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When it comes to soft materials, identifying links between formulas and processing, structure, and consumer perceivable physical properties is challenging. This article looks at relevant characterization methods and where to find them.
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During the past 100 years, the world’s most extensive and respected collaborative proficiency testing program for oil- and fat-related commodities, oilseeds, oilseed meals, and edible fats has grown to include more than 40 different series. AOCS continues to adjust and improve methods to keep pace with demands in the fats and oils industries; in 2019, the LPP will add a series on pulses analysis after an introductory partial-year test.

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The common feature of most soft materials—foods, cosmetics, personal and home care products—is the existence of an internal structure created by particle aggregation or the self-assembly of molecules, and by processing. This structure can change over time. It can change as the material is spread or kneaded or applied to skin, diluted, heated, cooled, chewed, or squeezed out of a bottle. It controls the performance and consumer perceivable attributes of soft materials based products.

• The performance of commercially relevant soft materials is controlled by their internal structure. Understanding this structure is important for both product development and quality control.

• Structural characterization requires a range of specialized techniques and expertise. Not even the largest companies have all of the equipment in-house.

• Identifying the links between formulas and processing, structure, and consumer perceivable physical properties is challenging. This article summarizes relevant characterization methods and identifies where to find them.

If a development team wants to move beyond iterative line-extensions and develop a completely new product (to use new types of bio-sourced ingredients or to meet a new consumer need, for example), they begin by understanding this structure. Structural characterization can also solve quality control mysteries. When a product goes out of specifications, it may be because the internal structure has changed unexpectedly.

Not even the largest consumer products, cosmetics, or foods company has all the advanced characterization equipment that may be relevant, and many restrict their in-house capabilities to techniques that will be used regularly for quality control. For researchers interested in forging new links between formulation, structure, and performance, an obvious destination might be their local university-based characterization facility. In recent years many US-based academic institutions have “put out a shingle” and will provide a materials characterization service, for a price.
Challenges for these methods, both for sample preparation and data analysis. It is rare to find user facilities that have experience with these materials.

“High-energy beam techniques, such as electron microscopy, break organic bonds and can alter the structure of soft materials,” explains Haugstad. “Both electron beam and X-ray scattering methods rely on variations of electron density but the variations in organic materials are meager, requiring selective staining with heavy elements. The lack of electrical conductivity can result in surface charging. You often need a thin layer of metal coating on the sample. Sample preparation can be very challenging for these soft organic materials.”

Haugstad and his colleagues at the CharFac have worked with well over 100 companies and a considerable amount of their work has been with soft materials, gels, and suspensions.

“There are the additional issues with materials that have liquid phases, such as colloidal suspensions,” explains Haugstad. “Vacuum-based methods such as electron microscopy require solid samples. The most common approach is to freeze the sample using cryo-vitrification. Liquid is wicked away on a metal mesh scaffold (the TEM grid), and then the sample is plunged into liquid ethane.”

These methods are quite well developed for simple aqueous systems, but not for non-aqueous systems, and for more complex or gel phase systems Haugstad sounds a particular caution.

“There are open questions regarding the veracity of imaged structures; I’ve seen published examples where the structure is induced by the cryo-vitrification process rather than being a characteristic of the material itself.”

It’s a reason to choose your characterization facility with care!

BRING IN THE FEDS

Alternative, and potentially free, options for accessing sophisticated analytical equipment in the United States are provided by the US Department of Defense (DOE)-funded Nanoscale Science Research Centers (NSRCs) and by other government-funded facilities such as NIST. These world-class facilities can be used by both academic and industrial researchers at no cost—if the proposed studies are deemed sufficiently interesting by a panel of expert (generally academic) reviewers. Although the majority of the techniques at the NSRCs are focused on characterizing and controlling the nanoscale structure of hard materials, such as next generation alloys and ceramics, there are some methods that are applicable to foods, cosmetics, or personal and home care products. For example, researchers from PepsiCo recently used 3D X-ray imaging capabilities at the Berkeley Lab in Berkeley, California, to study changes in structure as starch pellets are transformed into an aerated popped snack. And there is a very good reason to consider some of these research centers: They have neutron sources.

YOU MAY NEED NEUTRONS

Neutron scattering and neutron imaging are good at revealing structure in soft matter. X-rays or electron beams cannot easily reveal structures that are comprised of lighter elements such as carbon, oxygen, and hydrogen. But neutrons are even scattered by hydrogen.

The Oak Ridge National Laboratory in Tennessee has two of the most powerful neutron science facilities in the world—the High Flux Isotope Reactor, and the Spallation Neutron Source.

