth—particularly in water-stressed areas—are driving investment in membrane-based water and wastewater treatment systems. Increased attention to food and beverage safety regulations and rising interest in water reuse and material reclamation are also propelling membrane sales, Freedonia says.

The countries expected to see the fastest growth include the BRIC (Brazil, Russia, India, and China) countries and others with large, developing industrial bases and stressed local water resources. Combined, the United States and China, the two largest national markets for membranes in 2012, are expected to account for 43% of the market gains between 2012 and 2017.

A MEMBRANE SEPRATION PRIMER

Membrane separation—which is a purely physical process and, therefore, uses less energy than traditional thermal separation processes that depend on a change in state—varies depending on the size of the separated particles and the separation mechanisms.

membrane cleaning in the 21st century



Industrial membranes are either porous or nonporous. Porous membranes are either microporous or asymmetric; they separate components in a mixture with pressure as the driving force. Nonporous (or dense) membranes generally are used to separate gases from gas mixtures. In this case, the membrane layer attracts molecules with high affinity, which are then diffused through the membrane, whereas molecules with low affinity are retained. An important caveat: This article deals mainly with the cleaning of membranes fouled via pressure-driven membrane processes operating on liquid feed streams.

The four primary pressure-driven membrane separation processes are classified by the pore size of the membranes and the transmembrane pressure (TMP) required. They are:

- Microfiltration (MF)—0.1–5 μm , 1–10 bar
- Ultrafiltration (UF)—500–100,000 Daltons (Da), 1–100 nm, 1–10 bar
- Nanofiltration (NF)—100–500 Da, 0.5–10 nm, 10–30 bar
- Reverse osmosis (RO)—<0.5 nm, 35–100 bar

The RO process is widely used for desalination and wastewater treatment; the UF and MF processes are commonly used in food, bioprocessing, wastewater processing, and industrial separation processes.

Organic membranes generally are manufactured from various polymers, including cellulose acetate, polyamide, polysulfone, polyethersulfone, polyvinylidene fluoride, polypropylene, and the like. Inorganic membranes—produced from ceramic, metallic, or other inorganic materials—are mechanically strong and chemically and thermally stable. They also tolerate a wider variety of temperatures, pHs, and pressures. However, they are also much more expensive than polymeric membranes.

Industrial membranes are assembled within devices and hardware, often called modules or elements (see Fig. 1, page 552), in order to separate the feed stream into retentate and permeate streams. The four main types of modules are tubular, hollow fiber, flat sheet, and spiral-wound.

CONTINUED ON NEXT PAGE



Fig. 1. Membrane assemblies, known as elements, generally are thrown out once they are fouled, but it is more cost-effective to clean larger assemblies. Photo courtesy of Avista Technologies, Inc.

Suspended or dissolved substances deposited on the surface of industrial membranes—known as foulants—result in performance losses from a reduction in the permeability (or flux) of the membrane (see Table 1). Factors that influence the rate of fouling include:

- The nature and concentration of solutes and solvents,
- The type of membrane,
- The pore size distribution,
- The surface characteristics and material of the membrane, and
- The hydrodynamics of the membrane module.

MEMBRANE CLEANING BASICS

Cleaning time, temperature, hydrodynamic conditions, and concentration of cleaning agents all affect membrane-cleaning efficiency, as Hongyu Li and Vicki Chen point out in *Membrane Technology* (<http://dx.doi.org/10.1016/B978-1-85617-632-3.00002-1>). In addition, cleaning protocols must be adapted for each application and location. Table 2 summarizes the steps used in developing a cleaning regimen.

Membrane cleaning can be costly. Consultant Gerold Luss of Luss Consulting in Minneapolis, Minnesota, USA, notes that the annual cost of cleaning a single membrane unit in a dairy plant—which, of course, varies with size—can be as much as \$30,000 for a small unit to \$150,000 for a large unit. Membrane cleaning costs for seawater desalination also vary widely, Luss says, ranging from \$5.00 per cleaning for a small undersink unit to \$50,000 per cleaning for a large plant that provides water to a major city.

“Membrane chemical cleaning formulations are some of the more expensive formulations because they are tailored to the specifications of the membrane, the

application for which the membrane is used, and also the expected cleaning conditions,” says Luss. “That being said, I would place membrane cleaning chemicals at about the 75th to 80th percentile in terms of cost in industrial applications.”

Most membrane cleaning is done in place, generally when the flux rate through the membrane decreases by 10% and/or the TMP increases by 10% and/or the permeate water quality decreases by 10%. The typical clean-in-place (CIP) sequence for membrane systems includes a pre-rinse, detergent wash, post-rinse, acidified wash, a second alkaline wash supplemented with chlorine when the membrane is compatible with oxidizing agents, and a final rinse. The mechanisms involved include dissolution, chelation, oxidation, hydrolysis, and emulsification (see Table 3 on page 561).

Felicity Plansky, a membrane technical field specialist at Hydrite Chemical Co. in Brookfield, Wisconsin, USA, offered a handy mnemonic device for remembering which surfactants work well in membrane cleaning formulations during a workshop at Cornell

University in March 2014. In brief: cationic = catastrophic (because they are not compatible with negatively charged membranes), non-ionic = nonproblematic, and anionic = applicable.

Plansky also noted that membrane manufacturers issue very strict limits on the temperatures allowed for cleaning in place for many membranes. Typically, for UF and MF membranes, the temperature must be below 130°F, and for RO and NF membranes, the temperature should be below 118°F. However, recent

Table 1. Typical membrane foulants and fouling modes

Foulants	Fouling mode
Large suspended particles	Can be present in the original feed or created by scaling; can block membrane channels.
Small colloidal particles	Can rise to become a fouling layer.
Macromolecules	Can form a gel or cake on membranes or within the structure of porous membranes.
Small molecules	Can have strong interactions with plastic membranes; for example, antifoaming agents such as polypropylene glycols.
Proteins	Can interact with surface or pores of membranes.
Chemical reactions	Can lead to precipitation of salts and hydroxides with increase in concentration and/or pH.

Source: Shi, X., G. Tal, N.P. Hankins, V. Gitis, Fouling and cleaning of ultrafiltration membranes: A review, *J. Water Proc. Eng.* 1:121–138 (2014).

developments in polymeric membranes now allow CIP at high pH (12+) and higher temperatures (up to 180°F) for certain membranes.

HOW MEMBRANE CLEANING HAS CHANGED

Membrane cleaning has changed both quantitatively and qualitatively in the past 40 years, according to a number of industry observers.

For example, three membrane specialists from Sealed Air in Charlotte, North Carolina, USA, find that the biggest change has been in the use of more generic chemicals for cleaning membranes, especially for cleaning UF membranes. Dennis Schmidt, Flemming Skou, and Gaetano Redaelli—who submitted written comments for this article—also noted that even RO membranes are being cleaned more with basic acid and caustic than in the past. Schmidt is an account manager and Flemming is a senior applications fellow, both with Sealed Air Hygiene Solutions. Redaelli is vice president and global sector lead with Sealed Air Food & Beverage.

“When membrane technology first appeared, most cleaning was done with formulated cleaners,” they wrote, adding that the development of membranes that can withstand higher cleaning temperature and have higher pH tolerances “has taken membrane cleaning into new processes that weren’t possible before. For example, some membrane plants in gelatin production can now be cleaned without the use of active chlorine, yielding longer membrane life.”

The first membranes were made of cellulose acetate, which fouled less than today’s polymeric membranes, says Dan Comstock, a founder and director of R&D at Avista Technologies, Inc. in San Marcos, California, USA. Because of water shortages the world over, he added, feed waters for RO filtration are being used “that you would never have considered” in the past.

“The unit cost of generic cleaners is certainly less than formulated cleaners,” notes Comstock, “but generic cleaners are generally not as effective. Therefore, an overall economic case can be made for formulated cleaners, for they can extend membrane element life and reduce energy costs.” The proliferation of UF and MF membrane systems has increased the business opportunities for surfactant researchers and manufacturers, he adds, while noting the need for low-temperature cleaners as well as formulations that are compatible with hard water.

Comstock illustrates the analytical challenges of membrane fouling with an anecdote. “A reverse osmosis system owner thought he had a simple case of silica scaling,” he says. “An autopsy showed the presence of silica, aluminum silicates, iron phosphate, and calcium

Table 2. Developing a cleaning regiment

Define and characterize foulant composition and type
Determine the most severe fouling scenario (chemical and spatial distribution)
Select cleaning techniques and chemicals
Identify process inputs and variations (feedstock, operational parameters, fouling patterns, etc.)
Specify cleanliness acceptance criteria
Commission and calibrate equipment
Identify suitable measurement, testing, monitoring systems
Develop standard operating procedures and training
Implement cleaning regime
Troubleshoot membrane autopsies

Source: Adapted from “Membrane Fouling and Cleaning in Food and Bioprocessing,” Ch. 10, *Membrane Technology*, Hongyu Li and Vicki Chen, p. 233, <http://dx.doi.org/10.1016/B978-1-85617-632-3.00002-1>.

phosphate. With the aid of Chromatic Elemental Imaging and a process called heterogeneous nucleation, we were able to deduce that the aluminum silicates, iron phosphate, and calcium phosphate were triggering silica precipitation. By changing the scale inhibitor to one that inhibits these sparingly soluble salts, the customer has been able to operate his system silica scale-free.”

SCOPE OF THE OPPORTUNITY

Although water is one of the most important elements in membrane cleaning—representing 99% of any membrane cleaning solution—

surfactants, and particularly surfactants in tailored formulations, are critical to membrane cleaning.

“Surfactants are key and will be key in the future,” says Manuel Soria, global product manager, membrane chemicals, with GE Water and Process Technologies in Barcelona, Spain. Soria agrees that RO and NF membranes are now used under more stress conditions because of worse-quality water containing more foulants. This leads to the need for more sophisticated formulations.

“Today, the materials that create difficulty with removal are mainly organic and biofoulants,” he says. The GE labs receive membranes that have special problems, he notes. “In these cases, we found that 70–85% of the contaminated membranes are mainly fouled with organic material from bacteria. This is more difficult to remove. In order to keep the system working, you need to remove this material or you need to change the membrane, and surfactants are necessary for penetration of the cleaner into the membrane foulant.”

Among the difficulties in formulating membrane cleaners is ensuring the stability of formulations, Soria says, particularly with regard to formulations that are stable as solids but not as liquids. Also, increasing the concentration of surfactants in the formulation brings with it the risk of foaming. “As more surfactants are required to clean difficult foulants, there is a greater need to develop low-foaming surfactants with minimal environmental impact,” he adds.

Perhaps the greatest need, according to Comstock, is to “try to get rid of trial and error in cleaner formulation and membrane autopsies and inject science into the process.

“If I have any legacy,” he adds, “I hope that will be it.”

Catherine Watkins is an associate editor of Inform magazine. She can be contacted at cwatkins@aocs.org.

Next-generation cleaning products that "have it all"

Fiona Case

Regulatory pressure and consumer and retailer demands for less toxic, more sustainable, and environmentally benign products are driving materials innovation. This, in turn, is providing significantly improved performance in the next generation of high-efficiency cleaning ingredients.

This is good news for purchasing departments, customers, formulators, environmentalists (and elephants).

Over the last century, myriad products have superseded simple soaps and elbow grease to cleanse clothing, skin, hair, and the surfaces that surround us, with efficiency and panache. But as with other aspects of our modern lifestyles, the convenience these products bring bears an environmental, health, and safety (EHS) and sustainability price-tag that regulators and an increasing proportion of consumers are now unwilling to pay.

- Green solvents offer sustainability and price stability.
- New catalysts for ethoxylate surfactants enable significant performance gains.
- Novel enzyme inhibitors liberate formulation space.
- New chelators provide good biodegradability profiles.
- Biosurfactants promise technical performance, sustainability, and biodegradability.



"Environmental concerns have been driving innovation," says Erika Szekeres, who recently founded Stable Formulations consultancy after a 10-year career with The Clorox Company. "But inventory for greener alternatives has been very limited—with few choices, there is often a loss of performance when companies start formulating for EHS or sustainability."

Formulators are faced with the challenge of addressing regulatory pressures and supporting sustainability (reducing their petrochemical dependency) and EHS claims, while maintaining, or improving, product performance and price. Suppliers are coming to their aid with an inspiring new generation of additives for cleaning that are both greener and more efficient.

SOLVING DISSOLVING

From a technical perspective, cleaning is an extremely complex process, and performance requirements vary depending on what is being cleaned. The first step is to introduce the cleaner to the dirt. Surfactants allow a cleaning solution to penetrate between clothing fibers or to flow across surfaces and skin by reducing surface tension. Once there, water is an excellent solvent for many soils, particularly when introduced with rubbing or agitation. Unfortunately, removing oily soils requires organic solvents—those traditionally low-molecular-weight (and low-vapor-pressure) materials that are now experiencing regulatory scrutiny.

"The volatile organic content (VOC) level is now regulated in cleaning products, and it was recently reduced to very low levels in California," says Szekeres. This has triggered interest in solvents that have low VOC or are on the US Environmental Protection Agency (EPA) VOC-exempt list—"compounds that have been found by the EPA 'not to contribute appreciably to ozone formation,'" she says. Szekeres points to The Dow Chemical Co.'s P-Series glycol ethers derived from propylene oxide.



"The lower volatility of these solvents satisfies regulators, and they can provide equivalent performance to traditional high-VOC materials," she says. An added benefit to formulators is the ability to reduce oily and expensive fragrance levels, since there is less solvent smell to mask.

Sustainability is becoming a watchword for solvents, too. Elevance Renewable Sciences, Inc. in Woodridge, Illinois, USA, uses metathesis chemistry on reactive olefins naturally present in plant-derived oils (palm, mustard, soybean, canola) to create a range of VOC-exempt bio-solvents and personal care ingredients that are both greener and higher performing than traditional petrochemical products.

"Their plant-derived solvents achieve cleaning performance that is better than traditional materials in industrial degreasing. They also provide a renewable source of raw materials that fit performance and cost requirements for the manufacture of surfactants," says Szekeres.

Segetis, a green chemistry company in Golden Valley, Minnesota, USA, has developed two sustainably sourced solvents using levulinic acid from cellulosic biomass: One has similar performance characteristics as d-limonene, and the other can replace glycol ethers.

"In laundry products, solvents aid dissolution of detergent ingredients so that the product is still pourable at high concentrations." Method, headquartered in San Francisco, California, USA, uses this ethyl glycerine acetal levulinate in its 8X laundry products: "a formulation triumph; their competitors have only 4X or 2X," says Szekeres. "Less water in the product means less weight to transport, reducing fossil fuel use for trucks; it requires smaller bottles and less packaging material, further reducing environmental impact."

"Using materials from commodity plant crops rather than from oil also offers price stability," she adds. "I've seen significant increase in prices for materials from petrochemical sources because of geopolitical crisis."

ALCOHOL ETHOXYLATE REBIRTH

For oily dirt, grease cutting and emulsification are required. The surfactant solution must encapsulate and remove the hydrophobic material and prevent redeposition until the mixture is wiped or rinsed away. The formulator tunes the hydrophilic/lipophilic balance (HLB), or interfacial curvature at the oil/water interface to encourage emulsification of specific types of soil—while also controlling foaming (more, or less; small or large bubbles), viscosity, and flow. And don't forget the latest sensory "wow": colors, fragrance, sparkles. A toolbox of surfactants and polymers enables the design, but some of those tried and trusted tools must be replaced.

Alcohol ethoxylate (AEO) surfactants are short poly(ethylene oxide) head groups added to aliphatic alcohol tails that provide wetting and grease removal in modern hard-surface and laundry applications. Co-formulated with ionic surfactants, they also provide mildness and foam in personal care formulations. When regulatory and consumer pressure drove nonylphenol ethoxylates (NPE) off the table, many formulators turned to AEOs, but they were not delighted.

"Standard AEO surfactants contain a wide oligomer distribution," says Heather Byrne, a chemist with Huntsman Performance Products in Spring, Texas, USA. "The synthesis can leave about 25% free alcohol which doesn't contribute to the detergency. The residual alcohol can leave streaks reducing gloss retention in hard-surface applications and contributes to a lower cloud point."

But there is a revival for AEO surfactants. Byrne presented performance results for a new line of narrow range ethoxylate (NRE) surfactants at the 2015 AOCs Annual Meeting and Industry Showcases, May 3–6, in Orlando, Florida, USA.

"Our new calcium-based catalyst creates a peaked distribution of EO groups. The concentration of 1-4 dioxanes is reduced, and there is negligible unreacted alcohol. This raises the cloud point by about 10 degrees, which allows you to work at higher temperatures," she explains.

The NRE surfactants have lower interfacial tensions and great wetting performance compared to their broad-range counterparts. Byrne tested them in hard-surface cleaning and laundry. The new synthesis allowed her to optimize the surfactant structure for each application.

"I was very enthusiastic about the results," she says. "The narrow-range AEO 24-7, a surfactant with C12-14 tail and 7 ethylene oxides, had the best detergency in laundry. It outperformed everything in my study, including the NPE control. The narrow-range AEO 12-6 gave great results for hard-surface cleaning on tile soiled with a modified ASTM soil. My results were equivalent to the old nonylphenol materials, and there was high gloss retention: no streaks."

The one criterion that every formulator must consider, in addition to all the technical and aesthetic requirements, is cost. The greater efficiency of the new NRE materials allowed Byrne to use less surfactant to achieve better technical results.

"I used 4% surfactant and water; a low concentration because our customers want product at reasonable price. My customers are not moving away from nonylphenols because of price—they are moving so they can make sustainability claims—but we are always struggling to make a formulation that is lower cost with great cleaning," she says.

BIODIRT

For laundry, dish, and surgical cleaning applications, the soil includes high-molecular-weight biomolecules (food residues and dried biological fluids, for example) which can be firmly attached to clothing fibers and surfaces. Traditional approaches, such as boiling the laundry and rough treatment on a washboard or rock, are not appropriate for modern fabrics or consumers. However, since the 1960's enzymes such as protease, amylase, lipase, and cellulase have provided a great solution, happily chewing up the soil at low temperatures. The challenge is to curb their cannibalistic tendencies, particularly in liquid formulations.

"Currently, many liquid detergents that contain protease use boric acid or borate-based inhibitor systems to shut down proteolysis [protease degradation] while the product is in storage so the end user experience is better even if they have kept the product for several months," says Victor Casella, a staff scientist in technical services at Novozymes North America Inc. "There's nothing wrong, technically, with the boron-based systems, but greater regulatory scrutiny of boric acid and increased trade and consumer focus on sustainable chemistry makes an alternative desirable."

Novozymes has introduced a new boron-free reversible protease-inhibitor system, and while fulfilling this desire has created a product which provides new formulation benefits.

"The new system provides better enzyme stabilization than current boric/borate formate or built-in systems, particularly at higher pH, and it is much more efficient," says Casella. "The required concentration is low enough that it is added in the raw material. Current inhibitors occupy several percent of the formulation. As products become more concentrated, formulators want to minimize space taken up by ingredients that do not deliver an end benefit to the consumer. This new solution frees up formulation space and allows for further inclusion of different enzyme classes to provide even greater cleaning performance for the end user, particularly in North America, where liquid is the dominant detergent format," he explains.

MITIGATING METALS

Metal ions are bad news for cleaning. Detergent performance drops in hard water and foam vanishes as anionic surfactants are ensnared by their positive charms. Mineral deposits mar glasses, cutlery, and countertops; hard water plus soaps leads to scum. Ethylenediamine tetraacetate (EDTA) or nitrilotriacetic acid (NTA) chelating agents have mitigated this problem for many decades, but they are not biodegradable, nor sustainably sourced: Another opportunity for novel cleaning ingredients?

AkzoNobel, based in Amsterdam, the Netherlands, offers glutamic acid diacetic acid (GLDA), one of the new generation of chelating agents. Biochemical production from carbohydrate sources such as sugar, molasses, corn, or rice bolsters sustainability claims and provides the natural L-isomer that is required to meet the "readily biodegradable" profile.

Polyacids can sequester the problematic positive ions and also prevent redeposition of dirt. Poly(acrylic acid) is well established as a chelator and dispersant, but is light on sustainability credentials. Five years ago, AkzoNobel provided a new way to combine sustainably sourced material—polysaccharides—with synthetic materials providing technical benefits and a good biodegradability profile (the Alcoguard hybrid bio-polymer was introduced at the 2010 AOCs Annual Meeting & Expo), and the second generation provides further improvements in technical performance with 75% polysaccharide.

Itaconix Corp. in Stratham, New Hampshire, USA, provides sodium poly(itaconic acid), a chelator and dispersant for home, industrial, and institutional detergents and cleaners that is made entirely from biobased feedstock. Their itaconic acid raw material is produced by fermentation of carbohydrates such as corn with *Aspergillus terreus*, a common strain of soil fungus. The biomaterial is readily biodegradable with low aquatic toxicity.

BIOSURFACTANTS: THE PERFECT BREW?

The prescription for the ideal ingredient for a modern cleaning product includes excellent technical performance, good sustainability credentials (no fossil carbon), freedom from GMOs, proven biodegradability, and low- to zero-toxicity for people and the environment. Biosurfactants can fulfill all these criteria.

"It has long been known that certain microbes yield surface-active substances. Since the 1970's researchers have sought to establish industrial applications of these materials as biosurfactants, but these early investigations did not lead to commercial-scale use due to low productivity and quality," says Yoshihiko Hirata, the director of Biochemical Laboratory at Saraya Co., Ltd. in Japan. "We started looking at biosurfactants in 1997. We developed a method for volume production of sophorolipid (a glycolipid biosurfactant) and also found unique and useful properties, particularly for applications as biodegradants for household dishwashing and laundry."

One useful finding was that sophorolipids provide low-foaming properties along with excellent biodegradability.

"Many surface-active substances form foam, which is particularly undesirable in home laundry machines and dishwashers. The structural and formulation changes required for low-foaming with conventional surfactants leads to relatively poor biodegradability," says Hirata. "We discovered that our sophorolipid, SOFORO, had low-foaming properties while enjoying equal surface activity to that of synthetic surfactants. Biodegradability tests according to the Organisation for Economic Co-operation Data (OECD) Test Guidelines revealed that SOFORO reached 60% degradation in 10 days. 60% in 28 days is deemed achievement of the pass level for biodegradability. Biodegradation began immediately after the start of the test. SOFORO scarcely affected the activated sludge. This finding was the breakthrough for our success in commercialization of SOFORO biodegradants for household dishwasher and laundry," he says.

Aquatic toxicity testing showed that SOFORO caused almost no damage to aquatic life, such as Japanese rice fish, even at concentrations that provided surface activity.

"With regard to human toxicity, we conducted cytotoxicity testing using human epidermal keratinocytes, which are commonly used for safety evaluation of cosmetics," says Hirata. "The test results

revealed that SOFORO had lower toxicity than that of common synthetic surfactants including sodium dodecyl sulfate (SDS) and alcohol ethoxylates (AEO). SOFORO may be considered a highly-biocompatible surfactant.”

Sophorolipid has an unusual structure for a surfactant; it has two heads and naturally exists in two forms: the lactone sophorolipid form and the acidic form. Research at Saraya has revealed some surprising benefits to this structure.

“In detergent formulation, a product that meets consumer requirements is achieved by mixing several kinds of surfactant. Sophorolipids are naturally a mixture of two types of surfactants (lactonic and acidic). The lactonic-type sophorolipid alone is sparingly water-soluble; the acidic-type alone had low surface activity due to its high HLB. The mixing of both types increases solubility in water as well as surface activity. We called this property the ‘natural synergism’ of sophorolipid, and we have keenly investigated it,” says Hirata. “It seems as if the yeast has done the optimization of the biosurfactant in its evolutionary history through natural selection, by making the best of the synergy of the surfactants just like an ingenious detergent developer.”

The SOFORO surfactants are produced by fermentation of palm oil using wild yeasts (not genetically modified). The palm oil is RSPO-certified “segregated” palm oil that is produced with minimal damage to the environment and without violation of human rights. Saraya adds to its environmental credentials by working with the Borneo Conservation Trust (<http://www.borneotrust.org/>) on conservation efforts and supports an elephant sanctuary in areas impacted by irresponsible palm plantation expansion; the sophorolipid-based laundry and dishwashing products of Saraya are marketed as the Happy Elephant brand.

LOOKING FORWARD

Cleaning will continue to be influenced by the world’s meta-trends ; retailers and their customers will delight in (and want to purchase) better, cheaper, greener products with strong environmental back-stories. Based on recent examples, this is eminently achievable.