“We have a range of instruments that can be used to study soft matter,” says Volker Urban, an instrument sci-
Neutron scattering experiments reveal the shape and conformation of nano- to micron-scale structures. For example, in a microemulsion, SANS can measure the average droplet size, and also the size of the corona. It can reveal the number of lipids or surfactants stabilizing the emulsion droplets and study this over time to get exchange kinetics. In a surfactant solution, SANS can differentiate between spherical micelles, worm-like micelles, or lamella. In a lipid membrane, it can be used to quantify membrane fluctuations and lipid diffusion.

Neutron imaging provides a complementary picture of a specific part of the system.

“It’s like X-ray radiography, but while X-ray shows the heavy elements we can show where the hydrogens are which is important for soft matter,” explains Urban.

The technique depends on how different materials attenuate the neutron beam; the image is the shadow that is cast under the neutron illumination. Of particular value is the ability to use neutrons for contrast—to differentiate between different materials based on the relative percentage of hydrogen.

“Lipids or surfactants with long alkyl tails have lots of hydrogen,” says Urban. “Proteins also have hydrogen but also carbon, oxygen, and nitrogen. You can tune the neutron imaging or scattering experiment to see one or the other, for example to characterize lipid/protein complexes. In a food product you may want to know about the distribution of oil or water. In a system with carbohydrate, protein, and fat you can see where the fat is.”

X-rays or electron beams can damage soft samples, but the non-destructive nature of SANS and Neutron imaging means that the same samples can be characterized using several different techniques.

“Neutrons are low energy so you don’t get radiation damage. After the sample is scanned to check that it has not picked up any radioactivity, it can be handed back and the same material can be used in other characterization methods. We have found it particularly valuable to combine spectroscopic methods—IR and Raman—with neutron scattering,” says Urban.

Urban and his colleagues are interested in studying commercially relevant soft materials, such as foods, personal/home care products, cosmetics, and so on. He encourages industrial researchers to contact the Instrument Scientists at Oak Ridge before submitting a proposal.

“The reviewers recommend proposals based on the novelty of the research or the material,” he notes. “And unfortunately, not many industrial proposals are accepted based on these criteria. But, we can provide assistance with proposal writing, and while 75% of our time is devoted to those projects...
that pass the peer review, 25% of our time is discretionary. We use that time to develop our techniques, but we can also work on our own projects with industry. Contact us and talk to us!”

OPPORTUNITIES IN EUROPE

Peter Schurtenberger is a professor of physical chemistry at Lund University in Sweden who has considerable experience in characterization of colloids and complex soft matter systems. In particular, he is known for the development of dynamic light scattering techniques that can characterize very turbid colloidal suspensions such as milk (commercialized by LS Instruments in Fribourg, Switzerland). In tune with all the experts interviewed for this article, he emphasizes the value of using multiple characterization methods.

“Basic understanding and rational design of soft matter—liquids, colloids, polymers, foams, gels, granular materials, liquid crystals, and biological materials—requires access to a variety of advanced, non-standard equipment, and a broad spectrum of expertise,” says Schurtenberger. “You need static and dynamic characterization over different length and time scales.”

Schurtenberger’s Institute participates in the European Soft Matter Infrastructure (EUSMI) program that provides both industrial and academic users free access to a wide range of state-of-the-art characterization facilities across Europe (including funding for their travel and accommodation).

“We want researchers from industry or academia anywhere in the EU to be able to access the world-class facilities that have been established for this purpose,” he says.

The EUSMI program includes optical and electron microscopy tools (University of Edinburgh, UK; University of Bayreuth, Germany; and the University of Antwerpen, Belgium), light scattering (Lund University, Sweden; FZ Jülich, Germany; and FORTH Crete, Greece), neutron scattering (Jülich Center for Neutron Scattering, Munich, Germany), and X-ray scattering (Paul Scherrer Institute, Switzerland). There are also supercomputers for modeling work (FZ Jülich, Germany). In all these locations, the staff members who work with researchers are experts in handling soft materials. And this wealth of characterization methods is not exclusively for EU-based companies.

“It is important to point out that 20% of the access time is accessible to groups where a majority of the users are not working in the EU,” says Schurtenberger.

CALL THE CONCIERGE

With so many choices in Europe, identifying the most appropriate techniques and the people with the most appropriate expertise can be challenging. This is where Marc Obiols-Rabasa, the Industrial Liaison Officer (ILO) for the EUSMI program, comes in.

“My role is to help industrial users to identify projects that could benefit from EUSMI, and look for the most suitable infrastructure to characterize their material,” he says. “I provide support as they write the proposal, make the introductions, and get their visit to the chosen facility settled. The industrial user never has to take a decision alone. The ILO is there to assist.”

The EUSMI program is only a year old but it has already attracted some industrial users.

“There have been eight companies that have already performed studies,” says Obiols-Rabasa.

BUT WHAT DOES IT MEAN FOR MY PRODUCT?

If you are looking to go beyond characterization of your material, and to develop a model to relate structure to performance, then a longer term relationship with an academic research group may be a good approach.

---

Three approaches to characterizing soft materials

There are three general classes of characterization methods that can reveal structure in commercially relevant soft materials:

1. **Scattering methods** can characterize structure within a soft material. Soft materials are somewhat transparent to radiation such as electrons, X-rays, neutrons, and in many cases to light (visible, UV, or Infrared). The radiation is scattered by structures within the material, and the scattering pattern can be fitted to models to reveal the sizes and shapes of ordered nanoscale to micron-sized structures (the existence of characteristic distances). Depending on the type of radiation that is being scattered by the sample (light, electrons, X-rays, neutrons) and the angle at which the radiation hits the object and is scattered, the ordered arrangements of atoms (crystals) or the prevalence, sizes, and shapes of larger structures such as micelles and colloidal particles can be deduced.

2. **Microscopy** techniques use reflected radiation (light, X-rays, electrons, neutrons) to provide an image of specific portion of the material—the outside surface, or of a slice extracted from the interior of a frozen sample. Atomic force microscopy (AFM) measures surface attributes such as height and friction with a very sharp probe testing the mechanical properties of the material or providing a topographical map as the probe is rastered across the surface.

3. **Rheological methods** measure how easy it is to deform (squeeze or shear) a material, but in the hands of an expert the subtle details of that response can suggest much about the internal structure.

Commercially relevant soft materials can also be studied using the wide range of **spectroscopy** methods designed to reveal chemical structure (which molecules are present)—and this can be useful in situations where chemical degradation is suspected. Special spectroscopy techniques (e.g., diffusion and spin echo methods in NMR) can be used to deduce larger scale structures.
There are a number of groups that would be interested in industrial funding to support their work discerning the fundamental relationships between structure and physical properties in soft materials. These groups typically use simple model systems, and want to obtain data that is publishable. Companies may retain the PI as a consultant, and value the laboratory as a training ground for future hires. But to characterize commercial products, and solve real-world problems, companies may wish to target the smaller number of groups that are interested in this kind of work.

“When companies are looking for a partner to work with, experience of a characterization method is not enough. The group needs to be interested in working with complex materials, interested in solving industrial problems,” says Paschalis Alexandridis, a professor at the University at Buffalo in New York State, USA.

Alexandridis is known for his work characterizing and manipulating the self-assembly of amphiphiles, such as surfactants, lipids, proteins, and block copolymers—the building blocks for soaps and detergents, foods, coatings, and ink.

“My most common interactions with industry occur when an established product is not performing well, often due to a change in ingredients. We look at the nanostructure—the structure that comes from molecular interactions and from processing. Understanding this will enable you to understand whatever challenges you face with your product. If you are creating new materials, or introducing new ingredients, you can design based on knowledge of this structure,” he says.

Commercial products are necessarily complex, with broad molecular weight distributions, side-products, and additives (fragrances, flavors, preservatives) that introduce many vari-

### List of characterization services

We expect this list to grow. The most recent version can be found at https://docs.google.com/spreadsheets/d/1E-23hPKRyV1cWUWIW6d0Y1EVKjfcpl2rpMYY0eA1tCa/edit?usp=sharing. Also, if you provide structure characterization services and have a proven track record working with commercially relevant soft materials (foods, cosmetics, personal, or home care materials) please let us know at https://goo.gl/forms/Bg2GBD21OATE3kee2.

#### US UNIVERSITY CHARACTERIZATION FACILITIES:

A list of characterization facilities supported by the National Science Foundation’s Material Research Science and Engineering Centers initiative can be found here: https://mrfn.org/

- There are 23 characterization facilities, offering 1,155 instruments and 262 expert users! But the vast majority of the work carried out in these facilities is not focused on complex soft materials such as those found in foods, personal or home care products or cosmetics. The following facilities have specialized equipment and expertise for soft matter characterization:
  - The University of Minnesota Characterization Facility provides analytical services, machines for independent use (with training). http://www.charfac.umn.edu/about.html. Contact Greg Haugstad: cfac-dir@umn.edu.
  - The Center for Particulate and Surfactant Systems at the University of Florida and Columbia University in New York includes optical microscopy, light scattering, AFM, and rheometry and has a number of industrial members. http://cpass.mse.ufl.edu/. Contact Hollie Starr: info@perc.ufl.edu.
  - Stanford University Soft & Hybrid Materials Facility (SMF) does not provide analytical services, but they do provide machines for independent use (with training), https://smf.stanford.edu/equipment/smf/index.html. Contact tobi@stanford.edu.
  - The Soft Matter Facility, Texas A&M University will be opening shortly. It will provide analytical services and machines for independent use (with training). https://somf.engr.tamu.edu/. Contact Svetlana Sukhishvili: svetlana@tamu.edu.