“As suppliers are pushed towards more sustainable and biodegradable materials they often need to explore new chemistries and approaches, new raw materials and processes, and to

develop a deeper understanding and control of their materials,” says Szekeres “With new generations of ingredients, we may be pushed out of our traditional formulation comfort zones, but this is leading to new products that are better for everyone.”

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Yusuke Saraya, president of Saraya Co., Ltd. in Japan, poses with an elephant at the Bornean Elephant Sanctuary. Learn more about the company’s Happy Elephant Brand at <http://happy-elephant.info/>.



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Biobased solvent replacements that perform, comply, and satisfy

**Ryan Littich, Michelle Morie-Bebel,
and Steve Block**

*A complete list of references available at
<http://tinyurl.com/Biobased-Solvents>*

Solvents play an integral role in creating products that meet and exceed consumers' performance expectations, yet consumers increasingly value sustainability—not just performance—in determining their satisfaction with those products. The contradictions arising from these competing expectations are readily apparent. First, volatile organic solvents are widely used in finished products because of their high performance but they account for a significant percentage of documented anthropogenic emissions [1]. Second, the toxicological profile—both ecological and human—of certain solvents is incongruent with

consumer expectations. Such is the case with *N*-methylpyrrolidone (NMP), a tremendously effective solvent that is also present on California's Proposition 65 list of chemicals known to cause reproductive toxicity. Last, but certainly not least, consumers increasingly expect their products to be developed from resources that are naturally replenished, but today's solvents tend to have origins in finite petrochemical stores. The resolution of these contradictions is a standing challenge to ingredient suppliers and the consumer and industrial product formulators they serve.

- Growing consumer awareness is driving demand for home and personal care products formulated with eco-friendly and natural ingredients that perform as well as—or better than—existing, conventional products.
- Meeting these challenges has become increasingly difficult with regulations becoming stricter, customers demanding more and limited options, and large retailers such as Walmart and Target implementing sustainable chemistry policies that require suppliers to use non-hazardous ingredients that have minimal impact on the environment.
- Biobased raw materials derived from renewable resources are helping formulators meet these new requirements. This article highlights these trends and examples of commercial biobased replacement ingredients used in cleaners, paints, and personal care products.

Pressure from regulatory agencies and retailers has added urgency to this challenge. In the United States, restrictions on product volatile organic compound (VOC) content, administered as a result of local [2] and federal [3] clean air directives, have become more stringent. Proliferation of the globally harmonized system of classification and labelling of chemicals (GHS) for material safety data sheets further ensures the move away from suspected and confirmed toxic ingredients such as NMP and powerful, but persistent bioaccumulative and toxic (PBT) alkylphenol ethoxylate detergents. Meanwhile, retail competitors Target and Walmart have aligned to prompt their suppliers to offer more sustainable products. Priority chemicals and transparency, waste and packaging, and consumer behavior elements of sustainability were emphasized during the Beauty and Personal Care Products Sustainability Summit, sponsored by the retailers in the fall of 2014 [4, 5].

INNOVATION LOVES CONSTRAINTS

Although consumer, regulatory, and supply chain demands for sustainable products have evolved quite dramatically, so too have the technological innovations being put forth by ingredient manufacturers. Empowered by breakthrough chemical catalysis and biological fermentation technologies, new and existing suppliers are changing perceptions of what is achievable using renewable and sustainably sourced feedstocks. In some cases,

biobased materials and chemicals (BBMC) manufacturers are producing petrochemical drop-in offsets that provide known, tried-and-true ingredients with the tools to meet sustainability expectations. In other cases, manufacturers are providing next-generation products with enhanced performance and sustainable origins, to boot. A few suppliers are fielding both of these options. This change in the ingredient landscape is vast and compelling [6]. This article highlights ingredient innovations that are empowering product manufacturers and retailers to address three key performance-compliance-consumer satisfaction challenges.

VOC LIMITATIONS IN INDUSTRIAL AND INSTITUTIONAL CLEANING

Metal degreasing is an important undertaking in the automotive, metalworking, transportation maintenance/repair, and oil and gas segments. Consumers and service providers in these sectors are experiencing performance shortcomings in the degreasing and cleaning products they use due to escalated regulatory VOC constraints and the growing exclusion of aromatic solvents like benzene, toluene, and xylenes. To date, achieving compliance *together with* performance has been difficult with sustainably sourced solvent alternatives, usually because of product oxidative instability (as with methyl soyate), intrinsic high volatile organic content, or supply and price fluctuations (as with d-limonene).

Suppliers, new and established, have risen to the VOC challenge in different ways [7]. Eastman Chemical and Dow Chemical Company have harnessed petrochemical building blocks to manufacture VOC-compliant solvents Omnia™ (butyl 3-hydroxybutyrate) and Dowanol™ DiPPh (dipropylene glycol phenyl ether), respectively. DuPont uses its biotechnology (fermentation) manufacturing footprint to provide the hard surface cleaning solvent Zemea™ (1,3-propanediol; PDO). By this route, 40% fewer greenhouse gas emissions are generated than with existing petrochemical manufacturing processes for PDO. Elevance Renewable Sciences has highlighted with Elevance Clean™ 1200 (methyl 9-dodecenoate) the performance benefits of olefin metathesis catalysis in the refining of natural seed oils. The cleaning kinetics of products containing the VOC-exempt [8], biobased, and rapid biodegradable ingredient exceed those of use-restricted aromatic petrochemical and biobased terpene VOC solvents. These are just some of the ingredients empowering formulators to meet the consumer and regulatory VOC requirements.



Regulatory constraints on the use of aromatic solvents such as benzene, toluene, and xylenes have made degreasing gears and other metal parts particularly challenging. Fortunately, several new VOC-compliant, biobased, and rapidly biodegradable ingredients demonstrate strong degreasing performance.

REPRODUCTIVE TOXINS IN PAINT STRIPPING, GRAFFITI, AND ADHESIVE REMOVAL

Paint and adhesive removal products generally rely on highly concentrated or non-aqueous solvents to deliver the performance customers expect. NMP has held and continues to hold a strong position in paint-stripping products owing to its excellent solvency against pigments, binders, and surfactants deposited by paints. The same is true of NMP with respect to adhesives. However, with a growing understanding of the reproductive toxicity of *N*-methylpyrrolidone and its presence on California's Proposition 65 list, formulators are searching for viable replacement alternatives. Where ketones like acetone and methyl ethyl ketone or chlorinated solvents may come to mind in terms of performance considerations, the former happen to be contributors to smog [9, 10, 11] and the latter, ozone depletion [12].

Putting aside the notion that solvent replacement strictly requires a different solvent, Stepan Company introduced STEPOSOL® MET-10U, a specialty ingredient that has the attributes both of a low hydrophilic-lipophilic balance surfactant and a powerful solvent. Rooted in the catalytic upgrading of natural oil [13], this unsaturated, amide surfactant is characterized by strong solvent power registering higher than 1000 on the Kauri-butanol scale. STEPOSOL® MET-10U has changed the way formulators view the construction of paint, graffiti, and adhesive removers. Water-enriched, semi-aqueous cleaners based on this new ingredient are outperforming even 100 percent-solvent commercial products. Importantly, the biobased ingredient is considered a non-VOC [14], exhibits ready biodegradability, and does not appear on California's Proposition 65 list (see article on page 586). This combination of attributes has enabled consumer and industrial product manufacturers to overcome the performance-sustainability challenge not only in NMP replacement efforts, but others as well.

An array of solvent alternatives to NMP has been adopted as well, in view of the solvent's reproductive toxicity. First, lactic acid (2-hydroxypropionic acid) esters appear in a significant number of formulated paint and adhesive removal products [15], a result of the carbohydrate fermentation efforts of NatureWorks, Corbion Purac, Archer Daniels Midland, and others. Second, dibasic esters, originally positioned as methylene chloride alternatives in paint and adhesive removal products, have more recently been accepted in the effort to displace NMP. Today, these glutarate, succinate, and adipate ester mixtures are derived from intermediate byproducts of the nylon manufacturing process [16]. In the near future, it's plausible that biobased succinic and adipic acid ester options will become available, in view of the fermentative production landscape being built by companies like BioAmber, Succinity (BASF-Corbion Purac JV), and Myriant.



Other biobased ingredients are displacing N-methylpyrrolidone (NMP), a tremendously effective solvent used in adhesive removal products that is also on California's Proposition 65 list of chemicals known to cause reproductive toxicity.

PETROCHEMICAL AND SILICONE CARRIERS IN PERSONAL CARE

Consumer sentiment toward the ingredients used in their personal care products is one of the most audible feedback loops for ingredient suppliers and consumer packaged goods companies. Ingredient and product formulation landscapes have markedly shifted in response to it. In the early 90s, when consumers became increasingly dissatisfied with the use of refined petrochemical carriers from a sustainability perspective [17], Dow Corning and its contemporaries championed silicones as innovative, functionally equivalent petrol alternatives [18]. More recently in 2009, when regulatory assessments called into question the toxicity and environmental fate of cyclo-methicones D4 and D5, consumers took notice [19–22]. Although follow-on studies suggested that these volatile silicones were benign [23], a persistent negative consumer sentiment has motivated the supply chain to change. However, how do suppliers and formulators proceed when both petrochemical *and* silicone options can be undesirable to the customer?

Somewhat ironically, ingredient manufacturers have leveraged the interchangeability of certain hydrocarbons and silicones cited during the emergence of silicones in personal care. Linear hydrocarbons originating from renewable resources have been introduced to the product development community with a welcome reception. Elevance Renewable Sciences, for example, introduced Elevance Soft CE-1213 (proposed INCI: C9-C12 alkanes (and) tridecane) and CE-1500 (proposed INCI: C13-C15 alkanes) as volatile and persistent conventional carrier replacements, respectively. These products, which reflect the versatility of the metathesis refining process, deliver the spreadability, lubricity, and volatility required for formulation sensory enhancement [24]. Grant Industries and BASF have also presented plant-based hydrocarbons to the market, offering Vegelight 1214LC (INCI: coconut alkanes (and) coco-caprylate/caprate) and Cetiol® Ultimate (INCI: undecane (and) tridecane) into the market as volatile natural-based emollients. These and other manufacturers have brought new technology to bear to address formulators' petrochemical and silicone sourcing challenge in personal care.

In conclusion, the last several years have seen a significant movement to identify sustainable solvent replacements in a variety of markets and application areas. Importantly, the products highlighted in this article have found success in the marketplace because they have overcome significant dichotomies: performance *with* low/no VOC; performance *with* environmental, health and safety fitness; performance *neither with* petrochemicals *nor* silicones. Ingredient manufacturers celebrate customers' ascending performance and sustainability goals. These goals allow the supplier community to demonstrate the power and economy of biotechnology that is no longer emerging, but has arrived.

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Table 3. Examples of cleaning chemicals for specific applications (From Separation Anxiety, page 550)

Type of cleaner	Mechanism(s) of action	Common compounds	Advantages	Disadvantages
Caustic or alkaline cleaners	Saponification, neutralization, dispersion of colloidal material, hydrolysis, solubilization.	Sodium hydroxide, soda ash, phosphates, hypochlorite, potassium hydroxide.	Inexpensive; effective for some inorganic and many organic foulants, including protein-based foulants.	Functions as sequestering agent, aiding solubilization of soap residues by chelating Ca and Mg ions.
Acidic cleaners	Solubilization, pH regulation, dissolution of inorganic precipitates, acidic hydrolysis of certain macromolecules.	Nitric acid, hydrochloric acid, phosphoric acid, citric acid.	Can dissolve precipitates formed during cleaning. Removes mineral deposits. Good for heat-denatured protein residues.	Strong acids impact pH of solutions; cannot use chlorine because of potential release of chlorine gas and damage to membranes.
Sequestrants or chelating agents	Chelation.	Ethylenediamine tetraacetic acid, citrates, sodium triphosphate, sodium tripolyphosphate, trisodium phosphate.	Destroys the otherwise tough, cross-linked Ca^{2+} -HA fouling layer.	The cleaning efficiency of EDTA is pH dependent.
Enzymatic cleaning agents	Cleaves specific peptide bonds in proteins, leading to disassembly of the protein structure.	Alkaline proteases, lipase.	Operates in mild conditions, allows for reduction in chemical usage, lowers energy costs, produces biodegradable effluents.	Unstable in some cases; enzymatic residues must be controlled to prevent further fouling.
Surfactants or wetting agents	Emulsification, dispersion, surface conditioning.	Mainly nonionics and anionics, although cationic surfactants can be used as sanitizing agents as well as detergents with acid cleaners; low-foaming nonionic ethoxylated species can be combined with anionic surfactants, producing synergistic effects.	Can provide resistance to protein fouling over short periods; can emulsify fat, oil, and proteins in water; can improve rinsability; can disrupt functions of bacterial cell walls in cleaning fouling dominated by formation of biofilms.	Operational time must be sufficient to ensure the outlet stream is clean from permeate and foulants, especially for solutions containing surfactants; some surfactants can adsorb strongly to membrane surfaces, causing swelling of membranes.
Oxidants	Oxidation; disinfection through microbiocidal activity.	Sodium hypochlorite, hydrogen peroxide, and peracetic acid.	Reduces biofilm growth; easy to rinse off.	Can cause membrane disintegration.
Sanitizers and disinfectants	Used at the end of the cleaning cycle or with storage.	Hydrogen peroxide, hypochlorites, sodium metabisulfite, sodium bisulfites, methylene bithiocyanate, guanidine hydrochloride, urea, zinc dimethyldithiocarbamate, cetyltrimethylammonium bromide.	Sodium bisulfites work as reducing agents with high effectiveness at pHs below 4.	Adsorption is a concern in food and bioprocess applications.

Sources: *Membrane Technology*, Ch. 10, Membrane fouling and cleaning in food and bioprocessing, Hongyu Li and Vicki Chen, Elsevier Ltd., 2010; Xiafu Shi et al., Fouling and cleaning of ultrafiltration membranes: a review, *Journal of Water Process Engineering*, 2014.

AOCS Board Petition to Nominate

For each annual election of AOCS Governing Board officers, the membership may nominate up to four additional member-at-large candidates by petition. Petitioned candidates receiving at least 50 AOCS member signatures will be added to the ballot approved by the Governing Board. Preference will be given to the first four petitioned candidates with at least 50 signatures. Petitioned nominations must be received at the AOCS Headquarters no later than **October 30, 2015**.

Petition forms can be obtained by visiting www.aocs.org/BoardPetition. Please mail or fax completed petitions with at least 50 AOCS signatures to:

AOCS Nominations and Elections Committee
P.O. Box 17190
Urbana, IL 61803-7190 USA

Fax: +1 217-693-4852
Attn: Benjamin Harrison



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DSM Nutritional Products Ltd. (Kaiseraugst, Switzerland) and Evonik Nutrition & Care GmbH (Essen, Germany) are now partners in an algae-derived omega-3 pet food and aquaculture feed joint venture. "The aim is to meet the increasing demand for omega-3 fatty acids by harnessing naturally occurring marine algae using sustainable, biotechnological processes based on natural, nonmarine resources," Evonik said in a written statement.

■ ■ ■

Ecuador, which is responsible for about two-thirds of the global sales of fine-flavor cocoa, aims to industrialize its production through the use of large-scale cluster plantations, according to ConfectioneryNews.com. Fine-flavor cocoa accounts for about 5% of the world's cocoa supply and is made from beans with a floral or fruity flavor. Visit <http://tinyurl.com/Ecuador-Cacao> for more information.

■ ■ ■

Golden Peanut and Tree Nuts, a subsidiary of Archer Daniel Midland Co. (Chicago, Illinois, USA), announced in August that it would significantly expand the capacity of its peanut oil processing facility in Dawson, Georgia, USA. The company noted in a news release that the United States currently is a net importer of peanut oil, adding that the project is scheduled for completion in the second quarter of 2016.

IN MEMORIAM

Lynn A. Hawkins, 73, died on August 16, 2015, at Baptist Desoto Hospital in Southaven, Mississippi, USA.

Dr. Hawkins joined AOCS in 1969 and was a member of the AOCS Examination Board from 1998–2012. He was a founding partner of Tenent and Associates, which later acquired Barrow-Agee Labs LLC in Memphis.

Dr. Hawkins is survived by his wife, two sons, two step-daughters, one sister, one brother, five grandchildren, and one great-grandchild.

We offer our condolences to his family, including his son Michael, who is also an AOCS member.

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NEWS & NOTEWORTHY



Livestock feed will overtake biofuels

Demand for livestock feed will outpace demand for grain-based food for human consumption and biofuel production within the next decade, according to the United Nations' Food and Agriculture Organization (FAO) and the Organization for Economic Co-Operation and Development (OECD).

The FAO/OECD annual "Agricultural Outlook" report found that during the past decade, livestock feed was responsible for a 36% growth in grain consumption, but over the next decade, the demand for livestock feed is expected to reach 70%. (Visit www.agri-outlook.org/publication to download the report.)

Prices for crops and livestock products showed diverse trends in 2014. Among crops, two years of strong harvests put further pressure on prices of cereals and oilseeds. Tighter supplies due to factors including herd rebuilding and disease outbreaks supported record high meat prices, while the prices of dairy products dropped steeply from historic highs. Further adjustments to short-term factors are expected in 2015, before the medium-term drivers of supply and demand take hold.

In real terms, prices for all agricultural products are expected to decrease over the next 10 years, as on-trend productivity growth, helped by lower input prices,

outpaces slowing demand increases. While this is consistent with the tendency for long-term decline, prices are projected to remain at a higher level than in the years preceding the 2007–08 price spike. Demand will be subdued by per capita consumption of staple commodities approaching saturation in many emerging economies and by a generally sluggish recovery of the global economy.

The major changes in demand are in developing countries, where continued but slowing population growth, rising per capita incomes, and urbanization all increase the demand for food. Rising incomes prompt consumers to diversify their diets by increasing their consumption of animal protein relative to starches. For this reason, the prices of meat and dairy products are expected to be high relative to the prices of crops, while among crops the prices of coarse grains and oilseeds used for feed should rise relative to the prices of food staples. These structural tendencies are in some cases offset by specific factors, such as a flat demand for maize-based ethanol.

Lower crude oil (petroleum) prices are a source of downward pressure on food prices, principally through their impact on energy and fertilizer costs. Moreover, under the projected lower oil prices, the production of first-generation biofuels is generally not profitable without mandates or other incentives. Policies are not expected to lead to significantly higher biofuel production in either the United States or the European

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Union. On the other hand, a rise in the production of sugar-based ethanol in Brazil is expected to flow from the increase in the mandatory blending ratio in gasoline and the provision of tax incentives, while biodiesel production is being actively promoted in Indonesia.

In Asia, Europe, and North America, additional agricultural production will be driven almost exclusively by yield improvements, whereas in South America, yield improvements are projected to be complemented by additional agricultural area. Modest production growth is expected in Africa, although further investments could raise yields and production significantly.

Exports of agricultural commodities are projected to become concentrated in fewer countries, while imports become more dispersed over a large number of countries. The importance of relatively few countries in supplying global markets for some key commodities increases market risks, including those associated with natural disasters or the adoption of disruptive trade measures. Overall, trade is expected to increase more slowly than in the previous decade, while maintaining a stable share relative to global production and consumption.

The report notes that the current baseline reflects fundamental supply and demand conditions on world agricultural markets. "However, the Outlook is subject to a variety of uncertainties, some of which are explored by stochastic analysis," FAO/OECD said in an executive summary of the report. "If historical variations in yields, oil prices, and economic growth are projected into the future, then there is a high probability of at least one severe shock to international markets within the next 10 years."

Cargill opens its first canola refinery; second crushing plant in Canada

In July 2015, Cargill (Minneapolis, Minnesota, USA) opened its first canola refining plant in Clavet, Saskatchewan, Canada. The new facility has the capacity to refine one billion pounds of canola oil annually, making it the largest Cargill refinery in North America, according to the company.

Cargill also began production in July at a new canola-crushing plant in Camrose, Alberta, Canada. Calling it "state of the art," the company said the facility can process more than 1 million metric tons (MMT) of canola per year into about 600,000 metric tons (MT) of meal, which is almost double the amount of currently processed by the company's older plant in Saskatoon.

"The global demand for canola is on the rise, and we're targeting an increase to 26 MMT by the year 2025," noted Patti Miller, president of the Canola Council of Canada (CCC). "More than half of this will be processed in Canada for markets around the world. Cargill's new plant takes us a step closer to achieving this goal."

Demand for canola meal is also rising, thanks in part to recent research. For example, a new study supported by Agriculture and Agri-Food Canada (Sherbrooke, Québec) and the CCC found that canola meal can replace soybean meal and improve dairy cow productivity. The work was conducted by Glen Broderick of Broderick Nutrition and Research (Madison, Wisconsin, USA), Anthony Faciola of the University of Nevada (Reno, USA), and Louis Armentano of the University of Wisconsin (Madison, USA), and published in the *Journal of Dairy Science* (<http://dx.doi.org/10.3168/jds.2015-9563>, 2015).

Historically, soybean meal has been the most common protein source in dairy cattle production, in part because it has higher levels of crude protein than canola meal. In recent years, however, increased production of canola has led to a greater availability of canola meal as a protein supplement in livestock diets.

"But for a dairy cow, it's not necessarily about how much crude protein is in the ingredient but what that protein is made of," said Carson Callum, CCC's canola meal manager.

The new findings show that diet formulation using canola meal instead of soybean meal at the same level of dietary crude protein will increase milk yield and milk protein content. It can also increase nitrogen utilization, potentially lowering environmental nitrogen excretion.

This research also supports a meta-analysis conducted in 2013 by Agriculture and Agri-Food Canada. That review of 49 different studies found that inclusion of canola meal improved milk yield by 0.64 kg per day and increased protein yield when compared to other protein ingredients fed at the same level.

In related news, CHS Inc. (St. Paul, Minnesota, USA), a farmer-owned cooperative, announced it has acquired Northstar Agri Industries canola processing and refining plant near Hallock, Minnesota, USA, from PICO Northstar Hallock LLC, a majority-owned subsidiary of PICO Holdings, Inc. The Hallock canola plant processes more than 400,000 MT of canola annually into canola oil and canola meal.

West Central Cooperative announces \$27 million expansion

West Central Cooperative has announced plans for a \$27-million, multi-year project to increase soybean-processing capacity and build additional grain storage at its Ralston, Iowa, USA, complex. At full capacity, the expansion would create demand for an additional six million bushels of soybeans each year to produce SoyPlus[®], a high-bypass protein dairy feed ingredient.

The company plans to increase the current plant's capacity by up to 50% with production coming online in phases. When complete, the project would include an additional line of mechanical presses, soybean oil treatment, load-out access, and nearly three million bushels of additional soybean storage. New production volumes are slated to be available as early as fall 2016.

A new tool could pinpoint when fingerprints were left behind

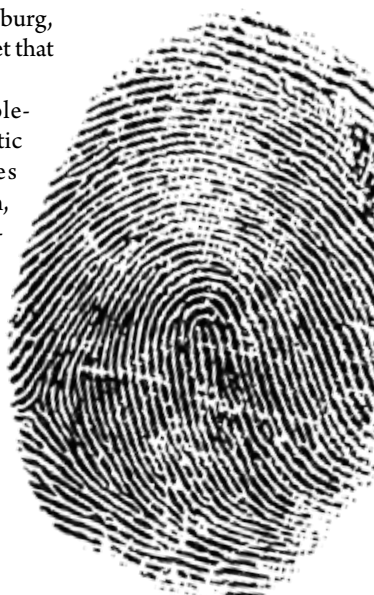
The crime scene investigators on television seem able to solve any mystery thanks to a little science and a lot of artistic license. Now there is a real-life technique that could outperform even fictional sleuths' crime-busting tools. And it involves palmitic acid, the most common fatty acid found in plants and animals.

Law enforcement officials have long relied on fingerprints left behind by criminals to help solve cases. In addition to patterns of whorls, loops, and arches specific to individuals, prints can also yield clues as to the owners' age and gender, as well as materials—such as explosives or make-up—that they may have touched. But determining just how long these residues have been at a crime scene is one aspect that has remained a challenge. The ability to date fingerprints would allow police to more easily rule certain suspects in or out of their investigations. Shin Muramoto and colleagues at the National Institute of

Standards and Technology in Gaithersburg, Maryland, USA, wanted to find a way to meet that need.

The researchers studied various molecules in fingerprints and found that palmitic acid migrates away from print ridges at a predictable rate. Based on this diffusion, the scientists could estimate how old a fingerprint was. Their findings apply to prints up to four days old, but they plan to expand that window to 10 days.

Their work appeared in *Analytical Chemistry* (<http://dx.doi.org/10.1021/acs.analchem.5b02018>, 2015). A video describing their method is available at <http://tinyurl.com/palmitic>.



IN MEMORIAM

George C. Cavanagh, an AOCS member since 1946 and AOCS president in 1969–1970, died at the age of 99 on August 20, 2015, in Claremont, California, USA.

Dr. Cavanagh was born in North Bend, Oregon, USA, in 1915, and graduated from Pomona College in 1938. He was hired by Producers Cotton Oil in Fresno, California, USA, in 1939 and volunteered for the US Army in 1942. He returned to work at Producers in 1946 after leaving military service. He left Producers for Ranchers Cotton Oil, also in Fresno, in 1951. Ranchers was using solvent extraction, which led to work that resulted in seven patents in vegetable proteins, solvent refining, and fractional crystallization of vegetable oils.

He was active on many AOCS committees and was general chairman of the joint AOCS–JOCs Annual Meeting in 1979 in San Francisco, and remained active in the organization after his retirement from Ranchers in 1984.

He is survived by his wife of 71 years, Virginia S. Cavanagh, son Douglas Cavanagh, who is also an AOCS member, daughter Margaret Merrill, and three grandchildren.■

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Cleaning well without hardness sequestration in consumer products

Dave Kitko

During the past four decades, changing consumer habits and practices, environmental legislation, regulatory requirements, and raw material costs have prompted a constant flow of innovation in consumer cleaning products. Many of these innovations have been associated with positive control of the impact of calcium (Ca) and magnesium (Mg) ions and their interactions with soils and surfactants.

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- In addition, falling wash temperatures, washing machine redesign, improving water quality, and changing consumer needs have given rise to surfactant system optimizations and the appropriate inclusion of a select set of additives to maintain strong wash performance.

- This article reviews the history behind those developments and takes a look at where the optimum is today.

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Where is the optimum today? Answering that question requires a step back in time.

FLASHBACK TO THE MID '70S AND '80S

By the mid-1970s, branched alkyl benzene sulfonates, such as dodecylbenzene sulfonate, had been eliminated from laundry detergents in North America, Western Europe, and Japan due to their lack of biodegradability. Sodium tripolyphosphate (STPP) was under fire from environmentalists, and sodium nitrilotriacetate (NTA) was the lead candidate to replace STPP. The STPP level had fallen in North America from its previous high of 50% to 35%. In North America and Western Europe, consumers were seeking to lower wash temperatures to reduce fabric damage and save on energy costs. Hypochlorite bleach was intermittently used on white loads to improve stain removal and whitening performance. More frequent use of hypochlorite resulted in yellowing, as the hypochlorite oxidized fluorescent whitening agents (FWA).

In the hand dishwashing product domain, Procter & Gamble (P&G) Co. had risen to dominance in North America after adding a Mg salt to its products to dramatically improve food grease and cooking oil removal (Dawn®, Joy®). Note: Hardness ions are not always a negative. In the personal cleansing domain, the negative influence of Ca and Mg on lather performance, mildness, and soap-scum deposition initiated a move toward liquid body washes and the dramatic growth of Unilever's Dove® brand, which employed cocoyl isethionate as a major surfactant component and had a lower pH in use. The product minimized soap scum and, importantly, improved mildness by limiting the deposition and penetration of Ca laurate and Ca oleate into the skin, preventing subsequent dry out. In the auto dish product domain, there was no meaningful alternative to STPP in products with hypochlorite-based bleach systems, and typical tub and shower cleaners continued to rely on high-pH, polyphosphate-based formulations.

It was an intensely competitive time in the home laundry world across geographies. Major detergent companies pursued organic peroxyacid bleach technologies as color-safe alternatives to hypochlorite, as whiteness losses increased with falling wash temperatures, and hydrogen peroxide-based bleaches only worked at high temperatures. NTA had been removed from consideration due to its alleged adverse health effects, and 4A zeolite had become the phosphate replacement in powder detergents. The initial zeolite-based powders were disappointing, because they left detergent residues on clothes and were noticeably weaker in performance compared to their polyphosphate precursors. Lower wash temperatures had forced the removal of tallow alcohol sulfate from powders, and alkylethoxysulfates were forced out of powders, as they were the source of organic plumes associated with spray-drying towers.

While powder forms struggled, laundry liquids seized the opportunity and gained important market share in North America. In the rework of the zeolite-based powders, reduced zeolite particle size helped overcome the residues, and a low-molecular-weight polyacrylate polymer (PAA) and low-molecular-weight polyethylene glycol (PEG) combined to produce a clay/soil dispersant system that significantly offset cleaning negatives in P&G's zeolite-based powders. This reformulation was in part driven by the research and discovery work that led to the launch of Liquid Tide® where the requirement to equal Tide powder's performance on mud/dirt cleaning and whiteness maintenance had been achieved by D. Rubingh and E. Gosselink's invention, US4659802 [1] and P&G's implementation of polyethylene pentamine polyethoxylate (PN5EO20) dispersant as a key component in the formulation. In Western Europe, tetraacetythylenediamine (TAED) was the peroxide activator of choice, allowing a move away from the "boil wash" to the 60 °C wash temperature.

OBSERVATIONS FROM RELATED RESEARCH

During research to comprehend Mg's role in improving grease cleaning more fully, P&G researcher T. Cripe made some important observations. Adding Mg to the Dawn surfactant system of the day did not decrease the thermodynamic interfacial tension on purified triolein; it actually increased it slightly. However, the addition of Mg improved the dynamic interfacial tension with beef fat grease. It was subsequently shown that Mg addition increased the critical micelle concentration, which was the underlying cause for the importantly reduced dynamic interfacial tension and consequently improved grease removal from a polypropylene cup surface.

Subsequent work looking at ways to increase Dawn's grease-cleaning performance uncovered another important point: At higher in-use pHs (9.0), Dawn would actually lose efficacy in harder water. The underlying cause was a Ca soap-scum formation on the surface of the grease that occurred when the Ca ions complexed with the ionized residual fatty acids in the soil and formed a surface deposit that shut down cleaning. An additional observation that appeared irrelevant at the time, but speaks to the role of dispersants in cleaning was that cationic oligomeric polypropoxylate polyethoxylates (tetronics) could inhibit grease re-deposition onto plastic items in the context of hand dishwashing. The important lessons here were that free Ca could negatively impact lipid removal at higher pHs, and that grease re-deposition can occur onto hydrophobic surfaces in a cleaning processes.

IMPORTANT LAUNDRY INNOVATIONS COME TO FRUITION

As the 1980s drew to a close, Tide with Bleach powder was successfully launched behind the peroxide bleach activator, nonanoyloxybenzenesulfonate (NOBS) and protease enzyme. Tide® Liquid continues its growth behind excellent cleaning from a well-designed ternary surfactant system, lauric and oleic acids as calcium control agents along with citric acid, which are critical to superior enzymatic performance, but require a key balance of pH and the surfactants to insure the Ca soaps formed are kept from depositing on fabrics. The role of the PN5EO20 dispersant was likely a critical component in this function as well.

This period was capped with Kao Corp.'s launch of a very strong-performing compact powder detergent, Attack®, which used a unique cellulase enzyme to support a claim of cleaning inside the fiber. The consumer convenience associated with this product form drove all major

detergent manufacturers to follow, and facilitated the complete removal of STPP in these geographies. In Western Europe, P&G strengthened the performance of their nil P products by including an acrylic acid/maleic acid copolymer and a clay dispersant based upon a methyl quaternized hexamethylene diamine polyethoxylate [2]. These components were critical for strong performance through the serial dilutions that constitute the rinse cycle in washing machines in Europe.

FURTHER INSIGHTS ON COLD WATER CLEANING

During the early 1990s, with the move to nil-phosphate powders complete, Kao researchers publish several articles on the cleaning problems in the Japanese home laundry process where wash temperatures are typically 25 °C and below. The first article [3] demonstrates the residual sebum build up on cotton undershirts and the correlation with whiteness loss using a simple laundry detergent with linear alkyl benzene sulfonate (25%) as surfactant, zeolite (20%) and sodium silicate (10%) as builder, sodium carbonate (10%), and polyacrylate/PEG (1% each) as the dispersant system. The residual sebum level built to an average of 27.4 mg/g on fabric before the 15th wash and 17mg/g after the 15th wash. A subsequent article [4] tracked the residual soil buildup on men's dress shirt collars. The sebum level on collar soils climbed to 184mg/g before wash and 160mg/g after the 15th wash. The protein content of these collar soils was also substantial (a 10mg/g level after the last wash cycle) and further darkened the soil. Kao identified this as unsatisfactory cleaning.

In 1994, S. Murata and S. Tsunetsugu, researchers at Procter & Gamble Far East, Inc. reported at the Japan Soap and Detergent Association meeting their findings on the benefits of hydrophobic peroxy acids in reducing this sebum-based whiteness loss. Specifically, the pernonanoic acid from the NOBS/peroxide system and a preformed peroxy acid, nonanalamidoperoxyadipic acid, provided important whitening benefits and an increase in residual lipid removal in the wash cycle using a simple LAS/nonionic surfactant system and the typical zeolite/polyacrylate/PEG builder/dispersant system.

In 1995, K. Asano and K. Sarnaik of P&G Far East conducted a wash/wear test similar to that of Kao's earlier research, employing only currently marketed products, Ariel®, Attack®, and Hi Top®. Their results showed that all three detergents had residual lipid and protein levels nearly an order of magnitude lower on undershirts than the Kao researchers had observed, and an even greater reduction was observed on collar soils. The study also identified Ca-based surfactant residues as an important component of this residual soil and showed that the remaining lipid components were the higher melting ones. At these reduced residual soil levels, there was still an important whiteness loss without the hydrophobic bleaching component on undershirts, and visible collar soil residuals for all three products. In response to these determinations, P&G Far East researchers pursued the incorporation of a methyl cellulose-based soil release polymer from Shin Etsu Chemical Co., Ltd. and combined it with a cationic co-surfactant to insure lower dynamic interfacial tension with body lipid [5]. This innovation greatly reduced the visible collar soil buildup on men's polyester/cotton dress shirt collars. However, the formulation was discontinued after casual dress became more popular and the cleaning of dress shirts became less of a concern.

PUSH FOR IMPROVED CLEANING

As the 1990s drew to a close, a big effort was put behind finding new surfactants with good surface activity (measured by low dynamic IFT with body lipid), cold water solubility, and hardness tolerance. Fatty alcohol ethoxy sulfates (FAES) were the best all-around choice, but not readily incorporated into granular detergents. Additionally, the move into a wider global domain where higher soil loads were often encountered renewed efforts to identify improved dispersant technologies. There was also high interest in stain-removal technologies, such as metal-based bleach catalysts, and a broader enzyme set that supported stronger advertising claims. Lauryl ethoxy sulfates (LES) became the primary surfactants in hand dishcare, body wash, and shampoo domains. Cosurfactants such as cocoamidopropyl betaine (CAPB) and cocoamine oxide were used to achieve the desired lather/suds profiles in these products. Bathroom tub and shower cleaners moved to low pH formulations with CAPB/nonionic surfactant systems and solvents to dramatically improve soap-scum removal, as returning the soap scum to fatty acid and free Ca/Mg ions greatly improved ease of its removal. Chemical suppliers worked in earnest to develop cost-effective Ca/Mg sequestrants that were more robust than sodium citrate/citric acid.

P&G researchers identified two structural variations on the leading laundry anionic surfactants—mid-chain methyl branching on alcohol sulfates and a similar branching pattern on high 2-phenyl LAS—that achieved the desired cold water solubility, hardness tolerance, and surface activity. They successfully collaborated with Shell Chemical Co. to bring the alkyl sulfate version into the marketplace initially for laundry granule applications and later for reapplication in laundry liquids. The LAS variant met with commercialization difficulties and failed to enter the marketplace. New dispersant technologies were identified by Unilever; hydrophobically modified acrylate copolymers; P&G in collaboration with BASF Corp. and Nippon Shokubai Co., Ltd., ethoxylated polyethyleneimines [6] and BASF and P&G, a zwitterionic dispersant from bis-hexamethylenediamine polyethoxylates [7] that was quaternized and partially sulfated; National Starch/AkzoNobel, styrene/acrylate copolymers all of which found successful application in laundry liquids; and Nippon Shokubai, low molecular weight maleic acid/acrylic acid copolymers for granule applications [8].

Metal catalysts were deemed too high a risk for fabric damage in laundry applications, but were successfully deployed in auto dish products. They facilitated a move away from hypochlorite bleach to peroxide/activator bleach systems combined with enzymes to deal with higher soil loadings. Polyphosphate replacements were yet to come.

COST PRESSURES/GROWING SUSTAINABILITY CONCERNS

The rising raw material prices and growing environmental awareness of the early to mid-2000 time period, coupled with a focus upon importantly reducing a product's "carbon footprint" and products being derived from "sustainable raw materials," facilitated laundry liquid detergent compaction. The cost savings for packaging and product distribution easily offset increased costs for higher solvent loadings and brought total delivered costs for laundry liquid products below powder detergents. This dramatically changed the North American laundry detergent marketplace, with only truly superior performing laundry powders like Tide with Bleach able to sustain a reasonable market share.

Over the last decade, detergent formulators began to recognize that truly effective dispersant systems could help offset the rapidly rising costs for surfactant components driven by the unprecedented rises in prices for petrochemical-based components and naturally derived hydrophobes from palm and coconut sources. The ethoxylated polyamine-based materials appear to stabilize the dispersions of both particulates and surfactant-emulsified hydrophobic components in the wash process, preserving reasonable cleaning at reduced surfactant levels. Hydrophobically modified polyacrylate co-polymers were also observed to be capable of stabilizing these dispersions and aid in lipid/oil cleaning from fabrics. These benefits allow formulators to reduce surfactant levels and maintain reasonable cleaning performance. The continuing growth of front loading, high-efficiency washing machines further enables this surfactant reduction as the reduced water volumes require less total surfactant used in the cleaning process. The only watch-out for products for these machines is that the surfactant system be lower sudsing.

SO WHERE IS THE OPTIMUM TODAY?

Looking back over the material innovations and product formulation changes over the past years, it becomes apparent that in the laundry cleaning task good performance can be achieved across the oily/lipid to particulate soil continuum by the following choices.

1. Lowering the wash pH to the 7.5–8.5 domain to prevent Ca soap skins on lipid/oily soils which shut down removal.
2. Choosing a non-precipitating, cold water soluble surfactant combination with free hardness present. This combination must have a low dynamic interfacial tension with lipid across the water hardness domain.
3. Including a dispersant component that suspends both oily/lipid and particulate soils in the wash process.
4. Selecting the enzyme combination that works well in the surfactant system above.

There is increased complexity when moving to the peroxide/activator product domain as the wash pH must remain in the 10 plus area to facilitate peracid generation. The criteria for the surfactant system remains and the dispersants that function best here must mitigate the Ca soap skin formation and be compatible with the peracid bleach. The best candidates for these

product formulations are the quaternized polyamine polyethoxylates and hydrophobically modified polyacrylate polymers.

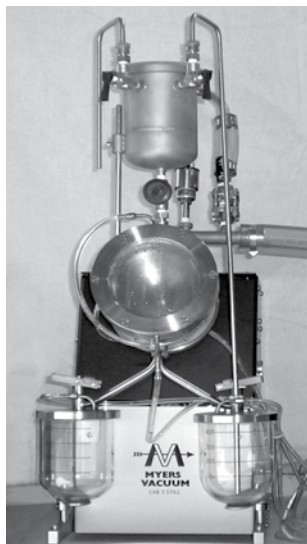
Chemical suppliers have commercialized a series of Ca sequestrants that are superior to citrate in their efficiency. BASF markets methylglycine diacetate (MGDA), Akzo Nobel markets glutamic acid diacetate (GLDA), and Bayer AG markets iminodisuccinate. Their desirability to detergent formulators will be determined by their cost effectiveness or how much of an improvement in cleaning performance they provide. The best point of application has been as a polyphosphate replacement in autodish care products.

Reflecting across the consumer cleaning product domain, managing the Ca soil component interactions is key to acceptable performance. This does not always require Ca sequestration. It does require choices in formula design that mitigate the Ca interactions with soil components (fatty acids, protein fragments, inorganic particulates) and prevent anionic surfactant precipitation.

Dave Kitko is a retired chemist who worked for the Procter & Gamble Company for 34 years across the company's family of consumer products—from laundry additives, hard surface cleaners, fabric softeners, and laundry detergents to hair shampoos and conditioners. He led P&G's upstream research in surfactants, polymers, and bleach technologies from 1995–2003 and returned to establishing an upstream program in hair care from 2003, until his retirement in October 2010. Dave currently consults independently, and acts as technical consultant in the chemicals and consumer products industry. He can be contacted at davidkitkoph.d@gmail.com.

Further reading

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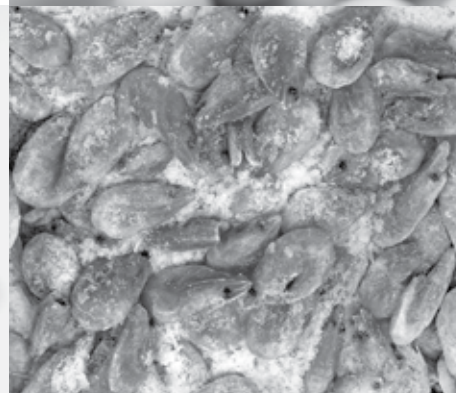


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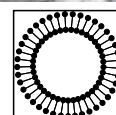
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On August 8, 2015, Argo Fuels in Logan, Utah, USA, began production using its new pilot NanoCatalyst supercritical processor. The company describes its process as “scalable and portable” on its website at www.argofuels.com. In fact, a processor with a six-million-gallon-per-year output capacity can be packed into a single 40-foot shipping container, according to Argo CEO Ray Dellinger. The processor can reach full reaction in four minutes, he noted, and can use feedstocks with up to 100% free fatty acid content.



San Francisco (California, USA) will replace petroleum diesel with renewable diesel in its municipal fleet of approximately 1,500 diesel vehicles by the end of 2015, according to Mayor Edwin M. Lee. Lee spoke on July 21, 2015, at a sustainability conference in Vatican City (Italy). Currently, most of the fleet uses B20—which is 20% biodiesel and 80% petroleum diesel—according to *Biodiesel Magazine*.

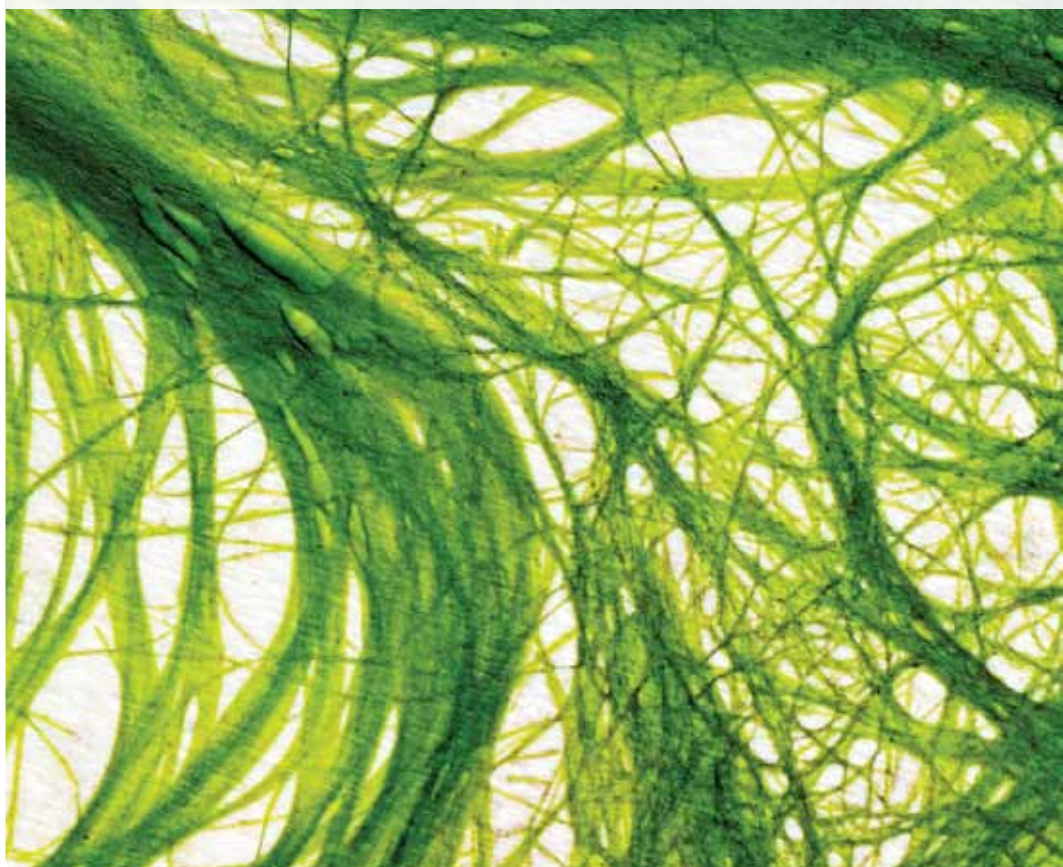


Red Rock Biofuels LLC (Fort Collins, Colorado, USA) will produce approximately three million gallons of low-carbon, renewable jet fuel per year for FedEx Express, a subsidiary of FedEx Corp. (Memphis, Tennessee, USA). The agreement runs through 2024, with first delivery expected in 2017. FedEx joins Southwest Airlines in purchasing Red Rock’s total available volume of jet fuel. Red Rock’s first refinery, funded in part by a \$70 million Title III Defense Production Act grant from the US Departments of Agriculture, Energy, and Navy, is scheduled to break ground this fall in Lakeview, Oregon, USA.



Renewable Energy Group Inc. announced in July 2015 that it will invest \$31 million to upgrade and enhance the company’s Danville, Illinois, USA, biodiesel refinery. Upgrades will include the addition of biodiesel distillation capabilities to the 45-million-gallon-per-year biorefinery. ■

ENERGY



Stress-free algae

Some algae like *Chlamydomonas reinhardtii* (or “Chlamy,” as it is known to its large research community) produce energy-dense oils or lipids when stressed, and these lipids can then be converted into fuels. However, researchers walk a fine line in not killing the goose that lays the golden eggs; in this case, stressing the algae just enough to produce lipids but not enough to kill them.

A team led by scientists from the US Department of Energy Joint Genome Institute (DOE JGI) analyzed the genes that are activated during algal lipid production, and in particular the molecular machinery that orchestrates these gene activities inside the cell when it produces lipids.

“We know how to stress the algae,” said the study’s first author Chew Yee Ngan of the DOE JGI. “What we don’t know is how to keep the algae alive at the same time, until now.”

As part of the DOE’s efforts to study algae for energy and environmental applications,

the DOE JGI has published over 75% of all publicly available algal genomes. One of these is the Chlamy reference genome, which was released in 2007. Until now, very little has been known about the protein factors that can regulate lipid production. To find out more about them, the team cultured Chlamy cells and starved them of nitrogen or sulfur, both of which are stress conditions to which Chlamy responds by producing lipids. They then analyzed the complex of DNA and proteins known as chromatin that defines what genes are being activated, as well as the expression profiles or transcriptome, and compared these to nonstressed Chlamy cells.

“We’re looking for changes in starved cells vs. cells that are happily growing,” Ngan explained. Through careful analysis of genome-wide data sets, they narrowed down their search to identify two transcription factors that appeared to play a pivotal role in lipid accumulation and then studied one of them, *PSRI*, in detail.

“In studying the chromatin modifications, we can read out changes in the proteins

bound to DNA on a genomewide scale and then specifically target those genes whose regulation profiles are changed under lipid-producing conditions,” said study co-author Axel Visel, DOE JGI deputy for science programs.

“The study also demonstrated how cells can be tricked into producing lots of lipid without dying of starvation by overexpression of *PSR1*,” he added, “which is a strategy that could potentially be applied in other industrial algal species better suited for large-scale biofuel production.”

Corresponding author Chia-Lin Wei pointed out that the study also demonstrated a strategy for the integration of epigenomic and gene expression data and methods, for example, the mapping of molecular tags that sit on top of the actual DNA sequence and affect its function.

“Such functional interrogation of the genomes . . . is expected to be widely applicable to more plants and fungi whose gene regulatory pathways still prove elusive,” said Wei, adding that Ngan and others at the DOE JGI are continuing this work in many other energy-related species.