#### US GOVERNMENT FACILITIES FOR SOFT MATTER CHARACTERIZATION USING NEUTRONS:

- Neutron scattering and imaging facilities at Oak Ridge National Laboratory: https://neutrons.ornl.gov/. Contact Volker S. Urban, urbanvs@ornl.gov, or neutronusers@ornl.gov.

#### EUROPEAN SOFT MATTER INFRASTRUCTURE (EUSMI) PROGRAM:

Free access to characterization facilities focused on soft materials at 15 top-level institutions (available for users outside Europe) https://eusmi-h2020.eu/. Contact Marc Obiols-Rabasa, the Industrial Liaison Officer (ILO): marc@crcom.se.

#### CONSULTANTS AND CHARACTERIZATION SERVICES WITH SPECIFIC INTEREST/EXPERTISE IN CHARACTERIZING COMMERCIALLY RELEVANT SOFT MATERIALS:

- SurfaceChar; contact Dalia Yablon: info@surfacechar.com.
- CR. Contact Anna Stenstam: anna@crcom.se.
ables. Characterization results are challenging to interpret, and publication can be difficult. Like most applications-focused research groups, Alexandridis’s team uses a range of analytical techniques, starting with the simplest.

“I assign great value to optical microscopy and rheology,” he says. “These techniques, if used correctly, can reveal information about the sizes and textures of the internal structures and their response during processing or consumer use. We also use X-ray diffraction to characterize the crystallinity in materials such as lipids. Commercial materials are always multi-component systems, but you can still get insight if you apply multiple characterization methods to the same system.”

Alexandridis is interested in establishing long-term relationships with industrial groups. He appreciates the funding, but also values the interesting challenges and industrially relevant experience it provides for his students.

“Academic and industrial researchers can learn together how to characterize the specific types of material in the commercial product, and to understand the link between structure and product,” he says. “I can use industrial money to pay the students who work on the industrial project (in addition to other projects). Publication is not essential; it is not the motivation for me.”

Julian McClements, a professor in the Department of Food Science at UMass Amherst, is also interested in working on longer-term products studying commercially relevant materials. McClements specializes in food biopolymers and colloids, and in the development of food-based delivery systems for bioactive components. He and his team have considerable experience in characterizing colloidal systems, such as emulsions, nanoemulsions, starch granules, and biopolymer nanoparticles using scattering methods and microscopy. For example, in a recent work they characterized the surface texture and nanoscale structures of potato granules using confocal laser scanning microscopy (CLSM) and scanning electron microscopy (SEM), and correlated this with the pasting properties of the starch.

“We tend to work with industry on specific contracts (typically 6 months to 2 years). My laboratory rarely does one-off characterization projects,” McClements says.

A longer term project may make it easier to justify the time and effort required to set up the collaboration, which can be considerable in the United States.

“The business aspects of setting up collaboration can be challenging,” says Alexandridis. “It’s easier to bring your expertise into corporate research in Europe. US universities view industrial research as a moneymaking opportunity, and there are challenges about assigning intellectual property. It has become increasingly complex in recent years, and this can add significant time and cost.”
McClements agrees, although he does see some recent improvements as universities recognize the barriers they have erected to collaboration.

"UMass has revamped its office dealing with academic-industry affairs making it much quicker to get contracts started," he says.

Alejandro G. Marangoni, a professor at the University of Guelph in Canada, is known for his work on characterizing and manipulating lipid systems—in particular for applying X-ray diffraction methods to the characterization of foods, biolubricants, and cosmetics. He faces a somewhat easier climate for industrial collaboration than his US colleagues.

"We do a lot of powder X-ray diffraction for companies. Their needs are pretty complicated," he says (Fig. 2, page 11).

Lipid crystallization and melting behavior is strongly influenced by other components in the system, for example the gluten in wheat flour in baked goods. In a recent example, Marangoni used powder X-ray diffraction to elucidate the crystal structure in a croissant, assessing the solid fat content by pulsed nuclear magnetic resonance (NMR), and the thermal behavior by differential scanning calorimetry (DSC). The work provided insight into the structures that determine baking behavior and mouth-feel and predicted the consequences of ingredient changes in laminated bakery products.

"A university-based analytical service laboratory may have the equipment, but not the personal experience to characterize these materials effectively," says Marangoni. "In an academic setting many professors would prefer to work on simpler model systems which can provide publishable data. There is a pretty big disconnect between the need and the supply for characterization services."

THE FULL SERVICE

The best approach, particularly if you need information fairly quickly, may be to work with a characterization consultant or consulting company with experience in your specific industry.

Dalia Yablon runs the SurfaceChar AFM measurement service in Sharon, Massachusetts.

"I’ve recently characterized both food materials and personal care products on behalf of industrial clients," she says.

Atomic Force Microscopy (AFM) is often called on to measure sample adhesion and modulus in addition to providing a topological map of the sample surface (no other technique can do this on such a small length scale).