The study appeared in *Nature Plants* (see <http://tinyurl.com/DOE-algae>; 2015).

Iranian scientists testing biodiesel from flaxseed oil

Renewable energy targets are now a defining feature of the global Researchers at the Islamic Azad University in Tehran are testing flaxseed oil as a feedstock for biodiesel, according to PressTV, Iran’s international news network.

“Engine and field tests of the synthetic green fuel are currently being evaluated,” Mehdi Alami, an analytical chemistry graduate student at the university, was quoted as saying. He also said that the research suggested flaxseed contains 22% oil; work from 2009 by Lithuanian researchers found that flaxseed seeds contained 32.2% oil (see <http://tinyurl.com/flaxseed>).

Flaxseed (*Descurainia sophia*) is sometimes called tansy mustard and is a member of the mustard family. It is native to Europe and northern Africa, and is found in 48 of the 50 US states (excluding Alabama and Florida). It also has been introduced in South America, Asia, southern Africa, and New Zealand. It can be grown without cultivation, herbicides, or irrigation, according to the Forest Service of the US Department of Agriculture.

India’s Central Railway continues biodiesel test

Mumbai-based Central Railway (CR)—which has a fleet of 4,000 diesel locomotives—has concluded its first year of biodiesel production, according to *The Economic Times* newspaper. Nearly 20,000 liters of the fuel was manufactured at the company’s workshop in Parel, the report noted.

“We have successfully achieved our target to produce 1,500 liters of biodiesel per month (since August 2014) from edible oils and [are] looking ahead [to] an uptick in this production, though we are facing [a] shortage of raw material like used edible

oil, which is its primary source,” an official from the workshop was quoted as saying.

The railway soon will approach hotel owners and edible oil manufacturers to donate used edible oil, according to CR’s chief public relations officer.

NREL survey shows dramatic improvement in B100 biodiesel quality

The latest national survey of 100% biodiesel (B100) “blend stock” samples by the US Department of Energy’s National Renewable Energy Laboratory (NREL; Golden, Colorado, USA) found that 95% of the samples from 2011–12 (the most recent year surveyed) met ASTM International fuel quality specifications. The ASTM standards serve as guidelines for industry and are designed to ensure quality at the pump for consumers—along with reliable operation of vehicles powered by biodiesel blends.

“The survey showed a major improvement over results from previous years,” said NREL Senior Chemist Teresa Alleman. “In our 2007 survey of B100 biodiesel, less than half of the samples met quality specifications. More stringent quality requirements, along with the voluntary BQ-9000 quality management program, are among the reasons for this marked improvement.” B100 is not commonly used as a fuel but is blended with petroleum diesel, typically in blends up to 20%, and has been part of the industry’s steady growth in the past decade. B100 production increased from 27.9 million gallons in 2004 to more than 1 billion gallons in 2012.

For the most recent survey conducted from August 2011 to February 2012, NREL researchers collected fuel samples from 53 producers and 14 terminals from across the United States. Terminals from the East and West Coasts, the Rocky Mountain region, and the Midwest were tested for a range of critical properties, such as free and total glycerin content, metals content, and cloud point, all of which could have an immediate impact on operability.

To ensure product quality, ASTM published the first B100 quality standards in 2002. ASTM International does not enforce fuel quality, but rather develops and delivers international voluntary consensus standards. NREL is among the organizations participating in the development of standards. ■

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<input type="checkbox"/> Agricultural Microscopy	\$16	<input type="checkbox"/> Lipid Oxidation and Quality	\$10	<input type="checkbox"/> Asian	\$15	<input type="checkbox"/> Indian	\$10
<input type="checkbox"/> Analytical	\$15	<input type="checkbox"/> Phospholipid	\$20	<input type="checkbox"/> Australasian	\$25	<input type="checkbox"/> Latin American	\$15
<input type="checkbox"/> Biotechnology	\$20	<input type="checkbox"/> Processing	\$10	<input type="checkbox"/> Canadian	\$15	<input type="checkbox"/> USA	FREE
<input type="checkbox"/> Edible Applications Technology	\$20	<input type="checkbox"/> Protein and Co-Products	\$15	<input type="checkbox"/> European	\$25		
<input type="checkbox"/> Health and Nutrition	\$20	<input type="checkbox"/> Surfactants and Detergents	\$30				
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AOCS: Your international forum for fats, oils, proteins, surfactants, and detergents.

This Code has been adopted by AOCS to define the rules of professional conduct for its members.

AOCS Code of Ethics • Chemistry and its application by scientists, engineers, and technologists have for their prime objective the advancement of science and benefit of mankind. Accordingly, the Society expects each member: 1) to be familiar with the purpose and objectives of the Society as expressed in its articles of incorporation; to promote its aim actively; and to strive for self-improvement in said member's profession; 2) to present conduct that at all times reflects dignity upon the profession of chemistry and engineering; 3) to use every honorable means to elevate the standards of the profession and extend its sphere of usefulness; 4) to keep inviolate any confidence that may be entrusted to said member in such member's professional capacity; 5) to refuse participation in questionable enterprises and to refuse to engage in any occupation that is contrary to law or the public welfare; 6) to guard against unwarranted insinuations that reflect upon the character or integrity of other chemists and engineers.

Scientists at the US Department of Agriculture Agricultural Research Service (USDA-ARS) and Oklahoma State University (both in Stillwater, USA) recently released a new variety of high-oleic, disease-resistant Spanish peanut called OLé. The peanuts, which were produced by traditional breeding methods, contain an oleic/linoleic acid ratio of 20:1. Consumption of oleic acid, a monounsaturated fatty acid, has been linked to cardiovascular benefits. In addition, the single double bond in oleic acid makes it more stable than polyunsaturated fatty acids like linoleic acid. As a result, OLé peanuts have an up to 10-fold increased shelf life compared with conventional varieties. In field tests, OLé plants were three times more resistant to Sclerotinia blight and seven times more resistant to pod rot than an older variety of Spanish peanut.



The Grocery Manufacturers Association (GMA) has filed a petition with the US Food and Drug Administration (FDA) to approve low-level uses of partially hydrogenated oils in specific food products, including pie crust mixes, pancake mixes, and shortenings, according to a GMA news release (<http://tinyurl.com/GMAtrans>). The petition follows the FDA's June 16 decision that partially hydrogenated oils, which contain trans fats, are no longer Generally Recognized as Safe (GRAS) and must be phased out of food products by 2018. However, food manufacturers can petition the FDA to allow continued use of the oils at low (food-additive) levels in specified products. The GMA argues that the small amounts of trans fats (0.01–3 g of trans fats per 100 grams of product) in the foods listed in the petition are as safe as naturally occurring trans fats in meat and dairy products.



Omega-3 fish oil pills may prevent the onset of schizophrenia in susceptible young people, according to a small study published August 11 in *Nature Communications* (<http://dx.doi.org/10.1038/ncomms8934>). Seven years ago, researchers led

FOOD, HEALTH & NUTRITION



Trans fats, but not saturated fats, linked to heart disease and death

Saturated fats are not associated with an increased risk of death, heart disease, stroke, or type 2 diabetes, according to a new meta-analysis published in *The BMJ* (<http://dx.doi.org/10.1136/bmj.h3978>, 2015). In contrast, trans fats are associated with a greater risk of coronary heart disease and death, but not of stroke or diabetes, the study indicates.

Dietary guidelines in the United States and elsewhere recommend limiting saturated fats to less than 10% of total calories. However, recent meta-analyses (e.g., <http://dx.doi.org/10.7326/M1377-1788>, 2014) have failed to find a link between saturated fats and coronary risk. The main dietary sources of saturated fats are animal products such as butter, milk, meat, and eggs.

Trans fats are produced industrially through the partial hydrogenation of liquid plant oils, and also can occur naturally in meat and dairy products derived from ruminant animals. Research (e.g., <http://dx.doi.org/10.1038/ejcn.2011.34>, 2011) has linked trans fats with an increased risk of heart disease and death, prompting the United States and other governments to take steps to remove partially hydrogenated oils from the food supply.

In the new study, researchers led by Sonia S. Anand at McMaster University (Hamilton, Canada) compiled data from 41 prospective cohort studies on associations between saturated fats and health outcomes. The researchers found no association between a higher intake of saturated fats and all-cause mortality, coronary heart disease, ischemic stroke, or type 2 diabetes in healthy adults. There was a trend for association of saturated fats with coronary heart disease mortality, but the association was not statistically significant.

On the other hand, data from 33 prospective cohort studies on trans fats and health outcomes revealed that a higher intake

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by G. Paul Amminger at the University of Melbourne, in Australia, enrolled 81 people aged 13–25 with early signs of schizophrenia in the trial. The participants took omega-3 supplements for 12 weeks, and one year later they had a reduced risk of being diagnosed with a psychotic disorder compared to a placebo group. Now, seven years later, the researchers followed up on the study participants. They found that only 10% of those taking omega-3 supplements ultimately developed schizophrenia, compared with 40% of the placebo group. ■

of trans fats (particularly industrial trans fats) was associated with a 34% increase in all-cause mortality, a 28% increased risk of coronary heart disease mortality, and a 21% increased risk of coronary heart disease. No associations were found between trans fats and ischemic stroke or type 2 diabetes.

The researchers acknowledge several important limitations of their study. For example, the meta-analysis did not consider the effects of replacement nutrients. Other studies have indicated that replacing saturated fats with polyunsaturated fats, but not carbohydrates, confers cardiovascular benefits. Also, the researchers note that observational studies can only establish association, but not causation, of nutrients with health outcomes.

Researchers uncover mechanism behind anti-inflammatory effects of HDL

When bound to high-density lipoprotein (HDL), a lipid signaling molecule called sphingosine 1-phosphate (S1P) can suppress inflammation in blood vessels, according to a recent paper in *Science Signaling* (<http://dx.doi.org/10.1126/scisignal.aaa2581>, 2015). The study provides insights into why HDL-cholesterol is considered “good cholesterol” and may suggest new avenues for treating atherosclerosis.

In the past, researchers have primarily linked the cardiovascular benefits of HDL to the lipoprotein’s ability to transport cholesterol from the tissues to the liver for excretion from the body. “While that is undoubtedly very important, there are additional actions of HDL that people have suspected but not fully characterized,” said Timothy Hla at Cornell University (Ithaca, New York, USA), corresponding author of the study, in an August 11 *Science Signaling* podcast (<http://tinyurl.com/HDLpodcast>). “Many of these actions we believe are due to S1P that’s bound to HDL.”

HDL particles are composed of apolipoproteins (e.g., ApoA-I, ApoA-II, or ApoM), antioxidant enzymes, various lipids such as cholesterol esters and triglycerides, and bioactive sphingolipids such as S1P. In the bloodstream, about 65% of S1P is bound to the ApoM+ subfraction of HDL, while the remainder is bound to a plentiful plasma protein called albumin. S1P binds to a receptor called S1P1, which is abundant in the endothelial cells that line blood vessels.

In the new study, Hla and his colleagues generated mice lacking the S1P1 receptor in their endothelial cells. Compared with wild-type mice, these mice produced more proinflammatory adhesion proteins,

such as ICAM-1 and VCAM-1. In contrast, mice that overexpressed the S1P1 receptor produced fewer of these proteins, suggesting that S1P1 protects against the vascular inflammation associated with cardiovascular disease.

In human endothelial cells treated with a pro-inflammatory cytokine (TNF α), S1P bound to ApoM+HDL suppressed the activation of a key inflammatory protein called NF- κ B. However, S1P bound to albumin did not have this anti-inflammatory effect, indicating the importance of HDL in the signaling process.

Finally, when hypercholesterolemic mice lacking the endothelial S1P1 receptor were placed on a high-fat diet (42% of calories from fat) for 16 weeks, they developed about twice the area of atherosclerotic plaques in the aorta than hypercholesterolemic mice that expressed the S1P1 receptor.

Taken together, these results suggest that HDL-dependent signaling through the S1P1 receptor protects the vessel wall from inflammation and atherosclerosis. According to Hla, the findings may partially explain the protective cardiovascular effects of HDL. The researchers are now working on finding ways to mimic HDL/S1P signaling pharmacologically, which could someday result in new therapies for atherosclerosis.



Scientists at Cornell University have identified another reason why HDL cholesterol is “good” for cardiovascular health.

Low-fat versus low-carb diets in the spotlight once again

A recent study in *Cell Metabolism* has reignited the controversy over whether cutting carbohydrates or cutting fat results in greater body fat loss (<http://dx.doi.org/10.1016/j.cmet.2015.07.021>, 2015). The research found that, in the short term (six days), cutting fat caused dieters to shed more body fat than cutting carbs. However, the authors of the paper acknowledge the study's limitations and its possible lack of applicability to real-world dieting.

Several randomized controlled trials ranging from three to 24 months in duration have indicated that low-carb diets cause significantly more weight loss than low-fat diets. But critics claim that the greater weight loss for low-carbohydrate diets can be explained by sodium and water imbalances or lean tissue loss, rather than fat loss. Also, most of these trials were outpatient studies, meaning that they relied on the participants' honesty in adherence to the diet.

So researchers led by Kevin Hall at the US National Institutes of Health (Bethesda, Maryland) conducted an inpatient study to compare the metabolic effects of cutting carbs versus cutting fat in 19 obese volunteers. The study participants resided in the NIH Metabolic Clinical Research Unit for two 11-day visits, separated by a two- to four-week "washout" period. They received a baseline diet (2,740 kcal) for the first five days of their visit, then for the next six days they consumed a 30% reduced-calorie diet (1,918 kcal). The calorie reduction came either from cutting fat (down to 17 g/day from 109 g/day) or cutting carbs (down to 140 g/day from 350 g/day). On the second 11-day visit, the reduced-fat and reduced-carb groups were switched, so that each participant tried both diets.

After six days, people on the reduced-carbohydrate diet lost more weight (on average, 1.85 kg, or 4 lb) than people on the reduced-fat diet (1.3 kg, or 2.9 lb). However, the reduced-fat group lost more fat (on average, 463 g, or 1.0 lb) than the reduced-carb group (245 g, or 0.54 lb).

The researchers acknowledge several limitations of their study. First, cutting 30% of calories from fat resulted in a very-low-fat diet (about 8% of calories from fat), whereas cutting 30% of calories from carbohydrates resulted in a lowered- (but not low- or very-low) carbohydrate diet (29% of calories from carbohydrates). Therefore, the data from the two diets may be difficult to compare. Second, the cost of performing inpatient studies in metabolic units limited the research to only 19 participants on a six-day diet. Whether these results can be extrapolated to larger populations for longer periods of time is questionable. And finally, the inpatient design of the study meant that diets were strictly controlled, with no access to outside food—a situation that bears no resemblance to real-world dieting. As such, the study cannot account for the effect of reduced appetite typically observed in low-carb versus low-fat diets.

"Our data tell us that when it comes to body fat loss, not all diet calories are exactly equal," says Hall in a news release dated August 13. "But the real world is more complicated than a research lab, and if you have obesity and want to lose weight, it may be more important to consider which type of diet you'll be most likely to stick to over time." ■



A small in-patient study claims that cutting fat is more effective than cutting carbs for short-term fat loss. CREDIT: Kevin Hall

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The parts of a research paper?

What your readers expect

Michael K. Dowd

Scientific papers are usually organized into Introduction, Methods, Results, Discussion, and Conclusions sections. This organizational format is easy for scientific readers to follow, as it mirrors the thought process most scientists go through when developing a project. Therefore, it has become standard at many journals, with occasional deviations. Sometimes Methods are placed at the end of an article or the Results and Discussion are combined, which the *Journal of the American Oil Chemists' Society* (JAOCs) allows but *Lipids* does not (except for method development reports). Similarly, theoretical and computational reports occasionally depart from this norm, and can be more effective when they do.

- Scientific papers are organized into sections that are easy for scientific readers to follow.
- This article summarizes the points that should be considered when writing the main sections of a research report.
- These typically include Introduction Methods, Results, Discussion, and Conclusion sections, as well as a separate Abstract.

THE ABSTRACT

Most journals require a separate Abstract that provides a concise summary of the project. In the abstract, the authors comment briefly on the problem being studied, the approach, the principal results, the most important discussion points, and the basic conclusions. The most important thing to remember is that the Abstract is a separate document and not part of the report. As such, it should be self-contained, without literature citations or references to tables or figures in the report.

Publishers and database managers use abstracts to index and catalog published work. Since abstracts are generally available to the public even when papers are not, journal readers often read through the abstracts first to decide which papers to read. In this respect, abstracts advertise the author's work.

THE INTRODUCTION: EXPLAINING THE PROBLEM

The Introduction broaches the topic under study and explains why it is important; in other words, it helps readers understand the purpose of the work. This section reviews related knowledge with appropriate citations. It explains why you have taken the approach you have, how your approach differs from previous efforts, and what you hope to accomplish by taking this approach. This can also be an appropriate place to provide some indication of your overall results and basic conclusions.

A good strategy is to develop the Introduction in three parts. First, explain the problem. Second, review related work and previous efforts to address the problem. Third, explain your approach and why it is presumably better than previous efforts.

It is often beneficial to review the Introduction carefully after you have written the discussion to make sure that it presents the important ideas that will be needed later in the report. Some writers even prefer to write the Introduction after they have drafted other sections of the report.

THE MATERIAL AND METHODS SECTION

A well-developed procedural section is mandatory for experiment-based research reports. The Methods should be presented in sufficient detail so that others can reproduce the work.

This section covers a lot of ground. It describes the materials used and, if appropriate, their history. It includes a full description of the conditions or parameters studied, along with the properties that were measured and calculated. It specifies analytical techniques and defines non-standard terms and calculated values. For instance, most readers understand “yield” to mean the amount of product obtained from a procedure divided by the amount of starting material and “recovery” to mean the amount of a specific material in a product divided by its mass in the original starting material. If these terms are used differently, they need to be defined. The term “efficiency” is sometimes used as a substitute for “yield” or “recovery,” which without definition is often confusing. Because “efficiency” can mean many things (effective use of capital, labor, time, one or more substrates or a catalyst, energy, and so on), it should probably always be defined.

This section of the report should also discuss replication of both the experiments and the measurement techniques. JAOCS’ Instructions for Authors discuss the need to make a clear distinction between experiment and analytical or measurement replication. Experiment replication is usually of most interest. Repeated measurement of the same experiential sample is secondary; although, depending on the accuracy or precision

of the measurement method used, it may be appropriate to replicate measurements.

Of course, details that have little bearing on the results are a waste of publishing space and are best left unmentioned. For example, the reader will not need to know the company that manufactured the pipet used to transfer a sample, but maybe they need to know if it was a class A, B, or C pipet as this has a bearing on the accuracy of the determination.

Authors often write the Methods section first. Sometimes they delegate this section to a technician, which is not always good practice. Incomplete discussion about samples, unclear procedures, and missing details regarding replication and statistical analysis are all frequent concerns of editors.

There is a tendency today for authors to reference prior papers instead of describing the methods themselves. I generally advise against this. If the cited method is fully described and you have followed it to the letter, it seems this should be sufficient. However, readers who are unfamiliar with the details of the cited work will be left in the dark, and reviewers will be unhappy about the additional

work they have to do to understand your report. At the very least, you should present the basis of the analytical techniques you have used, if not more. Of course, if the source of the method is obscure, or the method is not fully described, or you have made modifications, then full details must be provided. If there is any doubt that the reader will be able to understand your work, write out your methods.

REPORTING RESULTS

The Results section summarizes the data. It should also introduce the theme or purpose of each experiment without repeating the details covered in the method section.

This section is not a data dump. Authors are expected to whittle the data down to meaningful results. Hence, only relevant data needed to support the theme of the report should be included. This does not mean you get to remove inconvenient data points that are in conflict or are difficult to explain. Additionally, too many authors report properties simply because they can be measured and

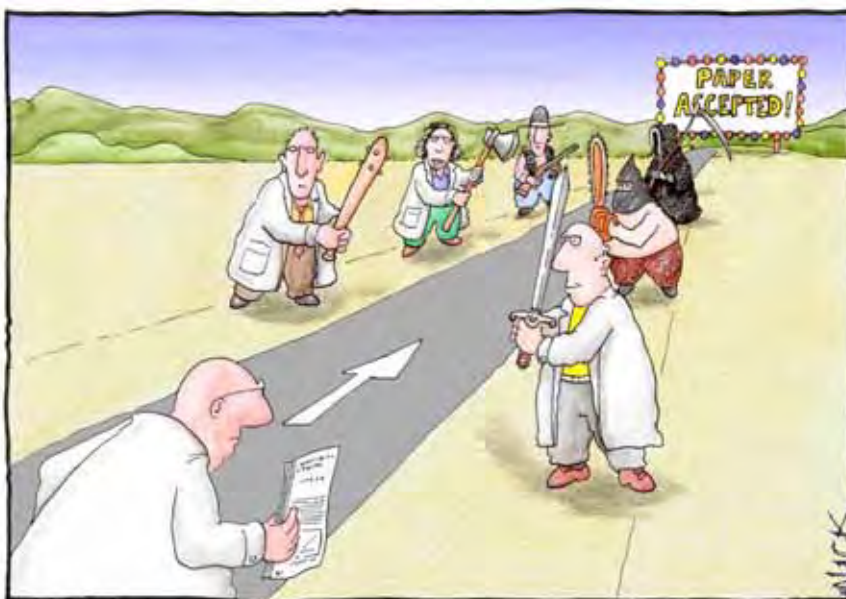
not because they are important to the discussion, which can be annoying to readers. Similarly, if 10 of 11 compounds did not achieve the desired result, your readers do not need to see a table containing 10 negative and one positive result. Such results are better covered within the text.

It is often at this point that you will need to make decisions regarding presentation of tables or graphs. In general, editors frown on presenting the same data in multiple forms, so you should choose

carefully. If your purpose is simply to relate an important trend, then a line or bar graph is a good option. If, however, you need to discuss elements of the data in detail, then a table may be more effective. You might also consider whether other scientists are likely to use the specific values, such as basic compositional or physical property data, in which case you might lean toward presenting the data as a table.

Tables and figures should be self-explanatory. In other words, they should be understandable if separated from the text. In general, tables and figures do not include method details, but should clearly describe what the data represents. Hence, titles should be informative and footnotes used to provide important explanations.

Technical writers often argue that tables should be oriented vertically, so that data is read down and not across the page [1]. Readers generally find it easier to scan data vertically, and publishers prefer this format, as it allows for smaller column widths. Short one- or two-line tables that span across the page should be avoided. This material can often be covered in the text.



Most scientists regarded the new streamlined peer-review process as "quite an improvement."

Credit: Nick D. Kim, Strange-Matter.net

Figures should be scaled to show trends, without leaving excess white space, and the important trends in figures should not be obscured by insets, pictures, pictorials, or other diversionary information. Figures need to be understandable when reduced to the journal column format, and specifics about figure reproduction can often be found in the journal's Instruction for Authors. If needed as a test, use a photocopier to reduce a figure to the expected publication size. Symbols and lines should be appropriately sized—large enough to be clearly observed but not so heavy to distract from the trends being presented. Axes headings and labels should also be readable when reduced to print size. Use sans serif fonts and other uncomplicated fonts as these tend to reproduce better in figures. If more than one data set is presented on a figure, each set should be distinguishable with distinct symbols or lines, but not both, as multiple differences are more for the reader to digest for no additional value.

I often receive figures that have been derived directly from instrument software. These raw instrument figures are frequently scanned into images files, further degrading their quality. Low line resolution, limited line and symbol contrast, small fonts, and unlabeled axes are common problems. These elements can be improved by altering instrument default settings and changing software file formats to meet journal requirements. For AOCS journals, vector-based graphics drawings should be submitted as eps files and images should be submitted as tiff files. If necessary, transfer the raw data into a graphic program and reproduce the figures accordingly. Finally, note that most journals do not want authors to embed the tables and figures into the text of their report, as the type setter will only have to “extract” them. Most journals prefer such images to be attached as separate files during the submission process.

A MEANINGFUL DISCUSSION

The Discussion is the heart of a research report. This section relates the data to the underlying problem. It should compare the results with prior studies and related literature, and discuss reasons for differences. It should also comment upon limitations of the approach, methods used, and unanticipated problems with data collection. It may include comments regarding aspects of the subject that need further study, implications for other areas of research, and the potential for commercialization. Some speculation is acceptable in the discussion, but over-speculation is not beneficial. Objectivity is what is called for here.

One issue I see frequently in submissions is the merging of the Results and Discussion sections used to disguise the absence of serious discussion. While some descriptive reports, such as seed oil compositional reports, may only require modest discussion, most reports are simply incomplete without a detailed discussion. Another frequent problem is one-sided discussion that details all of the particular benefits of a process but neglects the pitfalls. I'm sure there is a place for this kind of advertising, but it is not in your research report. A one-sided discussion will not improve your research paper and is unlikely to fool the reviewers. Also, do not oversell the results. If the results were not strong enough to be commercially meaningful, don't claim them to be.

THE CONCLUSIONS: FINISHING THE STORY

AOCS journals currently have different requirements regarding the Conclusions section. JAOCS requires a Conclusions section; *Journal of Surfactants and Detergents* (JSD) prohibits it; and *Lipids* avoids the issue by not mentioning it. When an explicit Conclusions section is not required, journals often request that authors end the discussion with a summary paragraph. JSD asks that the main conclusions be included as part of the Abstract.

After the abstract, the conclusions are often the second section casual readers will review to decide if a report is of interest. Some readers will only read the other sections of a report if the conclusions look interesting or novel. Hence, this section should not be an afterthought.

Usually, this section consists of a few sentences or a short paragraph that highlight the most important points learned from the study. These points should be based on the data presented and not delve into side issues or unexplored areas. The Conclusion should not restate the results from each set of experiments; nor should it allude to future work or the necessity of conducting additional experiments. While the need for additional experiments may be a valid discussion point, it is not a conclusion.

The above is a brief summary of the points that should be considered in the main sections of a research report. Further guidance on these sections as well as notes on acknowledgements, references, keywords, and other report details can be found in related literature [1–4].

Related literature

1. Day, R.A., and B. Gastel, *How to write and publish a scientific paper*, 7th ed., Greenwood-ABC-CLIO, Santa Barbara, CA, 2011.
2. O'Connor, M., and F.P. Woodford, *Writing scientific papers in English: an ELSE-Ciba foundation guide for authors*, Elsevier-Excerpta Medica-North Holland, Amsterdam, 1976.
3. Ebel, H.F., C. Bliefert, and W.E. Russey, *The art of scientific writing: from student reports to professional publications in chemistry and related fields*, VCH Verlagsgesellschaft, Weinheim, Germany, 1987.
4. *The ACS style guide: effective communication of scientific information*. 3rd ed., Ed. by Coghill, A.M. and L.R. Garson, American Chemical Society and Oxford University Press, New York, 2006.

Since the patent on the first-generation Roundup Ready soybean trait expired in the United States in March 2015, farmers have begun planting generic genetically modified soybeans, according to a July 30, 2015, article by Antonio Regalado in *MIT Technology Review* (<http://tinyurl.com/genericG-MOs>). Roundup Ready soybeans are resistant to the weed killer glyphosate, and more than 90% of soybeans grown in the United States are genetically modified with Monsanto's (St. Louis, Missouri, USA) glyphosate-resistance genes. Farmers can now buy off-patent seeds for half as much as they would pay for second-generation Roundup-resistant seeds from Monsanto, and farmers are free to save and replant the generic seeds for the next growing season. Several agricultural universities across the United States are launching their own generics. In 2009 Monsanto introduced a second-generation, higher-yield soybean called Genuity® Roundup Ready 2 Yield®. The Roundup Ready 2 Yield patent will extend until the end of the next decade.

■ ■ ■

Evogene (Rehovot, Israel) has successfully completed its first round of computational discovery of microbial genes with insecticidal properties, according to a news release on the company's website (<http://tinyurl.com/insecticidalgenes>). To discover novel candidate genes that can kill pests such as corn rootworm and corn earworm, Evogene researchers used a proprietary DNA database containing tens of millions of microbial genes and a dedicated analysis platform called BiomeMiner™. BiomeMiner uses advanced algorithms to rapidly identify and prioritize genes with potential insecticidal properties. The candidate genes will be inserted into crops and validated against target insects at Evogene's dedicated R&D site in St. Louis, Missouri, USA.

■ ■ ■

In a recent review in *Agriculture & Food Security*, Nina V. Federoff at Penn State University (University Park, Pennsylvania, USA) describes the many challenges of "Food in a future of

BIOTECHNOLOGY



Crop dusters may someday spray RNA instead of conventional herbicides or pesticides.

Oilseed crops that produce high levels of acetyltriacylglycerols

Researchers have engineered oilseed crops that produce the highest levels of modified seed lipids to date, according to a recent report in *Plant Biotechnology Journal* (<http://dx.doi.org/10.1111/pbi.12325>, 2015). The modified lipids, known as 3-acyl-1,2-diacyl-sn-glycerols (acetyl-TAGs), may find important industrial, food, and biofuel applications.

Acetyl-TAGs are unusual triacylglycerols that have an sn-3 acetyl group in place of a fatty acyl group. The acetyl group confers commercially useful properties, including reduced viscosity, lower melting point, and lower calorie content than conventional triacylglycerols. Synthetic acetyl-TAGs are currently used in a variety of food and industrial

applications, such as food coatings, emulsifiers, and plasticizers.

Researchers led by Timothy P. Durrett at Kansas State University (Manhattan, Kansas, USA) had previously identified an enzyme from a plant (burning bush; *Euonymus alatus*) that accumulates acetyl-TAGs as the major component of its seed oil. The enzyme, an acetyltransferase called EaDacT, synthesizes acetyl-TAGs from diacylglycerol and acetyl-CoA. When the researchers expressed this enzyme in the seeds of another plant, *Arabidopsis thaliana*, up to 45% of the seed oil was acetyl-TAGs (*Proceedings of the National Academy of Sciences USA*, <http://dx.doi.org/10.1073/pnas.1001707107>, 2010).

In the new work, Durrett and his colleagues identified another metabolic pathway that affects acetyl-TAG accumulation. They reasoned that the EaDacT enzyme likely competes with an endogenous plant enzyme, called diacylglycerol acetyltransferase (DGAT1), that makes

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10 billion" (<http://dx.doi.org/10.1186/s40066-015-0031-7>, 2015). Federoff provides an interesting historical perspective on the development of modern agriculture, from the earliest crop domestication to current genetic modification (GM) techniques. She notes that the Food and Agriculture Organization of the United Nations estimates that food production will need to increase by 70% by 2050, when the world's population is expected to reach 9.6 billion. Due to limitations in agricultural land, water, and energy, this increase will only be possible if GM crops are widely adopted, Federoff says. ■

conventional TAGs from diacylglycerol. When the team expressed *EaDacT* in *Arabidopsis* plants that lacked the *DGAT1* gene, acetyl-TAG levels increased to up to 65% of total TAGs in seeds.

Next, the researchers tested whether these genetic manipulations would work to increase acetyl-TAG accumulation in an oilseed crop, *Camelina sativa*. Because camelina is not widely used as a food crop, the plant is particularly suitable for the production of industrial and biofuel feedstocks. Camelina can grow on poorer-quality farmland and needs little irrigation or fertilizer.

In camelina, expression of *EaDacT* alone resulted in acetyl-TAG levels of up to 64% in seeds. Because camelina has three *DGAT1* homologs, the researchers used RNA interference (RNAi) to simultaneously suppress the expression of all three *DGAT1* enzymes. The expression of *EaDacT* together with RNAi of *DGAT1* produced very high levels of acetyl-TAGs: up to 85% of camelina seed oil. When Durrett and his colleagues grew three independent transgenic lines in the field, acetyl-TAGs accumulated to similar levels as in the lab (74.4–86.2%). Sizes and germination rates of the transgenic camelina seeds were similar to those of wild-type camelina, and the seedlings were able to metabolize acetyl-TAGs as rapidly as regular TAGs.

Acetyl-TAGs purified from transgenic camelina seed oil had a 34% reduced viscosity compared to conventional TAGs from wild-type oil. This viscosity falls within the range specified for diesel No. 4 fuel, which is used in some locomotive engines and stationary power plants. Most plant oils are too viscous for direct use as biodiesel and must first be transesterified. The low viscosity of the camelina-derived acetyl-TAGs may allow the oil to perform as a “drop-in” diesel fuel without transesterification, the researchers say. The oil also had a reduced freezing point, which may confer improved cold-temperature properties compared with traditional oils.

Finally, the team expressed *EaDacT* in soybean plants, which are not closely related to the family that includes *Arabidopsis* and camelina. Nonetheless, *EaDacT* expression caused acetyl-TAGs to accumulate to up to 70% in soybean oil. The researchers did not test the effect of simultaneously suppressing soybean *DGAT1* homologs. For food oils such as soybean, the replacement of a long-chain fatty



The oilseed crop *Camelina sativa* may be a renewable source of commercially important acetyltriacylglycerols.

acid with acetic acid would reduce the total calorie content by 6.3% compared with conventional TAGs.

Scotland to ban GM crops

AGenetically modified (GM) crops will not be allowed in Scotland, the Scottish government announced August 9, 2015, in a statement (<http://tinyurl.com/ScotlandGM>). The government will soon submit a request to the European Union (EU) that Scotland be excluded from any EU consents for the cultivation of GM crops, including those already authorized.

Under EU rules, GM crops must be formally approved before they can be cultivated in member states. The EU has already approved GM Monsanto maize MON810, and the authorizations of six other GM crops are pending. Recently, the EU passed a rule that allows member states to restrict or ban genetically modified organisms (GMOs) within their own borders.

“Scotland is known around the world for our beautiful natural environment—and banning growing genetically modified crops will protect and further enhance our clean, green status,” said Rural Affairs Secretary Richard Lochhead. “There is no evidence of significant demand for GM products by Scottish consumers, and I am concerned that allowing GM crops to be grown in Scotland would damage our clean and green brand, thereby gambling with the future of our £14 billion food and drink sector.”

Lochhead also cites the “precautionary principle”—in other words, it is better to be safe than sorry—as a reason for Scotland’s rejection of GM crops, despite a scientific consensus that GM foods are safe and beneficial. However, the ban is not expected to prohibit research on GM crops, which would be allowed to take place in Scotland under controlled laboratory conditions.

Organic and anti-GMO groups have praised the decision, whereas some Scottish scientists, including Douglas Tocher at the University of Stirling, have expressed dismay. “The blanket ban on the growing of GM crops in Scotland ignores the potential benefits the technology can provide, and the Scottish government has openly stated that the decision was not based on scientific evidence,” Tocher told *Inform*.

Tocher’s research at the University of Stirling involves developing GM oilseed crops to provide sustainable sources of long-chain omega-3 fatty acids to feed farmed salmon—Scotland’s largest food export. “The fact this technology can’t be applied in the very country where it can have direct benefits is, to say the least, ironic,” he said.



The Scottish government plans to ban GM crops to protect Scotland’s “clean, green status.”

RNA sprays: an alternative to GMOs?

The controversy surrounding genetically modified (GM) crops is unlikely to subside any time soon. Meanwhile, companies such as Monsanto (St. Louis, Missouri, USA) are working to develop RNA sprays that would protect crops from insects and disease without modifying their genomes, reports Antonio Regalado, senior editor of biomedicine at *MIT Technology Review*, in an August 11, 2015, feature article entitled, “The Next Great GMO Debate” (<http://tinyurl.com/RNAsprays>).

Monsanto is developing an RNA spray technology called BioDirect that uses RNA interference (RNAi) to destroy targeted RNA messages. One version of the spray kills Colorado potato beetles. This spray contains a short double-stranded RNA that marks for destruction an essential messenger RNA in the pest. Unlike conventional insecticides, the technology is specific enough to kill potato beetles without harming ladybugs or bees. The potato beetle spray could reach the market by 2020, Monsanto scientists estimate.

In another iteration of the BioDirect technology, Monsanto scientists seek to overcome glyphosate (Roundup) resistance in weeds. DNA sequencing of resistant weeds has determined that some have as many as 160 extra copies of a gene called EPSPS—which encodes the enzyme that glyphosphate blocks. In lab and field tests, soaking resistant weeds with a mixture of Roundup and double-stranded RNA targeting the EPSPS gene successfully killed them. Along with the Roundup and RNA, researchers applied a silicone surfactant that helped the RNA molecule slip inside the weed’s air-exchange holes. RNA sprays could be made much more quickly and cheaply than new GM crops to deal with an insect infestation or a virus, Regalado notes. Monsanto scientists are also working on sprays that can penetrate



plant cells to block certain genes, for example, to help plants survive a drought. Gene silencing effects last only a few days or weeks, so that when the drought ends, the plant would revert back to its usual characteristics. Because the cost of RNA synthesis has plummeted in recent years, as little as \$5 worth of RNA is enough to kill 100% of potato beetles on an acre of plants, Monsanto scientists estimate. Critics argue that the public is no more likely to accept RNA sprays than GMOs, especially since the RNA will be sprayed out in the open. A panel of experts commissioned by the US Environmental Protection Agency has stated that the “potential scale” of RNA used in agriculture “warrants exploration of the potential for unintended ecological effects.” ■

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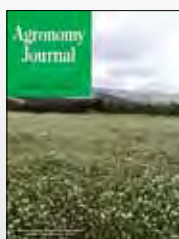
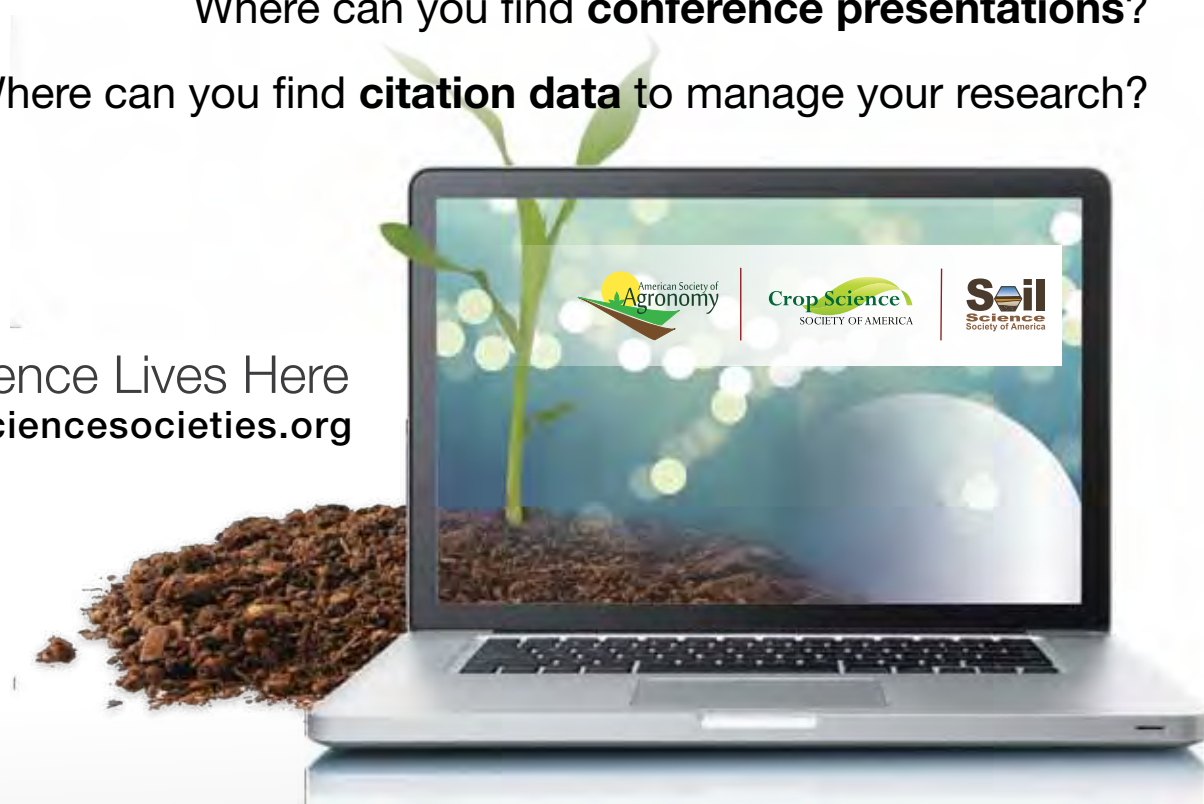
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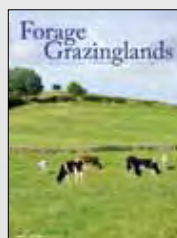
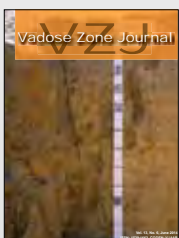
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Procter & Gamble (P&G; Cincinnati, Ohio, USA) has accepted an offer of \$12.5 billion from beauty products manufacturer Coty, Inc. (New York City, USA), to merge 43 color cosmetics, fragrance, and hair care brands. To save P&G from paying capital-gains taxes, the deal will be structured as a Reverse Morris Trust, in which the beauty brands will be separated from P&G and merged with a Coty subsidiary. Included in the transaction are hair care brands Clairol, Wella, and Nice & Easy; fragrance lines Hugo Boss, Dolce & Gabbana, and Gucci; and color cosmetics lines Max Factor and Cover-Girl. The transaction will mark Coty's first entry into hair care lines and will expand the company's geographical range into Brazil, Japan, and other large beauty markets.



REACH (Registration, Evaluation, Authorisation, and Restriction of CHemicals) may be having the unintended consequence of stifling innovation, according to a survey of 1,600 companies conducted by the European Commission. REACH, which took effect in the European Union (EU) in June 2007, regulates the production and use of chemicals with the goal of protecting human health and the environment. The preliminary results of the survey, announced at the Helsinki Chemicals Forum May 28–29, 2015, indicate that 35% of the companies surveyed say that REACH is negatively affecting their capacity to innovate, with only 10% claiming a positive impact. Almost 40% of chemical manufacturers said that REACH has affected their competitiveness compared to companies outside the EU, with 75% claiming the regulations have weakened their competitiveness. In contrast, 57% of chemical suppliers and almost 45% of end users said that REACH has strengthened their competitive positions.



Declining oil prices will slow growth in the oil field chemicals market for 2015–2019, according to a new global market study by IHS, Inc. (*The IHS Chemical 2015 Specialty Chemicals Update Report—Oil Field Chemicals*). ■

HOME & PERSONAL CARE



Surfactants called drilodefensins allow earthworms to digest dead leaves in soil.

Unique biosurfactants discovered in earthworms

Researchers have discovered a new class of biosurfactants, called drilodefensins, from the lowly earthworm (<http://dx.doi.org/10.1038/ncomms8869>, 2015). The surfactants help protect worms from a diet rich in polyphenols.

Plants produce polyphenols for defense against herbivores. Polyphenols bind to and precipitate soluble proteins, which inhibits enzyme activity. Mammals have evolved peptides in their saliva that sequester polyphenols into insoluble complexes, rendering them harmless. But until now, scientists did not know how earthworms cope with the high levels of polyphenols in the leaf litter that comprises most of their diet.

In the new study published in *Nature Communications*, researchers led by Jacob G. Bundy at Imperial College London (UK) used metabolic profiling, also known as metabolomics, to search for possible surface-active compounds in earthworms that could help protect them against polyphenols. With mass spectrometry, the researchers identified a highly abundant compound called 2-hexyl-5-ethyl-furan-3-sulfonate (known hereafter as Compound 1) from the worm gut, as well as some structurally related compounds. The identified compounds were all dialkylfuransulfonates—strong acids

with a lipophilic alkyl chain, and thus likely to be surface active.

Bundy and his colleagues discovered that the dialkylfuransulfonates, which they termed drilodefensins, were present in 14 different earthworm species but not in other worms such as leeches or aquatic worms. Compound 1 is so abundant that it comprises about 1.3% of the earthworm's body mass. The researchers used imaging mass spectrometry (IMS) to localize Compound 1 to the earthworm foregut.

Then, the team tested the surfactant properties of a pure synthetic preparation of Compound 1. They found that in aqueous buffer, Compound 1 decreased the surface tension and started to form micelles at a concentration of 6 mM. In comparison, the critical micelle concentration of the commonly used surfactant sodium dodecyl sulfate (SDS) is 8 mM. Compound 1 reduced the precipitation of proteins by polyphenols in vitro, without affecting the activity of an earthworm gut enzyme. SDS likewise prevented protein precipitation, but it reduced enzyme activity. The researchers suspect that earthworm enzymes and drilodefensins evolved together to be compatible with each other.

Interestingly, exposing earthworms to high-polyphenol diets increased the level of Compound 1 by about 50%. Also, when the researchers collected wild earthworms from different sites, they found that worms living in woodlands made higher levels of Compound 1 than those in grasslands.

CONTINUED ON NEXT PAGE

Drilodefensins could someday find environmental and commercial uses, for example, in cleaning up oil spills or as new surfactants in consumer products. Also, Bundy's team found evidence that enzymes toward the back of the earthworm's gut rapidly degrade the polyphenol-bound drilodefensins to recycle them. Hypothetically, these enzymes could be used to remove synthetic detergents from wastewater before they enter the environment. "It's too early to say yet what the specific applications of earthworm compounds could be, but I definitely think they could have a valuable commercial use," Bundy told the *Christian Science Monitor* (<http://tinyurl.com/csmworms>).

First commercial surfactant from microalgae oil

Solazyme, Inc. (South San Francisco, California, USA) and BASF (Ludwigshafen, Germany) have partnered in launching a high-performance algal betaine—the first commercial surfactant derived from microalgae oil. The surfactant, which is being sold by BASF under the trade name Dehyton AO 45, will likely find applications in home and personal care products.

Solazyme, a renewable microalgae oil and ingredients company, produces its AlgaPür microalgae oils using a highly controlled fermentation process in which microalgae convert low-cost sugarcane into high-value oils. The proprietary technology produces oils with high purity and efficiency, with minimal carbon, water, and land use. "We are excited to partner with BASF to launch the world's first commercial microalgae-derived betaine surfactant," said Robert Webber, chief commercialization officer at Solazyme in a statement (<http://tinyurl.com/algabetaine>). "BASF's broad capabilities in chemical conversion of triglyceride oils and deep expertise in surfactants enable significant growth opportunities in the future."

The new algal betaine is marketed as a high-performance alternative to amidopropyl betaines, amphoteric surfactants that are being used increasingly in cosmetics, toiletries, and home care products. Advantages of betaines include good detergency, foaming, and viscosity building; hard water compatibility; mildness to skin and hair; conditioning effects; broad pH range; and biodegradability. Betaines are often used as secondary surfactants to reduce the skin irritation of anionic primary surfactants. Amidopropyl betaines can be derived from vegetable oils such as coconut and palm kernel. As an alternative to amidopropyl betaines, Dehyton AO 45 is ideal for products that require a rich and gentle foam, such as hand dishwashing liquids, shampoos, and liquid soaps.

Elevance launches d-limonene alternative

Elevance Renewable Science, Inc., (Woodridge, Illinois, USA) has launched a bio-based cleaning ingredient that addresses the supply limitations and price uncertainty of d-limonene, according to an August 4 news release (<http://tinyurl.com/clean1000>). The company claims that Elevance Clean™ 1000 boosts the performance of formulations containing d-limonene, especially those used in degreasing and asphalt or tar removal applications.

Extracted from citrus rind, d-limonene is a green alternative to more toxic solvents such as acetone, toluene, and xylene. Alone or in combination with a surfactant, d-limonene helps remove greasy soils from surfaces. As the cleaning products industry moves increasingly toward renewable ingredients, the demand for d-limonene has outpaced its supply. Over the past two years, disease has decimated orange groves in the United States and Brazil, the two major orange-producing countries. This decline in orange supply has posed a challenge for manufacturers who use d-limonene in green solvents and cleaning products (see feature article on page 558).

Elevance Clean 1000 is produced by a proprietary olefin metathesis technology using natural plant oils such as palm, canola, and soybean as feedstock. These feedstocks are more diverse and stable than citrus, reducing the risk of supply problems and price volatility. Elevance's biorefinery in Gresik, Indonesia, supplies Elevance Clean 1000 at an industrial scale, and its availability will soon be bolstered by a second facility in Natchez, Mississippi, USA.

According to the company, Elevance Clean 1000 blends easily with d-limonene formulations because of its similar hydrophilic-lipophilic balance requirement. The new ingredient not only boosts the performance of formulations containing d-limonene but also allows manufacturers to cut costs by using less of the citrus-derived solvent. The new ingredient surpasses the solvency power of d-limonene and demonstrates three times the performance of d-limonene in the Gardner Cleaning Test (ASTM D4488-95). Elevance Clean 1000 outperforms d-limonene at neutral pH, eliminating the need for highly caustic cleaning products. Also, the flash point of the new solvent (102°C) is significantly higher than that of d-limonene (50°C), increasing product safety.