"I’ve studied properties of hair as a function of conditioner use or treatment type," says Yablon. "I’ve also identified the nano- to micron-scale structures responsible for unwanted stickiness during food processing, and characterized the adhesion of edible food coatings. These types of material are challenging because the samples tend to be heterogeneous and can be difficult to handle. Even relatively simple AFM topography imaging can give misleading results if it is not conducted correctly." (Fig. 3)

CR Competence AB in Lund, Sweden, works with about 20 companies each year, and is focused on characterization of commercially relevant soft materials using a wide range of techniques including scattering techniques (light, X-rays, and neutrons), surface imaging and characterization, and advanced NMR methods.

"We have worked with companies in Japan, Germany, France, Italy, UK, Denmark, Switzerland, Netherlands, Spain, Finland, Norway, Indonesia, and Turkey. We’ve only worked with one US company so far, but our relationship with P&G goes back more than 10 years," says Anna Stenstam, the co-founder and CEO of the company.

A typical project for CR compares different formulations to assess how structure relates to product performance or stability. Changes in surface deposition behavior can be linked to structural information obtained from small angle X-ray scattering (SAXS) and to aggregation behavior characterized by NMR.

"Analysis of the effects of charge densities, incorporation of surfactants, and processing steps on active release, product stability, and film formation on a specific surface are typical requests," says Stenstam.

Most of the work done by CR is on specific characterization projects with deliverables and go/no go points, but they also provide consultancy on an Ad hoc basis with a monthly retainer or on an hourly basis.

"This is useful so a team can call us without asking a manager first, or when we are discussing a project with our clients, or reshaping the project based on new findings," says Stenstam. "We can be super agile."

According to Eric Johnson, a research fellow, with P&G Beauty, "CR has brought great insights and value to real world problems in our product development cycle. They have a great balance of being focused on the end product and also bring best in class scientific thinking and approaches."

"Our objective is to provide a scientifically sane basis for business decisions. That basis can come from structural characterization set in context," says Stenstam.

Fiona Case is a freelance writer based in San Diego California. She can be reached at Fiona@casescientific.com.
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Frank Hahn can tell you the details of a method off the top of his head. Want to know the moisture content in cotton seed? “You weight out 10 grams of cotton seeds into a dish and put it in an oven for 3 hours at 130 degrees C,” says Hahn. “At the end of three hours, you take it out and re-weigh it. Your loss is what is evaporated out. That is your moisture.”

Hahn owns and operates Hahn Laboratories in Columbia, South Carolina. The company has served as an analytical support laboratory for seed oil manufacturers for over 70 years. Hahn’s father, Col. Edward R. Hahn established the company in 1947. They are part of a cluster of independent laboratories whose oil chemists established the American Oil Chemists’ Society (AOCS). Such laboratories are still dedicated Society members and provide an important service in the industry.

Michael Hawkins started working in his father’s lab in 1983, but the legacy of Barrow-Agee, in Memphis, Tennessee, stretches back a hundred years. Hawkins says he has seen a lot of changes over the years—new technology has made some analysis methods faster and safer—but the AOCS Laboratory Proficiency Program (LPP) has been a constant. “The benefit to us is showing your customers that you are doing good work,” says Hawkins. “It is a valuable tool for the lab and a necessary one too. We also use it for quality control.”

AOCS has been central to the lives of Hahn and Hawkins for decades. Though their fathers have passed, the two men continue serving the analytical needs of companies in the fats and oils business guided by the standards established by AOCS methods. They are proud to be a part of the LPP to provide validate product composition data to their customers.

**SIMPLE BEGINNINGS**

Like AOCS itself, Hahn Laboratories can credit its start to cotton. Edward Hahn attended Georgia Tech University in Atlanta, Georgia, in the 1930s. As a chemical engineering major, he had the opportunity to co-op with a local business to put himself through school, working one semester then taking classes the next.

After the American Civil War ended in the late 1800s, the city of Atlanta rebounded from the destruction it suffered by focusing on manufacturing. Cotton was the primary crop in the region. Large cotton textile plants in the city originally viewed cotton seeds as waste, but eventually realized seeds could be monetized. Oil in the seeds was purified and sold as cooking oil, and remaining seed cake was processed into agricultural feed and fertilizer.
The lab was a competitor to Barrow-Agee, one of the first cotton seed analysis labs to open as the seed oil industry grew out of the South and spread into mid-western states. Barrow-Agee was established in 1917, by two of the founding fathers of AOCS, E.R. Barrow and G. Worthen Agee. Hawkins worked for Woodson-Tenet until 1972, when he and a partner opened their own lab. In 1975, the two purchased Barrow-Agee and it has been in the Hawkins family ever since.

Michael Hawkins started working at his father’s company in 1983. “I was just following in my father footsteps,” says Hawkins. “He told me to go to college and get a business degree, so that is what I did.” Hawkins says there was so much work at the laboratory that he quit school a year shy of his degree. “I have been a junior [at the University of Memphis] since 1984,” he says with a laugh.