When blended with d-limonene, Elevance Clean 1000 will be particularly well suited for degreasing, especially in mechanical and immersion cleaning application, the company says. The new ingredient is especially targeted for removing industrial greases, asphalt, tar, and bitumen. Elevance Clean 1000 is biodegradable, listed on the Toxic Substances Control Act (TSCA) inventory, and a nonirritant for skin and a mild irritant for eye.



A new d-limonene alternative could boost performance and cut costs for degreasing and asphalt or tar removal applications

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ECHA to increase ability to revoke REACH registrations

Emma Chynoweth

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

ECHA says changes to the REACH dossier technical completeness check (TCC)—its main tool for implementing REACH’s “no data, no market” principle—will make it easier for it to revoke registration numbers.

The agency plans to publish an information package on the changes and their impact, by the end of the year.

In some cases, the agency will conduct a manual examination of dossiers as part of the changes to the TCC process.

The “no data, no market” provisions of REACH are pursued in the registration process and the compliance check. ECHA’s Doris Thiemann says the agency is planning to enforce TCC by checking if all necessary information is included in the registration dossier. She says: “We expect this to be a major way forward to improve the quality of registration dossiers and to enforce ‘no data, no market’.”

Until now, the TCC process has been automated, allowing dossiers containing data of questionable quality to receive registration numbers. NGOs say such dossiers flout REACH’s “no data, no market” principle.

They also say it is difficult to trace how member states respond to evaluation decisions, which come at the end of the dossier compliance check process. These are issued by ECHA for dossiers deemed to be non-compliant. So far, registrants have updated dossiers in response to over 600 such decisions.

When following up a registrant’s actions in response to them, ECHA may decide that the dossier is still non-compliant and issue a statement of non-compliance (SONC), inviting national enforcement authorities (NEAs) to take action.

ECHA says the number of cases provided to NEAs is “steadily growing” and the process works well because the latter’s actions “result in information requested in the decisions being submitted to ECHA.”

According to ECHA’s 2014 evaluation progress report, 72% of registrants complied with evaluation decisions before a SONC was issued; 11% complied after receiving a SONC; and in 1% of cases a new compliance check has been started. The remaining 16% are still in progress.

So far, ECHA has released 46 SONCs to *Chemical Watch* following access to document requests. The latest batch includes 23 registrations.

There is no deadline in REACH by which ECHA should check registrants have responded to an evaluation decision, but it aims to perform the follow-up within six months of the deadline set within it, for at least 75% of the cases.

The agency says the process can be “laborious,” especially in cases where “adaptation of the standard information requirements has been done.”

It typically takes about ten months, from when a SONC is issued, for the registrant to comply. So far, the agency has not come across a case where revocation of a registration number was deemed necessary.

According to the information released to *Chemical Watch*, some evaluation decisions refer to multiple data gaps in the dossier. However, ECHA would not confirm whether these might be so-called “google dossiers” and said the evaluation process does not categorize cases, based on the level of information included.

The update to the TCC process aims to identify dossiers containing information that is inconsistent with the IUCLID data field.

At the most recent ECHA Management Board meeting, the European Commission confirmed that the changes to the TCC process are within the scope of the completeness check, and can apply to updates “as long it is reasonable and necessary.”

It also said ECHA may invalidate registration numbers in well-justified cases.

Many board members welcomed this step as major way forward to improve the quality of dossiers.

At present, ECHA says it only withdraws registration numbers in exceptional cases or after the TCC process. So far, revocation has been considered in cases where registration numbers were assigned to registrants found to be non-existent, and where they failed to pay the correct registration fee.

Erwin Annys, CEFIC REACH director, said it is difficult to predict the impact of the revised TCC process.

He says: “A manual check of all dossiers does not seem to be realistic. Everything will depend on which dossiers will be manually verified. And this creates quite some commotion within industry, not knowing what will happen if a registration number is not obtained in time.”

Tips from **inform|connect**

Tips from inform|connect is a regular Inform column that features tips and other discussion highlights from the community forum board at <http://www.informconnect.org/home>.

Q: A chemical engineer at a vegetable oil processing facility in Colombia had a customer who asked for deodorized palm oil with a saponification value between 8 and 8.1. The engineer tried using neutral, acidic, and various combinations of adsorbents, but meeting these color specifications decreased the overall processing capacity of the plant, which ordinarily produces up to 500 tons of vegetable oil per day. He asked if there was there a process or combination of processes that would allow the plant to fulfill the customer's request while maintaining the overall capacity of the plant.

A: A community member noted that the main color component in palm oil is red β -carotene, which is heat sensitive. Therefore, decreasing the throughput increased the residence time at high temperature and ensured that the thermal breakdown was sufficient to meet the color specifications. For that reason, the community member suggested that the engineer try operating at a somewhat increased temperature to see if that reduces the color.

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Fats and oils labeling in Argentina and Mercosur

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

Leslie Kleiner

To understand fats and oils labeling in Argentina and Mercosur, and also learn about that region's position on trans fats, I interviewed Cintia Skverer, food scientist and specialist in foreign trade at the Argentinean "Administración Nacional de Medicamentos, Alimentos y Tecnología Médica (ANMAT)", (National Administration of Drugs, Food, and Medical Technology). Skverer provided detailed information on the regulations that affect labeling in Mercosur ("Mercado Común del Sur"/Common Market Group), which is comprised of the member countries of Argentina, Brazil, Paraguay, Uruguay, and Venezuela, as well as the associate countries of Chile, Bolivia, Colombia, Ecuador, and Peru.

Q: How are fats and oils labeled in Argentina?

A: The regulations for labeling food in Argentina, including those for labeling fats and oils, are specified in Chapter V of the "Código Alimentario Argentino (CAA)" (Argentinean Alimentary Code). The joint resolutions 149/2005 and 683/2005 incorporate the norms "RESOLUCIÓN GRUPO MERCADO COMÚN (GMC)" (Resolutions Common Market Group), which establish the regulations for packaged foods. In this sense, GMC regulation N° 26/03 focuses on general labels, while GMC N° 46/03 and GMC N° 06/94 focus on the nutritional label and ingredient declaration, respectively.

According to GMC N° 46/03, it is mandatory to label the quantitative amount of total, saturated, and trans fats, when labeling packaged foods. Furthermore, according to GMC N° 06/94, refined oil ingredients different than olive oil must be demarcated either by their corresponding "vegetable" or "animal" qualifier (such as vegetable oil) or by their specific animal or vegetable origin sources (such as soybean oil).

Hydrogenated or partially hydrogenated oils must be qualified as such by adding the corresponding qualifiers

("hydrogenated" or "partially hydrogenated") to the specific animal or vegetable oil or the specific animal or vegetable oil source. For refined fats excepting butter, the ingredient must be declared as "fat" along with the term "vegetable" or "animal" depending on the origin.

Q: Are there any differences between food labels in Argentina and the rest of Mercosur, or are they standardized?

A: The member countries of Mercosur have worked on harmonizing the national norms, creating the aforementioned GMC resolutions. The CAA incorporated these norms to establish rulings on labeling of packaged foods (chapter V), which are current and of mandatory compliance.

Q: Are there particular specifications for the labeling of margarine and other fats?

A: Chapter VII of CAA establishes specific regulations for various types of fats and oils (Argentinean national resolutions). For example, article 551 establishes that the commercial name of “margarine” must be written in red font and the label must specify “to spread” or “for baking, accordingly. If the product is fortified with vitamins A and/or D, their presence and concentration must also be part of the label. In another example, Article 540 establishes the labeling of fats of animal source. In this case, fats meeting the corresponding requirements, must be labeled as “porcine butter,” “porcine fat,” “bovine fat,” or similar. However, if these fats were subjected to refining, the qualifier “refined” must also be added.

Q: How are structured lipids (inter- and/or intraesterified labeled)?

A: Structured lipids packaged for sale or used as ingredients in foods must comply with the labeling requirements established for all other foods per Chapter VII of the CAA. Article 548BIS—(Res 2012, 19.10.84) establishes that in the label, the words “interesterified” or “transesterified” need to be specified accordingly.

Regarding trans fats, what are the current limits allowed in foods? Also, given the recent ruling in the United States regarding the phasing out of trans fats from the market, do you anticipate a similar movement in Argentina and Mercosur?

Article 155 TRIS establishes trans fats limits in national and imported foods. This is a national regulation which was implemented in the year 2012 for vegetable oils and margarines for direct consumption, and in the year 2014 for the remaining foods. This article also establishes that the trans-fat content should not exceed 2% of total fats in vegetable oils and margarines destined to direct consumption, and not to exceed 5% of the total fat content of remaining foods. These limits are not applicable to fats arising from ruminants, including milk fat. Article 155 TRIS was incorporated to CAA during the year 2010 and it allowed a transition period to industry of up to 2 years for vegetable oils and margarines for direct consumption, and 4 years for remaining foods.

Thank you to Maia Varela for the information provided, and to ANMAT for the present collaboration.



Latin America Update is produced by Leslie Kleiner, R&D Project Coordinator in Confectionery Applications at Roquette America, Geneva, Illinois, USA, and a contributing editor of *Inform*. She can be reached at LESLIE.KLEINER@roquette.com.



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Fatty acid compositions of plant tissues

Lipid Snippets is a regular Inform column that features select content from The AOCS Lipid Library (<http://lipidlibrary.aocs.org/>).

The fatty acid compositions of the seed oils of importance to commerce have been reviewed [1], and data for a few typical ones are listed in Table 1. Maize (corn), sunflower, and safflower oils are of nutritional value since they contain appreciable amounts of the essential fatty acid—linoleic acid.

Table 1. The fatty acid compositions (weight % of the total) of some seed oils [1].

Fatty acid	Seed oil					
	Soybean	Maize	Safflower	Rapeseed ^a	Olive	Palm
16:0	11	11	6	3	12	42
18:0	4	trace	3	1	2	4
18:1	23	25	12	11	72	38
18:2	51	57	73	13	8	9
18:3	7	1	1	9	1	
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^a newer cultivars can contain much less erucic acid

Table 1. The fatty acid compositions (weight % of the total) of some seed oils [1].

Excessive amounts of linolenic acid, as in soybean oil, can lower the commercial value of an oil because it is then more susceptible to rancidity problems caused by autoxidation; it is therefore a common industrial practice to subject the oil to hydrogenation. In contrast, there are no such problems with olive oil, an important lipid constituent of the ‘Mediterranean’ diet, with its high content of oleic acid. Palm oil contains a higher proportion of saturated fatty acids than most seed oils. Similarly, cocoa butter consists largely

of molecular species with saturated fatty acids in positions *sn*-1 and 3 and oleic in position *sn*-2. Rapeseed is one of the few oil crops capable of being grown in northerly climates. In its native form, it tends to have a high content of erucic acid (22:1(*n*-9)), which may have some properties that may be harmful to the consumer, although this is still a matter for controversy. However, new cultivars with negligible levels of this component (“canola”) are now widely grown. Cottonseed oil resembles maize oil in its composition, but also contains small amounts of the cyclopropene fatty acid, ‘sterculic’ or 9,10-methyleneoctadecenoic acid, which has well-established toxic properties and must be removed during refining. Palm kernel and coconut oils are noteworthy for a high content of saturated fatty acids of medium chain-length.

The picture will become more complex as new genetically modified seed oils are introduced. In addition, there are many seed oils which may have limited or negligible commercial value at present, but contain fatty acids with unusual substituent groups and are of great interest to biochemists.

Each of the lipids in a plant tissue can have a characteristic fatty acid composition and for illustrative purposes, some results for spinach leaf lipids are listed in Table 2.

Table 2. The fatty acid compositions (weight % of the total) of the individual lipids of spinach leaves [2].

MGDG ^a	Lipid class				
	DGDG	SQDG	PG	PC	PI
trace	6	27	22	20	41
trace			35	trace	
30	3			trace	
	1		trace		1
1	4	6	2	11	6
1	3	39	5	30	35
67	84	28	36	40	27

^a MGDG, monogalactosyldiacylglycerol; DGDG, digalactosyldiacylglycerol; SQDG, sulfoquinovosyldiacylglycerol; PG, phosphatidylglycerol; PC, phosphatidylcholine; PI, phosphatidylinositol; PE, phosphatidylethanolamine.

Table 2. The fatty acid compositions (weight % of the total) of the individual lipids of spinach leaves [2].

Glycosyldiacylglycerols tend to consist mainly of the unsaturated fatty acids, linoleic acid and especially linolenic acid; a hexadecatrienoic (16:3(n-3)) acid may be present also with certain species. On the other hand, the glycerophospholipids contain higher proportions of saturated fatty acids, generally palmitic acid, in addition

to the unsaturated components. Phosphatidylglycerol is unique in that it contains a substantial amount of an unusual fatty acid, i.e. trans-3-hexadecenoic acid. The fatty acid compositions of plant tissues can vary with climatic and other cultivation conditions, and with the stage of development of the tissue, and major species differences occur. However, the results listed in Table 2 are typical.

This Lipid Snippet document is based on part of Chapter 1 of the Third edition of Lipid Analysis by the author and published by P.J. Barnes & Associates (The Oily Press Ltd). It was omitted from the Fourth edition of the book to save space.

Further reading

1. Sheppard, A.J., J.L. Iverson, J.L. and Weihrauch, Composition of selected dietary fats, oils, margarines, and butter. In: *Handbook of Lipid Research. Vol. 1. Fatty acids and Glycerides*, pp. 341-379 (ed. A. Kuksis, Plenum Press, New York) (1978).
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AOCS MEETINGS WATCH

- **October 27–30, 2015.** SODEOPEC2015, Hyatt Regency Miami, Miami, Florida, USA. <http://sodeopec.aocs.org>
- **November 12–13, 2015.** AOCS Oils and Fats World Market Update 2015, The Convention Centre Dublin, Dublin, Ireland. <http://worldmarket.aocs.org>
- **May 1–4, 2016.** 107th AOCS Annual Meeting & Expo, Calvin L. Rampton Salt Palace Convention Center, Salt Lake City, Utah, USA. <http://annualmeeting.aocs.org>
- **October 5–7, 2016.** World Conference on Fabric and Home Care—Singapore 2016, Shangri-La Hotel, Singapore. <http://singapore.aocs.org>

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PATENTS

Edible water in oil emulsion

De Man, T., Conopco, Inc., US8993035, March 31, 2015

A sealing material is provided, which has excellent sealing propThe invention relates to an edible water in oil emulsion wrapper product wherein the amount of oil exudation is less than 1% at the temperature at which the amount of solid fat content on total product is 3%, wherein the fat is essentially free of hydrogenated fat and wherein said fat comprises less than 35 wt% saturated fatty acid (calculated on total fat) and less than 1 wt% trans unsaturated fatty acid (calculated on total product). The invention also relates to a process for the preparation of an edible water in oil emulsion wrapper product according to the invention.

Making epoxidized esters from epoxidized natural fats and oils

Hagberg, E., *et al.*, Archer Daniels Midland Co., US20150407, April 7, 2015

Processes are described for making epoxidized fatty acid esters from epoxidized natural fats and oils, wherein low moisture fats and oils are identified and selected or made and used in a transesterification process. The products undergo phase separation, and reduced molar excesses of alcohol may be employed compared to processes not employing a low moisture feedstock.

Encapsulated acid, method for the preparation thereof, and chewing gum comprising same

Boghani, N., *et al.*, Intercontinental Great Brands LLC, US9011946, April 21, 2015

Delayed release in chewing gum of a food-grade acid, is provided by encapsulating the food-grade acid in specific amounts of a poly(vinyl acetate) and a fatty acid salt. When incorporated into a chewing gum, the food-grade acid encapsulated in poly(vinyl acetate) and fatty acid salt provides a more delayed release of a sour flavor than food-grade acid encapsulated in poly(vinyl acetate) alone.

Crispy French fries

Bilet, M.J.J., *et al.*, Elwha LLC, US9011950, April 21, 2015

Crispy French fry-style food products are prepared by modification of the water content and the texture of the products' surface layers prior to deep frying. The surface modifications include surface firming, enzyme treatments, starch infusion and/or ultrasound treatment.

Low-fat water-in-oil emulsion containing substantial amounts of HOH triglycerides

Bartoccini, M., *et al.*, Conopco, Inc., US9011960, April 21, 2015.

The invention relates to water-in-oil emulsion comprising 50 to 85 wt% of an aqueous phase and 15 to 50 wt% of a fat phase, wherein said fat phase comprises HOH triglycerides in an amount of 8 to 50% of the total weight of the fat phase, and HHH triglycerides in an amount of 1 to 6% of the total weight of the fat phase, wherein at least 25% of said HHH triglycerides contain at least two different fatty acid residues, where "H" denotes a saturated fatty acid residue with 16 to 24 carbon atoms and "O" an oleic acid residue. The invention further relates to a process for the preparation of an edible water-in-oil emulsion according to the invention. The invention also relates to the use of a HHH triglyceride fat to promote inversion of an oil-in-water-emulsion comprising 15 to 50 wt% of a fat phase, into a water-in-oil emulsion, wherein said fat phase comprises HOH triglycerides.

Method for producing regenerated clay, regenerated clay, and method for producing purified fats and oils

Negishi, S., *et al.*, The Nisshin Oillio Group, Ltd., US9023749, May 5, 2015

The present invention provides a method for performing regeneration of a decolorization capacity of waste clay that has been used for purification of fats and oils, and production of a thermally recyclable compound as a biofuel from oily ingredients in the waste clay at the same time in a convenient manner. That is, a method for producing purified fats and oils of the invention includes: a method for producing regenerated clay including the steps of mixing waste clay that has been used for purification of fats and oils, lower alcohol, and an acidic catalyst; and performing extraction of oily ingredients from the waste clay, and an esterification reaction between the fats and oils and/or a free fatty acid in the oily ingredients and the lower alcohol at the same time so as to regenerate a decolorization capacity of the waste clay; regenerated clay that is produced by the method for producing the regenerated clay; and a process of decolorizing the fats and oils using the regenerated clay.

Patent information is compiled by Scott Bloomer, a registered US patent agent with Archer Daniels Midland Co., Decatur, Illinois, USA. Contact him at scott.bloomer@adm.com.



EXTRACTS & DISTILLATES

Innovative approach to the accumulation of rubrosterone by fermentation of *Asparagus filicinus* with *Fusarium oxysporum*

Li, Y., et al., J. Agric. Food Chem. 63: 6596–6602, 2015, <http://dx.doi.org/10.1021/acs.jafc.5b02570>.

Rubrosterone, possessing various remarkable bioactivities, is an insect-molting C19-steroid. However, only very small amounts are available for biological tests due to its limited content from plant sources. Fungi of genus *Fusarium* have been reported to have the ability to convert C27-steroids into C19-steroids. In this study, *Asparagus filicinus*, containing a high content of 20-hydroxyecdysone, was utilized to accumulate rubrosterone through solid fermentation by *Fusarium oxysporum*. The results showed that *F. oxysporum* had the ability to facilitate the complete biotransformation of 20-hydroxyecdysone to rubrosterone by solid-state fermentation. The present method could be an innovative and efficient approach to accumulate rubrosterone with an outstanding conversion ratio.

Long-chain *n*-3 PUFAs from fish oil enhance resting state brain glucose utilization and reduce anxiety in an adult nonhuman primate, the grey mouse lemur

Pifferi, et al., J. Lipid Res. 56: 1511–1518, 2015, <http://dx.doi.org/10.1194/jlr.M058933>.

Decreased brain content of DHA, the most abundant long-chain *n*-3 polyunsaturated fatty acid (*n*-3 LCPUFA) in the brain, is accompanied by severe neurosensorial impairments linked to impaired neurotransmission and impaired brain glucose utilization. In the present study, we hypothesized that increasing *n*-3 LCPUFA intake at an early age may help to prevent or correct the glucose hypometabolism observed during aging and age-related cognitive decline. The effects of 12 months' supplementation with *n*-3 LCPUFA on brain glucose utilization assessed by positron emission tomography was tested in young adult mouse lemurs (*Microcebus murinus*). Cognitive function was tested in parallel in the same animals. Lemurs supplemented with *n*-3 LCPUFA had higher brain glucose uptake and cerebral metabolic rate of glucose compared with controls in all brain regions. The *n*-3 LCPUFA-supplemented animals also had

higher exploratory activity in an open-field task and lower evidence of anxiety in the Barnes maze. Our results demonstrate for the first time in a nonhuman primate that *n*-3 LCPUFA supplementation increases brain glucose uptake and metabolism and concomitantly reduces anxiety.

Dietary cholesterol and cardiovascular disease: a systematic review and meta-analysis

Berger, S., et al., Am. J. Clin. Nutr. 102: 276–294, 2015, <http://dx.doi.org/10.3945/ajcn.114.100305>.

Dietary cholesterol has been suggested to increase the risk of cardiovascular disease (CVD), which has led to US recommendations to reduce cholesterol intake. The authors examine the effects of dietary cholesterol on CVD risk in healthy adults by using systematic review and meta-analysis. MEDLINE, Cochrane Central, and Commonwealth Agricultural Bureau Abstracts databases were searched through December 2013 for prospective studies that quantified dietary cholesterol. Investigators independently screened citations and verified extracted data on study and participant characteristics, outcomes, and quality. Random-effect models meta-analysis was used when at least 3 studies reported the same CVD outcome. Forty studies (17 cohorts in 19 publications with 361,923 subjects and 19 trials in 21 publications with 632 subjects) published between 1979 and 2013 were eligible for review. Dietary cholesterol was not statistically significantly associated with any coronary artery disease (4 cohorts; no summary RR), ischemic stroke (4 cohorts; summary RR: 1.13; 95% CI: 0.99, 1.28), or hemorrhagic stroke (3 cohorts; summary RR: 1.09; 95% CI: 0.79, 1.50). Dietary cholesterol statistically significantly increased both serum total cholesterol (17 trials; net change: 11.2 mg/dL; 95% CI: 6.4, 15.9) and low-density lipoprotein (LDL) cholesterol (14 trials; net change: 6.7 mg/dL; 95% CI: 1.7, 11.7 mg/dL). Increases in LDL cholesterol were no longer statistically significant when intervention doses exceeded 900 mg/d. Dietary cholesterol also statistically significantly increased serum high-density lipoprotein cholesterol (13 trials; net change: 3.2 mg/dL; 95% CI: 0.9, 9.7 mg/dL) and the LDL to high-density lipoprotein ratio (5 trials; net change: 0.2; 95% CI: 0.0, 0.3). Dietary cholesterol did not statistically significantly change serum triglycerides or very-low-density lipoprotein concentrations. Reviewed studies were heterogeneous and lacked the methodologic rigor to draw any conclusions regarding the effects of dietary cholesterol on CVD risk. Carefully adjusted and well-conducted cohort studies would be useful to identify the relative effects of dietary cholesterol on CVD risk.

Stability of virgin olive oil phenolic compounds during long-term storage (18 months) at temperatures of 5–50 °C

Krichene, D., *et al.*, *J. Agric. Food Chem.* 63: 6779–6786, 2015, <http://dx.doi.org/10.1021/acs.jafc.5b02187>.

Virgin olive oil (VOO) phenolic compounds have high nutritional and biological properties. The purpose of this research was to study the stability of VOO phenolic compounds during long-term storage (18 months) at different temperatures (5, 15, 25, and 50 °C) and to verify the advantage of storing VOO at a temperature lower than the usual commercial conditions (20–25 °C). Four monovarietal VOOs that differed in their fatty acid profile and content of natural antioxidants were used in this study. The degradation of secoiridoid phenolics during storage displayed pseudo-first-order kinetics and depended on the initial content of phenolics related to olive oil variety. The initial degradation rate was similar at 5 and 15 °C but increased considerably at 25 °C and was even faster at 50 °C. Tyrosol derivatives were more stable than hydroxytyrosol compounds, especially in closed bottles with limited oxygen availability. The increase in the content of simple phenolics, the decrease of their secoiridoid derivatives, or the ratio of simple to secoiridoid phenolics could be used as indices of the oxidative and hydrolytic degradation of VOO phenolics. The shelf life of the studied VOO was considerably extended at reduced storage temperature (15 vs 25 °C). Moreover, storage conditions affected VOO phenolic content and therefore the expiration date of the health claim that olive oil polyphenols contribute to the protection of blood lipids from oxidative stress.

The supramolecular chemistry of lipid oxidation and antioxidation in bulk oils

Budilarto, E.S. and Kamal-Eldin, A., *Eur. J. Lipid Sci. Technol.* 117: 1095–1137, 2015, <http://dx.doi.org/10.1002/ejlt.201400200>.

The microenvironment formed by surface active compounds is being recognized as the active site of lipid oxidation. Trace amounts of water occupy the core of micro micelles and several amphiphilic minor components (e.g., phospholipids, monoacylglycerols, free fatty acids, etc.) act as surfactants and affect lipid oxidation in a complex fashion dependent on the structure and stability of the microemulsions in a continuous lipid phase such as bulk oil. The structures of the triacylglycerols and other lipid-soluble molecules affect their organization and play important roles during the course of the oxidation reactions. Antioxidant head groups, variably located near the water-oil colloidal interfaces, trap and scavenge radicals according to their location and concentration. According to this scenario, antioxidants inhibit lipid oxidation not only by scavenging radicals via hydrogen donation but also by physically stabilizing the micelles at the microenvironments of the reaction sites. There is a cut-off effect (optimum value) governing the inhibitory effects of antioxidants depending inter alia on their hydrophilic/



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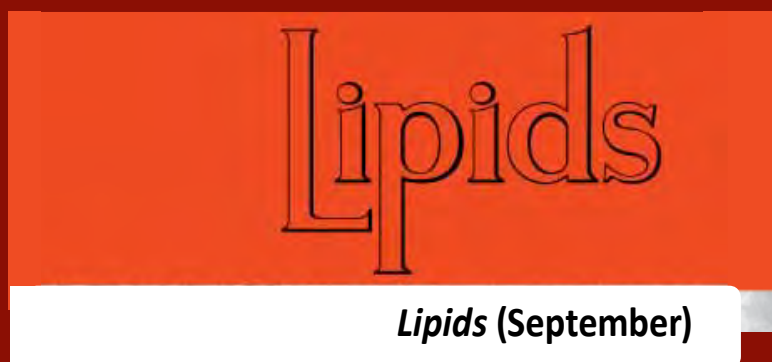
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lipophilic balance and their concentrations. These complex effects, previously considered as paradoxes in antioxidants research, are now better explained by the supramolecular chemistry of lipid oxidation and antioxidants, which is discussed in this review. The evolution of micellar size and number during and after the induction period of lipid oxidation. Amphiphilic compounds, including lipid hydroperoxides, contribute to micelle formation and act as prooxidants. Antioxidants and retarders stabilize micelles and prevent their breakdown.

Lipid peroxidation in a stomach medium is affected by dietary oils (olive/fish) and antioxidants: the Mediterranean versus Western diet

Tirosch, O., et al., *J. Agric. Food Chem.* 63: 7016–7023, 2015, <http://dx.doi.org/10.1021/acs.jafc.5b02149>.

Red meat is an integral part of the Western diet, and high consumption is associated with an increased risk of chronic diseases. Using a system that simulated the human stomach, red meat was interacted with different oils (olive/fish) and lipid peroxidation was determined by measuring accumulation of malondialdehyde (MDA) and lipid peroxides (LOOH). Olive oil decreased meat lipid peroxidation from 121.7 ± 3.1 to 48.2 ± 1.3 μM and from 327.1 ± 9.5 to 77.3 ± 6.0 μM as assessed by MDA and ROOH, respectively. The inhibitory effect of olive oil was attributed to oleic acid rather than its polyphenol content. In contrast, fish oils from tuna or an ω -3 supplement dramatically increased meat lipid peroxidation from 96.2 ± 3.6 to 514.2 ± 6.7 μM MDA. Vitamin E inhibited meat lipid peroxidation in the presence of olive oil but paradoxically increased peroxidation in the presence of fish oil. The inhibitory properties of oleic acid may play a key role in the health benefits of the Mediterranean diet.

How fish oils could support our friendly bacteria

Bentley-Hewitt, K.L., et al., *Lipid Technol.* 27: 179–182, 2015, <http://dx.doi.org/10.1002/lite.201500035>.

Commensal gut bacteria are generally considered to be friendly bacteria, since they can help their host in numerous ways. These can include breaking down undigested food to produce metabolites (by-products), which can be a fuel source for gut cells and can help to regulate the immune system, amongst many other beneficial functions that support the host's health. Probiotic bacteria are bacteria that offer a benefit to their host. They are used in dietary supplements and many are of the genus *Lactobacilli*. We tested whether gut cells respond differently to a commensal bacterium (*Lactobacillus gasseri*) and two pathogenic bacteria (*Escherichia coli* and *Staphylococcus aureus*), and also whether the responses could be altered with PUFAs. We used a cell co-culture model containing a layer of colorectal cells, with immune cells in a porous compartment beneath. This model represents the outer cell lining of our lower gut and the immune cells that sit underneath in an area called the lamina propria. We showed that commensal *L. gasseri* increased the secretion of the immune signaling protein TGF- β 1 (Transforming Growth Factor β 1), along with increased expression of its encoding gene signal. TGF- β 1 has an important role in promoting tolerance towards commensal bacteria and has a role in dampening immune responses following inflammation. The pathogenic bacteria had no

effect on the amount of TGF- β 1. Our results indicate that *L. gasseri* could have a way of promoting its own survival in the gut by inducing tolerance towards itself, an effect which pathogenic bacteria do not have. When eicosapentaenoic acid was added to the cell culture model along with *L. gasseri*, there was a greater increase in TGF- β 1 gene expression. This early research shows the potential of combining fish oil with probiotic bacteria to promote probiotic survival in the gut and/or dampening inflammatory responses.

Sterol composition in infant formulas and estimated intake

Claumarchirant, L., et al., *J. Agric. Food Chem.* 63: 7245–7251, 2015, <http://dx.doi.org/10.1021/acs.jafc.5b02647>.

Sterol contents in infant formulas (IFs) from the European market were determined, and their intakes by infants between 0 and 6 months were evaluated. Total animal sterols (mg/100 mL) ranged from 1.71 to 5.46, cholesterol being the main animal sterol (1.46–5.1). In general, cholesterol and desmosterol were lower than the human milk (HM) values indicated by other authors. Total plant sterol (mg/100 mL) ranged from 3.1 to 5.0. β -Sitosterol, the most abundant phytosterol, ranged from 1.82 to 3.01, followed by campesterol (0.72–1.15), stigmasterol (0.27–0.53), and brassicasterol (0.14–0.28). Cholesterol intake (mg/day) ranged from 9 to 51 and plant sterol intake (mg/day) from 19 to 50. The sterol profile of IFs is highly dependent on the type and quantity of fats used in their formula. The use of bovine milk fat and milk fat globule membrane in the IFs can approximate the profile of animal sterols to those found in HM, though cholesterol intakes in breastfed infants are still higher than in formula-fed infants.

Role of conventional and biotechnological approaches in genetic improvement of castor (*Ricinus communis* L.)

Singha, A.S., et al., *Ind. Crops Prod.* 74: 55–62, 2015, <http://dx.doi.org/10.1016/j.indcrop.2015.05.001>.

Castor (*Ricinus communis* L.), one of the most important crops of the family *Euphorbiaceae*, is grown widely for its oleaginous seeds which yield the much valued castor oil. Because of its unique chemical properties, the oil from castor seeds has wide variety of uses and therefore makes castor an industrially important crop. Castor, though being monotypic, has good variability for most of the morphological and agronomic traits, and this variability has also been exploited for its improvement. Great scope still exists for genetic improvement of castor through conventional breeding methods like inter-specific hybridization and induced mutations. However, narrow information of genetics of little and unpredictable yields and susceptibility to diseases and insects are major challenges in successful breeding of castor. Biotechnology offers alternative and effective tools for genetic improvement of castor. The current status of development and use of genetic markers in castor genetic improvement is reviewed. Genetic transformation is also likely to play an important role in future genetic improvement effort in castor. The efforts for development of efficient in vitro propagation system, which is reproducible, rapid, and applicable to a wide range of genotypes, and genetic transformation, are also discussed.

LIPID OXIDATION

Interactions between the lipid core and the phospholipid interface in emulsions and solid lipid nanoparticles

Bricarello, D.A., et al., *Food Biophys.*, August 2015, <http://dx.doi.org/10.1007/s11483-015-9413-4>.

This study evaluates interactions between the lipid core and the phospholipid interface in oil in water emulsions and solid lipid nanoparticles. Interactions between the core and the interface are characterized based on changes in structural order and lateral mobility of the phospholipid interface as a function of physical state of the lipid core (solid vs. liquid) and composition of phospholipids and bile salts at the interface. Changes in structural order of the lipid core are also evaluated as a function of composition of the interface. Emulsions (liquid core) and solid lipid nanoparticles (solid core) are formulated using an eicosane lipid core. Phospholipid with long carbon chain (C16)-high melting phospholipids (41 °C) and short carbon chain (C12)-low melting phospholipid (−1 °C) are selected as emulsifiers. The results of fluorescence anisotropy measurements show that physical state of the lipid core does not significantly influence molecular order of the phospholipid interface. These measurements also demonstrate that molecular order of the lipid core is only marginally impacted by composition of the interface. Excimer formation measurements with pyrene labeled phospholipids illustrate that the composition of phospholipid and bile salts has a significant impact on lateral mobility of emulsifiers at the interface. Results also show that physical state of the lipid core has no significant influence on lateral mobility of emulsifiers at the interface. In summary, these results highlight that properties of phospholipid emulsion interface are a strong function of composition of emulsifiers and co-emulsifiers and are independent of physical state of the lipid core.

The effect of protein-lipid-salt interactions on the sodium availability in mouth and consequent perception of saltiness: as affected by hydration in powders

Yucel, U. and D.G. Peterson, *J. Ag. Food Chem.*, accepted August 2015, <http://dx.doi.org/10.1021/acs.jafc.5b02312>.

There is a broad need to reformulate lower-sodium food products, but without affecting their original taste. In the present study, we focus on characterizing the role of protein-salt interactions on the salt release in low moisture systems and saltiness perception during hydration. Sodium release from freeze-dried protein powders and emulsion powders formulated at different protein:lipid ratios (5:0 to 1:4) were characterized using a chromatography column modified with a porcine tongue. Emulsion systems with protein structured at the interface were found to have faster initial sodium release rates, faster hydration and were perceived to have a higher initial salt intensity with a lower salty aftertaste. In summary, exposure of the hydrophilic segments of the interface-structured proteins in emulsions was suggested to facilitate hydration and release of sodium during dissolution of low moisture powder samples.

Biomimicry—an approach to engineering oils into solid fats

Wang, T.-M. and M.A. Rogers, *Lipid Technol.* 27: 175–178, 2015, <http://dx.doi.org/10.1002/lite.201500036>.

The ability to eliminate trans fats, without incorporating additional saturated fats, is limited by the physico-chemical properties of the processed food and what role the lipids play in the food structure. To maintain the levels of cardio-protective unsaturated fats alternative methods to structure them are desperately needed. One such strategy is to utilize oleogels or molecular gels comprised of small molecules. Herein, we illustrate the potential of biomimicking the assemblies formed by the intercellular lipids in stratum corneum using stearic acid, ceramide III and replacing cholesterol with β -sitosterol.

NMR spectroscopy for assessing lipid oxidation

Hwang, H.-S., *Lipid Technol.* 27: 187–189, 2015, <http://dx.doi.org/10.1002/lite.201500037>.

Although lipid oxidation involves a variety of chemical reactions to produce numerous substances, most traditional methods assessing lipid oxidation measure only one kind of oxidation product. For this reason, in general, one indicator of oxidation is insufficient to accurately describe the oxidation status of lipids, and two or more methods should be used for the accurate assessment of lipid oxidation. An ¹H NMR method that can quantitatively track the disappearance of triacylglycerol starting materials during the oxidation process has been found to be a very reliable method, which gives a satisfactory description of lipid oxidation status throughout the entire course of oxidation, regardless of the kinds of oxidation products produced. It was found that the NMR method not only correlates well with traditional methods, but also it offers additional advantages such as higher accuracy, non-destructiveness, and a faster and more convenient procedure.

LIPIDS ADDS RAPID COMMUNICATIONS

To meet the emerging needs of the lipid research community, *Lipids* is introducing in October 2015 a new rapid communication format designed to get key findings into the literature more quickly. The anticipated turnaround time from first submission to acceptance is expected to be approximately 30–35 days. All rapid communications will be peer reviewed, and authors will have a two-week period in which to revise the manuscript; only minor revisions will be considered.

“The Editorial Board of *Lipids* felt this mechanism was important to institute as it represents a currently unmet need in the field of lipid research,” said Eric J. Murphy, editor-in-chief of *Lipids*. “Just as when the journal began in 1966, we are responding to what we perceive our authors need,” he added. “This is particularly important as we celebrate the 50th volume of *Lipids* this year and look forward to many more years of serving the needs of lipid researchers.”

For the complete Instructions to Authors, visit <http://tinyurl.com/Lipids-Authors>.

Sustainable use of marine resources—turning waste into food ingredients.

Nurdiani, R., et al., *Int. J. Food Sci. Technol.*, online first, July 2015, <http://dx.doi.org/10.1111/ijfs.12897>.

Fish by-products are considered low value and discarded. With proper handling and processing, fish by-products may provide high nutritional value to consumers. This study aimed at establishing compositional differences in valuable compounds from fish by-products of selected species, including Salmon, Flathead, Silver warehou and Barramundi. Simple extraction methods were employed, and obtained fractions were analyzed for their chemical and physical properties. The chemical composition of four fish species differed significantly ($P < 0.05$) with the protein content ranging from 14.7 ± 0.09 to $16.8 \pm 0.41\%$. Adjusting pH to 2.5 yielded two times more of extracted oil than at pH 4.5. Salmon and Barramundi oils contained high amounts of monounsaturated fatty acids (MUFAs) (39–50%), while Silver warehou and Flathead oils contained 46–49% of saturated fatty acids (SFAs). The particle size of the protein fractions was small, ranging from 126.9 to 489.5 nm. Molecular weight of extracted proteins was fish species dependant – Silver warehou and Barramundi samples contained proteins of 250, 120 and 100 kDa, while these bands were absent from Salmon and Flathead samples. The data obtained indicate that extracted fractions from fish by-products likely have high nutritional value and could find a potential use in food formulations.

INDUSTRIAL APPLICATIONS

Investigation of the effects of microalgal cell concentration and electroporation, microwave, and ultrasonication on lipid extraction efficiency

Garoma, T. and D. Janda, *Renewable Energy* 86: 117–123, 2016, <http://dx.doi.org/10.1016/j.renene.2015.08.009>.

This study investigated the effects of *Chlorella vulgaris* (*C. vulgaris*) concentrations and pretreatment methods, electroporation, ultrasonication, and microwave, on lipids extraction. The *C. vulgaris* concentrations were varied in the range of 8.4–28.8% for chloroform/methanol/water solvent system and in the range of 7.6–32.0% for n-hexane/methanol/water solvent system. A maximum total lipid yield of 0.248 g/g of dry *C. vulgaris* was achieved at biomass concentration of about 15% for the chloroform/methanol/water system. This is the highest yield reported for lipids extracted without pretreatment. On the other hand, a maximum lipids yield of 0.139 g/g of dry *C. vulgaris* was obtained at about 24% biomass concentration for the n-hexane/methanol/water system. When pretreated with electroporation, ultrasonication, and microwave, the yield for lipid extraction increased by 5.3, 26.4, and 28.9%, respectively. Although electroporation resulted in the least amount of yield, it was the most efficient in terms of energy gain per energy input.

Biodiesel production from microalgae: influence of pretreatment on lipid extraction

Veillette, M., et al., *Int. J. Sust. Dev. Plan.* 10: 382–394, 2015, <http://dx.doi.org/10.2495/SDP-V10-N3-385-398>.

By having the objective of reducing their global emissions of carbon dioxide (CO₂) and their petroleum dependency, many industrialized countries like European Union countries support the sustainable development and will increase, by 2020, the ratio of biofuel (bioethanol or biodiesel) blend with transportation fuel to 10% (v/v). However, this objective could deprive the world of arable lands needed to feed 320 to 460 million people. To replace conventional vegetable oils (for example, canola oil) to produce biodiesel, microalgae could be used as the bulk material, as their total lipid yield can be as high as 75% (w/w). To produce biodiesel, the lipids must previously be extracted from the wet microalgae. This study showed that microalgae could be directly extracted without dewatering process with a yield of 29.0% (w/w) by using boiling pretreatments (water phase). The yield obtained was slightly lower than the traditional extraction methods (33.0% w/w) implying the costly technique of freeze-drying. The results also showed that the chemical physicochemical pretreatment considered had no influence on the composition of the fatty acid methyl esters of the biodiesel produced with methyl palmitoleate as the major component with up to 28.0% (w/w).

Opportunities for switchable solvents for lipid extraction from wet algal biomass: an energy evaluation

Du, Y., et al., *Algal Res.* 11: 271–283, 2015, <http://dx.doi.org/10.1016/j.algal.2015.07.004>.

Algae are considered an important sustainable feedstock for lipid extraction to produce food ingredients, cosmetics, pharmaceutical products and biofuels. Next to the costs for cultivation, this route is especially hindered by the energy intensity of drying algae prior to extraction and solvent recovery afterwards. Most commonly used lipid extraction methods that can be applied on wet algae biomass were reviewed in this paper. In this work the methods for wet extraction of algae lipids using traditional organic solvents, supercritical CO₂ and CO₂ switchable solvents are compared with dry extraction on an energy consumption basis. Conceptual process designs have been made to calculate and compare the energy flows. Results show that a significant positive energy balance for lipid extraction is only achieved using a switchable solvent extraction method, making this a very promising method for extracting lipids from algae for use in energy applications.

SYNTHETIC BIOLOGY

Metabolic engineering of fatty acid biosynthetic pathway in sesame (*Sesamum indicum* L.): assembling tools to develop nutritionally desirable sesame seed oil

Bhunia, R.K., et al., *Phytochem. Rev.*, <http://dx.doi.org/10.1007/s11101-015-9424-2>, DOI: 10.1007/s11101-015-9424-2, accepted July 2015.

Vegetable oils are an essential component of human diet, in terms of their health beneficial roles. Despite their importance, the fatty acid profile of most commonly used edible oil seed crop plants are imbalanced; this skewed ratio of fatty acids in the diet has been shown to be a major reason for the occurrence of cardiovascular and autoimmune diseases. Until recently, it was not possible to exert significant control over the fatty acid composition of vegetable oils derived from different plants. However, the advent of metabolic engineering, knowledge of the genetic networks and regulatory hierarchies in plants have offered novel opportunities to tailor-made the composition of vegetable oils for their optimization in regard to food functionality and dietary requirements. Sesame (*Sesamum indicum* L.) is one of the ancient oilseed crop in Indian subcontinent but its seed oil is devoid of balanced proportion of ω -6: ω -3 fatty acids. A recent study by our group has shed new lights on metabolic engineering strategies for the purpose of nutritional improvement of sesame

seed oil to divert the carbon flux from the production of linoleic acid (C18:2) to α -linolenic acid (C18:3). Apart from that, this review evaluates current understanding of regulation of fatty acid biosynthetic pathways in sesame and attempts to identify the major options of metabolic engineering to produce superior sesame seed oil.

Bob Moreau (US Department of Agriculture) and Bryan Yeh (Intrexon) are regular contributors to *Extracts & Distillates*.

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The power of "ene" in water-based cleaners and removers

Ron A. Masters, Marshall Nepras, and Mike Wiester

Elevance Renewable Sciences (Woodridge, Illinois, USA) uses Nobel Prize winning Grubbs catalyst technology to produce large quantities of vegetable-oil-based materials at the company's biorefinery in Gresik, Indonesia. These materials include monounsaturated C10 and C12 methyl esters. Stepan Co. uses a range of derivatization chemistries to convert such methyl esters to surfactants and solvents. Recently, researchers at Stepan discovered that the C10 unsaturated methyl ester, converted to N,N-Dimethyl 9-Decenamide, creates a unique molecule that as-is would seem to be a solvent, but in water-based cleaners functions as a low hydrophilic-lipophilic balance (HLB) surfactant that imparts organic solvent-like behavior to water-based formulations. Stepan manufactures the saturated equivalent as a solvent for the agricultural market. The differences between saturated ("ane") vs. unsaturated ("ene") are minor for the materials as-is, but surprisingly major for water-based formulations.

- A range of derivitization chemistries can be used to convert methyl esters to surfactants and solvents.
- In this case, the differences between amides derived from saturated vs. unsaturated methyl esters are minor for the materials themselves, but are surprisingly major for water-based formulations.
- This article describes a unique molecule created from the conversion of an unsaturated methyl ester to a dimethyl amide that imparts organic solvent-like behavior to water-based formulations.

N,N-DIMETHYL-9-DECENAMIDE: MANUFACTURE, CHEMISTRY, PROPERTIES

Both the saturated and unsaturated amides are produced in a single step with purification that yields 97+% pure materials. The molecule is depicted in Fig. 1, by standard drawing and quantum mechanical representation of *ab-initio* calculations of electrostatic charge mapped over electron density.

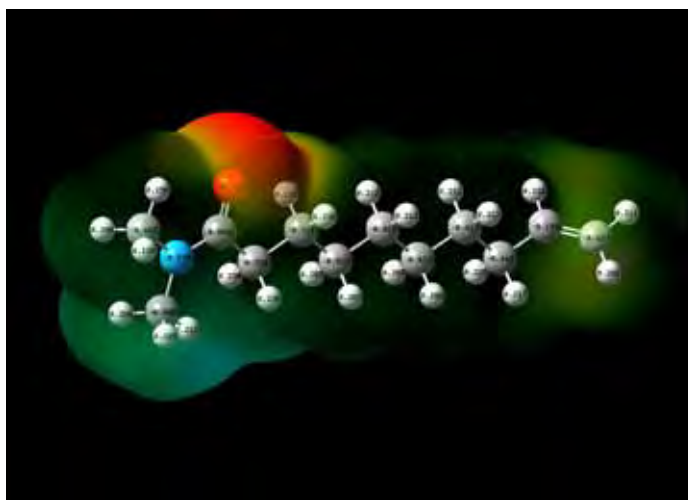


FIG. 1. Electrostatic map of the "ene" molecule

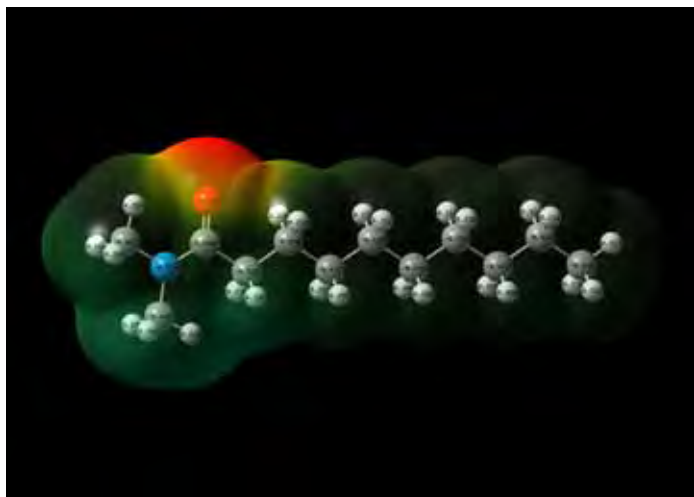


FIG. 2. Electrostatic map of the “ene” amide

Fig. 2 is the same for the saturated amide. Note the extra negative electrostatic charge distribution for the “ene” terminal chain carbons. This could potentially interact with the positive charge distribution at one side of the amide dipole, the main dipole moment for this molecule.

Residual water is controlled to 0.5% maximum, residual ester to 1% maximum, and other residuals to 100ppm maximum. The boiling point at atmospheric pressure is 297 °C, thus classified for reportable volatile organic compounds (RVOC) as 0%, low vapor pressure VOC (LVP-VOC) exempt in cleaning formulations. At room temperature there is no measurable evaporation of thin films. At temperatures of 100 °C to 120 °C, thin films can be made to evaporate over several hours. The measured heat of evaporation at the boiling point is 205 J/g, small enough to perhaps find use in high temperature vapor degreasing applications. The flash point (PMCC) is 134 °C.

Some of the most important properties of N,N-dimethyl-9-decenamide originate from its extremely high Kauri-Butanol (KB) value of >1000, which means that Kauri resin is essentially fully miscible with this unsaturated amide. The saturated amide has been measured to have a high but comparatively lower KB value of about 300. An example KB value for a common solvent is about 67 for d-Limonene.