LABS IN THE NEW MILLENNIA
Both Hawkins and Hahn say it has been fascinating to watch their families’ businesses change over the years. They have both seen steady growth through the decades. “In 1983, if we got a hundred samples, we thought we were busy,” he says. “Now it is not uncommon for us to get in excess of a thousand samples a day.” Hahn adds that his business serves a wider audience today than it did when he started. “Instead of just coming from the 48 states, our customers are much more international,” he says.

Technology has obviously had a significant impact on the analysis conducted in their laboratories. “Starting in the 80s and doing this for 36 years means seeing a lot of things change,” says Hawkins. He says the technical advancements that have led to method improvements have been particularly welcome. One method he and his father were happy to see updated was the Kjeldahl method for analyzing the protein content in meal.

“Kjeldahl was originally a digestion in sulfuric acid with a couple of other chemicals,” says Hawkins. “It was a nasty test. Very technique oriented. You had to be really good to perform that test accurately.” The advent of the gas chromatograph provided an opportunity for the development of a new method which the Hawkins gladly helped establish. “The combustion method that we use now really impacted our ability to push samples through the lab, because the Kjeldahl method was so labor intensive and slow,” he says. “It became a much safer test to run.”

Both men say that the LPP has been a valuable measure for how their scientists and the equipment they use are performing. Despite routine calibration of their instruments, they have both had the rare experience of the LPP identifying a quality control problem and providing an opportunity for a correction.

More often, the program provides a sense of pride. “It is valuable in your sales efforts, but it is also valuable in your quality program,” says Hawkins. “The LPP series is valuable to our business,” says Hahn. “I don’t think we would be able to exist without being able to prove we can do what we say we can.” Both men look forward to getting their results back from the series and finding that their data coincide with the results.
of all the other labs that participated. Hahn says he even keeps the samples to rerun the series months later as an internal check.

THE FUTURE OF THE LPP

Cotton seed is not used for human consumption as much as it was in the past. Hahn Laboratories now mostly analyzes cotton seed that will be added to dairy feed. Hahn says his company also stays busy with a range of tests on the quality of other oils, as well as, fats, grease, and meal. He continues to run the company as he has for the past 30 years. His role is not likely to change any time soon. All three of Hahn’s sons have worked in the lab, but have since gone on to other careers. Hahn is happy with the responsibility of maintaining his father’s legacy. He once asked his father why he worked so hard. “He said, ’Frank, I am their chemist. They don’t have a chemist. So, I am going to make sure that what I do is correct,’” says Hahn.

In 2018, Hawkins sold Barrow-Agee. The new owners adjusted the company’s leadership and hired a new CEO. They expanded their analytical capabilities with new instrumentation and with experts in the areas of metal contaminants and biofuels. Hawkins has stayed on to work in grain analysis, one of his primary areas of focus for nearly 40 years.

AOCS continues to adjust and improve methods to keep pace with demands in the fats and oils industries. In 2019, the LPP will add a series on pulses analysis after an introductory partial-year test. The series currently contains five analytical methods, with more being added as the series is expanded to a full year. The organization is also working to adopt ISO (International Organization for Standards) methods for this new pulses series. In addition, AOCS is collecting methods for future LPP series, such as the latest planned for release in 2020.

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

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AOCS Official Method Ba 4f-00, Reapproved 2017, Combustion Method for Crude Protein in Soybean Meal

AOCS Official Method Aa 3-38, Reapproved 2017, Moisture and Volatile Matter in Cotton Seeds
Soft materials which respond to external stimuli, such as foam and emulsion systems, are on the leading edge of materials research. Their macroscopic responsivity relies on the ability of multiphased systems to react at microscopic or mesoscopic scales. Stimuli-responsive surfactants that can change their structure in response to a trigger such as pH, temperature, or light are particularly attractive due to their versatile applications in various fields, such as pharmaceutics, biomedicine, and nanomanufacturing. A change in the molecular design of a surfactant activated by stimuli can affect its self-assembled structure in water and interfacial activity, which can in turn control macroscopic properties such as emulsion and foam stability.

As the surfactant industry strives for sustainable development, it is searching for environmentally safer surfactants from renewable sources to replace petrochemical-based ones. Fatty acids, which are anionic surfactants, originate from renewable resources that are available in large amounts in nature. They are also biocompatible. [3]. Fatty acid molecules have an aliphatic tail and a polar headgroup. As a function of medium conditions, the alkyl tail can be in its crystalline or liquid state, and the headgroup can either be protonated (-COOH) or deprotonated (-COO-). Due to these intrinsic properties, fatty acid molecules behave as responsive surfactants under the action of pH, salt, and temperature. Changes occurring at the molecular level under stimuli lead to changes in the self-assembled structure of fatty acids in aqueous solution at the mesoscopic scale, which controls physical properties at the macroscopic scale.