N,N-DIMETHYL-9-DECENAMIDE: AQUEOUS SOLUTION PROPERTIES

Formulation behavior in aqueous solution arises from an estimated low HLB value of about 6, obtained by comparing water solubility versus known alcohol ethoxylates for which HLB can be calculated, use of Davies method of adding group contributions, and lab testing of various oils and fats with required HLB values.

Water solubility of the unsaturated amide at room temperature is about 0.15%, compared to only 0.05% for the saturated amide. This opens a useful window of opportunity, as the “ene” forms micelles at about 0.1%, observed in the surface tension versus concentration curves in Fig. 3. The curve for the saturated amide is typical for a solvent, showing a gradual decline

in surface tension, in contrast to the surfactant-like curve for the unsaturated amide. Were the “ene” sufficiently soluble, it likely would form micelles too, but the surface tension at its solubility limit is too high, indicating that surface excess is not achieved at concentrations where it is still soluble. The “ene”, in contrast, reaches surface excess and lowest surface tension at a concentration within its solubility limit.

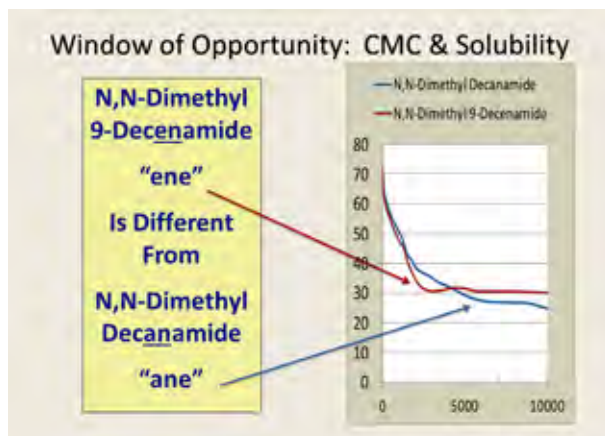


FIG. 3. CMC curves “ene” vs. “ane”

The lab-measured Hansen solubility parameters are $\delta_D=16.58$, $\delta_P=9.58$, $\delta_H=8.45$, with a large radius of 12.9, larger than the 12.5 measured for the saturated amide, with increased hydrogen bonding character. Importantly, in the Hansen screening work, Stepan discovered that ethylene carbonate is miscible with the “ene” but not the “ane” at room temperature, as shown in Fig. 4.

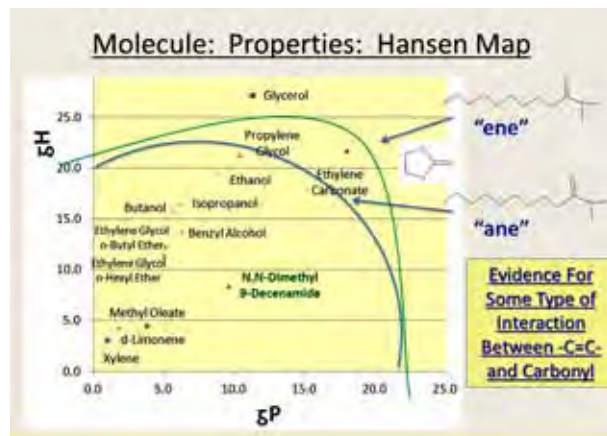


FIG. 4. Hansen map “ene” vs. “ane”

This indicates interaction between the pi-electrons in the terminal double bond of the “ene” with the ethylene carbonate pi-electrons in the carbonyl. This is a consequence of the electrostatic charge distribution differences (Figs. 1 and 2).

In dilute aqueous solution, water associated with the “ene” behaves quite differently from that associated with the “ane,” as observed in attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy bulk-water-subtracted spectra (Fig. 5).

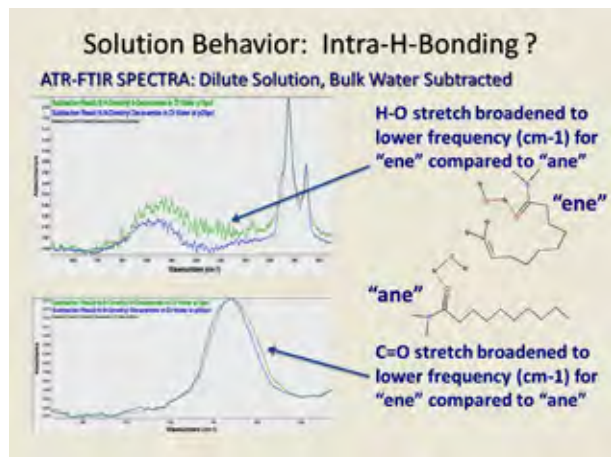


FIG. 5. FTIR “ene” vs. “ane”

Use of the ATR technique analyzes about a micron thickness of adsorbed amide from aqueous solution, along with bulk and associated water. Subtraction of the bulk H-bonded water yields spectra of the amides plus water associated with them. As expected for the saturated amide, a small water peak is observed at about 3500 cm⁻¹. It is understood that water H-bonds to the carbonyl, hence the presence of this small peak after subtraction of bulk H-bonded water. Surprisingly, the O-H stretch for the associated water for the unsaturated amide is strongly broadened toward lower frequency. This indicates the water associated with the “ene” experiences a weaker H-O bond, or “heavier” hydrogen, compared to the “ane,” and across a range of electrostatic environments. These effects could potentially be explained by a loop conformation for the “ene,” potentially driven by the electrostatic charge distribution mentioned earlier.

To research this proposed loop structure, from a fundamental standpoint, *ab initio* density functional calculations were initiated, and are progressing on the potential energy surface to one of the theoretical local minimum structures, using a large basis set. Preliminary calculations with a small basis set, and Hartree-Fock theory, indicated the possibility of a local minimum with a loop configuration that brings the pi electron density at the end of the “ene” chain, into the proximity of the amide head group by a non-bonding interaction. This calculation is still in progress at the time of this writing.

A different and likely more relevant driving force that could favor a loop conformation is the high-energy condition of hydrophobe in dilute aqueous solution. This disfavored high-energy condition drives surfactant hydrophobe chains to surfaces, air, and the interior of micelles. A loop conformation for an isolated molecule in solution (oblate spheroid shape) will present less area of hydrophobe to bulk water than an extended chain conformation (extended rope shape). The saturated amide experiences the same driving force to isolate hydrophobe from water, but would not have a possible electrostatic energy well to favor a loop conformation, and no pi-electrons

in the terminus of the carbon chain to interact with those of the amide. The limited water solubility of the “ane” causes its hydrophobe to associate intermolecularly even in dilute solution and phase split from water at concentrations below surface excess.

A hypothesized loop conformation, oblate spheroid shape for the “ene” would result in faster diffusion through bulk water, compared to the extended chain conformation that is present for the “ane.” The left-most cylinder in Fig. 6 shows nearly instant wetting of a cotton skein (Draves test, ASTM D2281-68) for the unsaturated amide, where the adsorbed air appears to “boil” off the fibers as it enters the dilute solution.

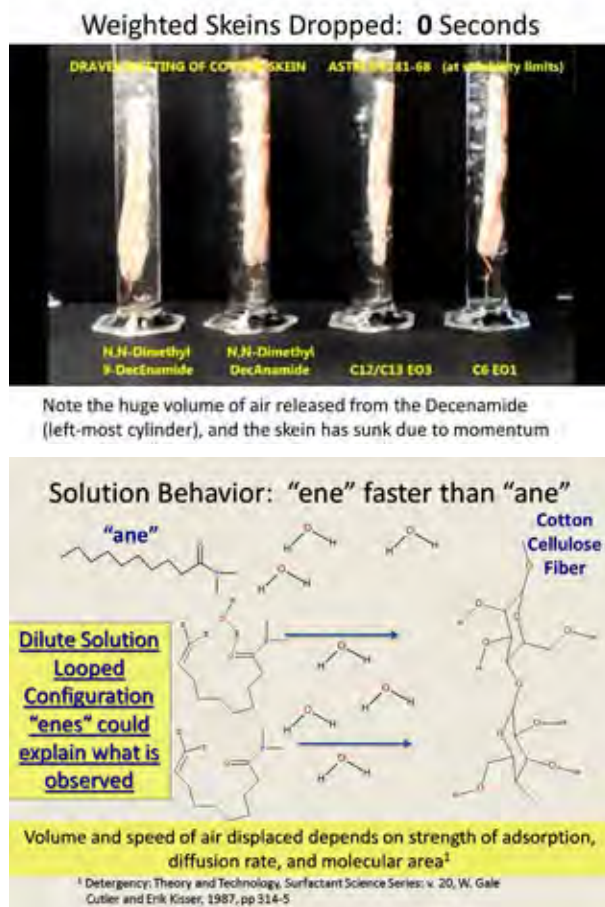


FIG. 6. Draves test, a. wetting of cotton skein (top) b. Draves mechanism (bottom)

In contrast, the saturated amide solution in the adjacent cylinder does not wet the skein until 16 seconds pass. Although both rates are fast, this kinetic wetting difference in dilute aqueous solution lends support to a hypothesized difference in conformation of the molecules. At the saturation limit in dilute aqueous solution, the “ene” will contain micelles, leading to slower expected kinetics rather than faster, compared to the “ane” which would be present mainly as monomers. Thus, it is challenging to explain this large difference in kinetics for two molecules that are so similar, without invoking H-bonding or conformational comparisons.

Proton nuclear magnetic resonance (H-NMR) studies are being conducted to further examine a hypothesized loop conformation. First, comparative tests of dilute solutions of the “ene” versus “ane” in D₂O do not show any difference in

chemical shift for the amide protons when run at identical temperatures (25 °C). This is being repeated in cyclohexane-D12 to compare with the D2O results. The chemical shifts are expected to change with the change in solvent: D2O hydrogen bonds with the amides, whereas cyclohexane-D12 does not. If the changes in shift due to change of solvent differ for the “ene” versus “ane,” that could lend support for the hypothesized conformation. Separately, Pulsed Gradient Spin Echo (PGSE) Diffusion Ordered Spectroscopy (DOSY) experiments have been run to measure diffusion rates of “ene” vs. “ane” in dilute D2O solution. These are definitive, and reveal a slower rate of $\approx 3.5 \times 10^{-6} \text{ cm}^2/\text{sec}$ for the “ene,” and faster rate of $\approx 4.9 \times 10^{-6} \text{ cm}^2/\text{sec}$ for the “ane,” confirming micelle structures for the “ene” compared to monomer for the “ane.” Residual H2O in D2O shows equal diffusion rates of $1.73 \times 10^{-5} \text{ cm}^2/\text{sec}$ for both “ene” and “ane,” validating the runs. So, the challenge to understand the faster kinetics of wetting remains, and perhaps is made more challenging by these results. Also, one could argue that the temperature of these experiments would need to be low enough to freeze favored conformations. However, the differences in properties and performance between the “ene” and “ane” are observed at room temperature, so if conformational structure is responsible, it should be observed in the data even at room temperature, as it is for the FTIR data.

N,N-DIMETHYL-9-DECENAMIDE: APPLICATIONS

By associating with water in the conformation described above, very interesting dilute aqueous solution applications become possible. Figs. 7 and 8 show two of many different applications: nail polish remover and Latex dissolution (chewing gum). Typically, these applications require large proportions of organic solvents to produce effective products. Nail polishes historically use acetone, ethyl lactate, ethyl acetate, propylene carbonate, and similar solvents, with very little water present. This creates objectionable fumes and potential skin irritation, and can also affect the underlying nail. Chewing gum and other latex dissolvers typically contain methylene chloride and d-Limonene. That a water-based solution containing 80+% water could disintegrate this polymer is quite unexpected. Examining Figs. 7 and 8, one can see that an 85%–87% water-based cleaner, utilizing small amounts of carbonate and d-Limonene, and/or MEA, is capable of both removing nail polish and disintegrating latex-based chewing gum.

Applications: Nail Polish Removers

Requires >50% Acetone and/or Ethyl Acetate
Typically contain 90+% Organic Solvent

85% Water
5% N,N-Dimethyl 9-Decenamide
5% MEA

OR

87% Water
3% N,N-Dimethyl 9-Decenamide
1.5% Citrus Fragrance
0.3% Sodium Carbonate
Balance: High HLB co-emulsifier

Patent Pending
blend with great
synergy and new
mechanism(s)
WO2013162926(A1)

“Making Water Work Like an Organic Solvent”

FIG. 7. Nail polish removal

The rates in water-based solution are slower than if the N,N-dimethyl-9-decenamide were combined with d-Limonene or methylene chloride in a water-free composition, but the rate is sufficiently fast to be useful. Water-based formulas have reduced odor and contain their own follow-up cleaning agents along with the water to rinse, all in one step. Typically, solvent-based products require follow up treatment with a water-based product to complete the cleaning. Note that Fig. 8 shows comparative attempts to dissolve latex using alkaline cleaners and degreasers and solvents.

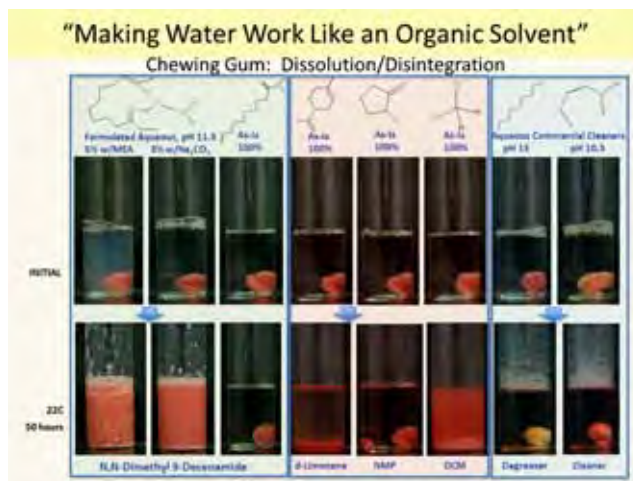


FIG. 8. Latex gum dissolution

The only solvents that function from the selected group are methylene chloride and d-limonene. Even 100% N,N-dimethyl-9-decenamide fails to affect the gum. But, when formulated into dilute aqueous solution, the effect is dramatic. This extends to other latex cleaners as well, such as brush cleaners and “waterless” hand cleaners for painters and mechanics.

Ron A. Masters is a research associate at Stepan Company. He can be contacted at RMasters@stepan.com. Marshall Nepras is also a research associate, and Mike Wiester is a senior research chemist at Stepan.

The entire Stepan company team is to be thanked for their role in the metathesis derivative program and the commercialization of STEPOSOL® MET-10U, the Surfactant Solution to Solvent Replacement™.

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Gas Chromatography

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Gas Chromatography

Honorable Mention
Jeremy Dehner
ADM Clinton
Clinton IA 52732 USA

Gas Chromatography

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Diane Simmons,
Rosalin Manalang
California Oils Corp
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Gas Chromatography

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Helen Cianciolo, Derek Gum
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New Zealand

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Oilseed Meal

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Oilseed Meal

Honorable Mention
Ardin Backous,
Anders Thomsen,
Brian Gilchrist, Keith Persons,
Kent Karsjens
Eurofins Scientific
Des Moines IA 50321-3157
USA

Oilseed Meal

Honorable Mention
Mumtaz Haider
Inspectorate America Corp
Webster TX 77598 USA

Oilseed Meal

Honorable Mention
Amanda Self
Barrow-Agee Laboratories
Memphis TN 38116-3507
USA

Oilseed Meal

Honorable Mention
Tuyen Mai
Intertek Agri Services
New Orleans LA 70122 USA

Oilseed Meal 100%

Crude Fiber
First Place
Gordon Whitbeck,
John Dillard
Whitbeck Laboratories Inc.
Springdale AR 72764-2204
USA

Oilseed Meal 100%

Crude Fiber
Honorable Mention
Brad Newton Beavers,
Jennie Stewart
Carolina Analytical Services
Bear Creek NC 27207 USA

Oilseed Meal 100%

Crude Fiber
Honorable Mention
Renato M. Ramos
Admiral Testing Services
Luling LA 70070-4060 USA

Oilseed Meal 100%

Crude Fiber
Honorable Mention
Mumtaz Haider
Inspectorate America Corp
Webster TX 77598 USA

Oilseed Meal 100% Moisture

First Place
Mike White, Brian Eskridge
ATC Scientific LLC
N Little Rock AR 72114-5120
USA

Oilseed Meal 100% Moisture

Honorable Mention
John Reuther, Eric de Ronde
Eurofins Central Analytical
Labs
New Orleans LA 70122 USA

Oilseed Meal 100% Moisture

Honorable Mention
Tuyen Mai
Intertek Agri Services
New Orleans LA 70122 USA

Oilseed Meal 100% Moisture

Honorable Mention
Janet Smith
Fieldale Farms Corp
Baldwin GA 30511 USA

Oilseed Meal 100% Moisture

Honorable Mention
Sandy Harrison
Illinois Crop Improvement
Assn.
Champaign IL 61822 USA

Oilseed Meal 100% Moisture

Honorable Mention
Chin Chaothaworn
SGS Thailand Ltd
Bangkok 10120 Thailand

Oilseed Meal 100% Moisture

Honorable Mention
CHS Israel QA Laboratory
CHS Israel
Ashdod 77121 Israel

Oilseed Meal 100% Nitrogen

Ba 4d-90
First Place
Aaron Jusko
SGS North America
Agricultural Div
Deer Park TX 77536-2518
USA

Oilseed Meal 100% Nitrogen

Ba 4e-93
First Place
Frank Tenent, Edgar Tenent
K-Testing Lab Inc
Memphis TN 38116-3507
USA

Oilseed Meal 100% Nitrogen

Ba 4e-93
Honorable Mention
Paul Thionville, Boyce Butler,
Andre Thionville,
Kristopher Williams
Thionville Laboratories, LLC
New Orleans LA 70123-3227
USA

Oilseed Meal 100% Nitrogen

Ba 4e-93
Honorable Mention
Renato M. Ramos
Admiral Testing Services
Luling LA 70070-4060 USA

Oilseed Meal 100% Nitrogen

Ba 4e-93
Honorable Mention
Tuyen Mai
Intertek Agri Services
New Orleans LA 70122 USA

Oilseed Meal 100% Oil

First Place
George Ducsay, Bruce Kerr
Isotek Laboratories LLC
Oklahoma City OK 73127-5801 USA

Oilseed Meal 100% Oil

Honorable Mention
Tuyen Mai
Intertek Agri Services
New Orleans LA 70122 USA

Oilseed Meal 100% Oil

Honorable Mention
Sandy Holloway
Intertek Agri Services
Memphis TN 38113 USA

Oilseed Meal 100% Oil

Honorable Mention
Mumtaz Haider
Inspectorate America Corp
Webster TX 77598 USA

Oilseed Meal 100% Oil

Honorable Mention
Renato M. Ramos
Admiral Testing Services
Luling LA 70070-4060 USA

Oilseed Meal 100% Oil

Honorable Mention
Melinda Graham
Hartsville Oil Mill
Darlington SC 29540-1027 USA

Olive Oil Part A

First Place
Jamie Ayton
NSW Dept of Primary Industries
Wagga Wagga NSW 2650 Australia

Olive Oil Part A

Honorable Mention
William Spence, Ryan Cole
SGS North America
St Rose LA 70087-4030 USA

Olive Oil Part B

First Place
Maria Garzon
Pompeian Inc
Baltimore MD 21224-1699 USA

Olive Oil Part C

First Place
Dr Giorgio Cardone
Chemiservice SRL
Monopoli Bari 70043 Italy

Palm Oil

First Place
Specialty Fats Lab
PT. Musim Mas
Medan N Sumatra 20371 Indonesia

Palm Oil

Honorable Mention
James Houghton
AAK
Louisville KY 40208 USA

Palm Oil

Honorable Mention
Magdy Rashwan
IFFCO Egypt
Suez 204 Egypt

Palm Oil without SFC

First Place
Specialty Fats Lab
PT. Musim Mas
Medan N Sumatra 20371 Indonesia

Palm Oil without SFC

Honorable Mention
Mrs. Hajar Musa
Malaysian Palm Oil Board
AOTD
Selangor 43000 Malaysia

Palm Oil without SFC

Honorable Mention
Ai Tin Khor
ITS Testing Services (M) Sdn Bhd
Port Klang Selangor 42000 Malaysia

Peanut Seed

First Place
Brownfield Lab Analytical Team
JLA Intl
Brownfield TX 79316 USA

Phosphorus in Oil

First Place
Maria Lina Dionisio
Sovena Oilseeds
Almada 2801-801 Portugal

Phosphorus in Oil

Honorable Mention
QA Laboratory Technicians
Riceland Foods Quality Assurance Lab
Stuttgart AR 72160 USA

Phosphorus in Oil

Honorable Mention
Heather M Brogan
Pacific Coast Canola
Warden WA 98857 USA

Solid Fat Content by NMR

First Place
Bill Zubrinic
Bunge Canada
Hamilton ON L8N 3K7 Canada

Solid Fat Content by NMR

Honorable Mention
Eddie L Baldwin,
Helen Cianciolo, Derek Gum
Stratas Foods RDI Center
Bartlett TN 38133-4009 USA

Soybean Oil

First Place
Renato M. Ramos
Admiral Testing Services
Luling LA 70070-4060 USA

Soybean Oil

Honorable Mention
Paul Thionville, Boyce Butler.
Andre Thionville,
Kristopher Williams
Thionville Laboratories, LLC
New Orleans LA 70123-3227 USA

Soybeans

First Place
Renato M. Ramos
Admiral Testing Services
Luling LA 70070-4060 USA

Soybeans

Honorable Mention
Tuyen Mai
Intertek Agri Services
New Orleans LA 70122 USA

Soybeans

Honorable Mention
Mumtaz Haider
Inspectorate America Corp
Webster TX 77598 USA

Specialty Oils

First Place
Jocelyn Alfieri
Silliker Canada Co
Markham ON L3R 5V5 Canada

Tallow & Grease

First Place
Jose Garcia
National Beef Packing Company
Liberal KS 67901-2879 USA

Tallow & Grease

Honorable Mention
Adalberto Coronado
National Beef Packing Company
Liberal KS 67901-2879 USA

Tallow & Grease

Honorable Mention
Montreal Analytical Team
Sanimax Inc
Montreal PQ H1C 1G2 Canada

Tallow & Grease

Honorable Mention
LaboratoryTeam
Sanimax Aci Inc
Charny PQ G6X 3R4 Canada

Trace Metals in Oil

First Place
Jitendra Patel
Viterro Canola Processing
Ste Agathe MB R0G 1Y0 Canada

Trace Metals in Oil

Honorable Mention
Heather M Brogan
Pacific Coast Canola
Warden WA 98857 USA

trans by GC

First Place (tie)
QA/QC Laboratory
ADM Mankato Refinery
Mankato MN 56001 USA

trans by GC

First Place (tie)
Paul Thionville, Boyce Butler,
Andre Thionville,
Kristopher Williams
Thionville Laboratories, LLC
New Orleans LA 70123-3227 USA

trans by GC

Honorable Mention
Specialty Fats Lab
PT. Musim Mas
Medan N Sumatra 20371 Indonesia

trans by GC

Honorable Mention
Tae Sugiyama
The Nisshin OilliO Group Ltd.
Yokosuka 239-0832 Japan

trans by GC

Honorable Mention
Wakako Tsuzuki
National Food Research Institute
Tsukuba Ibaraki 305-8642 Japan

trans by GC

Honorable Mention
Ang Chee Loong
PGEO Edible Oils Sdn Bhd
Pasir Gudang Johor 81707 Malaysia

trans by GC

Honorable Mention
Bill Zubrinic
Bunge Canada
Hamilton ON L8N 3K7 Canada

trans by IR

First Place
QA/QC Laboratory
ADM Mankato Refinery
Mankato MN 56001 USA

Unground Soybean Meal

First Place (tie)
Michael Hawkins, Amanda Self
Barrow Agee Labs LLC
Memphis TN 38116-3507 USA

Unground Soybean Meal

Honorable Mention
Ms Bui Thi Ngoc Chi
Bunge Vietnam Limited
Ba Ria Vung Tau Vietnam

Unground Soybean Meal

Honorable Mention
Mike White, Brian Eskridge
ATC Scientific LLC
N Little Rock AR 72114-5120 USA

Unground Soybean Meal

Honorable Mention
John Reuther, Eric DeRonde
Eurofins Central Analytical Labs
New Orleans LA 70122 USA

Unground Soybean Meal

Honorable Mention
Frank Tenent, Edgar Tenent
K-Testing Lab Inc
Memphis TN 38116-3507 USA

Vegetable Oil Color Only

First Place
George Hicks
Dallas Group
Jeffersonville IN 47130-9607 USA

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