This article highlights how fatty acids as responsive surfactants can be used to design smart soft materials. It also illustrates how the self-assembly of fatty acid soap can be used to produce multi-stimuli responsive foams [1,3–6]. Responsive foams have changeable states that can be reversibly tuned between ultrahigh stability and immediate
destabilization under stimuli [1]. Other technologically important materials are made by assembling colloidal particles into structures that can be simple chains or filaments. A variety of techniques are available to assemble particles into chains, but so far it has proven challenging to make flexible permanent chains. We developed a new method for assembling flexible particle chains based on capillary attractions between particles coated with liquid fatty acids. This method is broadly similar to the way sandcastles are bound by small volumes of liquid. Here we explain how the fatty acid capillary bridges between colloidal particles can be used to provide new opportunities for assembling particles in the form of responsive filaments, and self-repairing networks [1–7].

**MULTI-RESPONSIVE AQUEOUS FOAMS BASED ON FATTY ACID SOAPS**

An aqueous foam is a dispersion of gas bubbles in a fluid aqueous phase. A foam is a thermodynamically unstable system, and any foam vanishes with time. One of the prerequisites to obtain a foam is to use a stabilizer which can adsorb at the gas/liquid interface. Fatty acids salts are surfactants known to easily produce foams [3]. Such foam stabilizers have to produce an irreversibly absorbed elastic layer at that interface, preventing film breaking between bubbles (coalescence) and gas diffusion (coarsening). Moreover, the stabilizer has to limit the drainage due to the gravity in the foam liquid channels. Stable foams will be the ones for which these three concurrent destabilization mechanisms (drainage, coalescence, and coarsening) are drastically slowed down. Responsive foams are foams for which the stability can be switched from low to high stability under an external stimulus [3].

To design responsive foams, we used specific fatty acid soaps based on the 12-hydroxystearic acid (12-HSA) [1]. 12-HSA is a widely available molecule that is synthesized from the hydrogenation of a sustainable material, i.e., ricinoleic acid, and is available in large amounts at low cost. The 12-HSA is a long-chain fatty acid which crystallizes in water, limiting its use as foam stabilizer. To disperse the 12-HSA in water, we used various soluble organic counter-ions, such as amino acids or alkanolamines [6]. Under such conditions, the 12-HSA molecules self-assembled into multilayer micron-size tubes of about 10 micrometers in length and 600 nm in diameter (Fig. 1). Foams produced from the 12-HSA tubes were stable for several months and termed ultrastable. This high stability originates from the presence of 12-HSA tubes both jammed in the foam liquid channels between the air bubbles and adsorbed at the air/water interface, which prevents foam destabilization by reducing the three main destabilization mechanisms: drainage, coalescence, and coarsening.

Upon heating, the 12-HSA tubes transformed into nano-sized spherical micelles in water due to the alkyl chain melting process triggered by temperature. This led to a change in the packing parameter. The transition from tubes to micelles is reversible and happens at a precise temperature depending on both the type and concentration of the counter-ion used to disperse the 12-HSA. Thus, the temperature transition between tubes and micelles can be easily and precisely tuned between 20°C and 75°C. The disappearance of the 12-HSA tubes into micelles inside the liquid films by heating the foam above the characteristic tube-to-micelle structural transition temperature led to a rapid and complete destruction of the foam. The foam was much less stable in the absence of tubes, since fatty acid micelles are known to make films with weak stability (Fig. 1). Remarkably, on decreasing the foam temperature below the transition temperature between tubes and micelles, tubes were re-formed inside the foam liquid channels between the air bubbles. The destabilization was completely stopped, and the foam became ultra-stable again. This transition was almost instantaneous and completely reversible. The origin of the foam responsiveness at a macroscopic scale is directly linked to the modification of self-assembled structures at the mesoscopic scale triggered by temperature, which is in turn controlled by the alkyl chain state at the molecular level [6].

The responsiveness of the foam is not limited to thermally induced stabilization-destabilization, and can be programmed by chemical design and composition of the liquid forming the foam. Recently, we obtained responsive foams from 12-HSA mixed with colloidal particles that enable destabilizing foams when light is used as an external stimulus. The foam stability

**FIG. 1. Illustration of the ability of 12-HSA tubes to produce foam, which can remain stable for several months but be destabilized in few minutes by increasing the temperature above the characteristic transition temperature. This leads to the formation of spherical micelles inside the foam. The transition between stable and unstable foam is completely reversible.**
is controlled in a contactless way [4]. Indeed, it is known that as a control stimulus, light offers significant advantages over temperature. Light can be focused at a precise location without direct contact with the foam [5]. We found a simple and efficient way to make the 12-HSA tubes photosensitive: by combining them with carbon black particles (CPB) which convert light stimulus to heat by a photothermal effect [4]. Both CPB and 12-HSA tubes are trapped in the foam liquid films and lamellae, leading to ultrastable foams. Under UV or solar illumination, CPB particles absorb light, causing an increase in temperature inside the foam liquid channels above the temperature transition from tubes to micelles. This results in very rapid foam destabilization (Fig. 2). The foam can be re-formed and destabilized for multiple cycles without changing the composition of the system. To obtain foams responding to multiple stimuli, we extended the previously described approach by replacing CBP with carbonyl iron (CI) particles, which are magnetic. Magnetic particles are known to also be intense absorbers of light irradiation. The use of CI particles in place of CBP still resulted in the destabilization of the foam system by light. However, we were also able to easily destabilize these ultrastable foams at room temperature by the application of an external magnetic field (Fig. 2). Therefore, by mixing magnetic particles with 12-HSA tubes, we easily obtained foams that exhibited stability and could be tuned by three stimuli: temperature, light, and magnetic fields (Fig. 2) [4].

Another approach to designing photoresponsive foams based on 12-HSA is to use the well-known response of fatty acids to pH changes [3]. The pH of the aqueous medium makes it possible to tune the degree of ionization of the polar headgroup between the protonated (-COOH) and deprotonated state (-COO-). This, in-turn, alters the effective area of the fatty acid headgroup due to hydrogen bonding and corresponding molecular packing parameter.

Our idea was to combine fatty acid self-assemblies with a photoacid generator molecule (PAG) in solution [5]. PAGs are commercially available, relatively inexpensive compounds, which get photolyzed by UV light. The photolysis of the PAG in aqueous solution results in the generation of an acid and causes a decrease in the pH. We mixed 12-HSA with an excess of counter-ion salt in water. In these conditions, all the 12-HSA molecules were in a deprotonated state, leading to a high headgroup area. This results in a low packing parameter, and formation of spherical micelles. The PAGs were added to the 12-HSA micelles solution. After applying UV irradiation, the PAGs get photolyzed and generate acid, inducing a progressive pH decrease. The change of pH occurring during UV irradiation leads to the protonation of some deprotonated 12-HSA molecules, producing carboxylic group (-COOH). When the two molecular forms (protonated and deprotonated) coexist, hydrogen bonding formation reduces the headgroup area. This leads to higher packing parameter values and, in turn, to a change from self-assembly to the previously described micron-size multilamellar tubes. By applying UV irradiation, 12-HSA tubes are recovered in water. Therefore, when the initially unstable foam with small micelles is subjected to UV irradiation, tubes are formed inside the foam and become stable. These foams are photo-stabilized due to the pH effect on the head group of the 12-HSA molecules unlike the previous foams described which were photo-destabilized due to alkyl chain melting phenomena.

Aqueous foams are widely used in industry. Specific industrial processes, such as washing and material recovery, can benefit both the formation of a stable foam as well as controlled foam destabilization [8]. Previous approaches to rapid destruction of foam on demand typically require the addition of environmentally hazardous chemical de-foaming agents. In view of their numerous applications, the alternative approach of designing responsive foams with simple components such as fatty acid soaps was an important discovery [8]. It opened new scientific research areas both of fundamental interest and with potential for commercial applications. The field has grown rapidly following this work, as other fatty acid-aggregated structures have been shown to be efficient in producing similar pH and thermo-responsive foams [9–10].

RESPONSIVE NETWORK AND FILAMENTS

We also used fatty acid soaps to design other types of soft, responsive, and reconfigurable materials based on particles, including chains, filaments and flexible networks [2, 7, 11]. The best known example of reconfigurable materials based on particles and multiphasic liquid is the sandcastles, which are made...
of sand and water. The water wets the surface of the grains and forms capillary bridges between them, holding the sand particles together into a complex shape (Fig. 3).

Our approach to the assembly of colloidal particles was to use capillary bridges between particles as a mean of binding them together in aqueous solution. We mixed the 12-HSA fatty acid described previously with iron oxide magnetic nanoparticles in water [2]. The fatty acid salts condensed onto the nanoparticles surfaces forming a fluid shell (Fig. 4). We then used an external magnetic field to overcome the weak electrostatic repulsion between the fatty acid coated particles, and to enable the assembly process. Upon application of a magnetic field, these coated particles assembled into permanent filaments along the direction of the applied field (Fig. 4). When the particles were attracted by the magnetic dipolar force, the nanoparticles formed a capillary bridge. After switching off the magnetic field, the assembled filaments retained their shape due to the capillary bridges between the particles [2].

The liquid bridges between the particles allow for frictionless particle-to-particle rollover, giving rise to a very high flexibility of the filaments (Fig. 5). For example, when a filament is driven by a rotating magnetic field, it follows the direction of the applied field and forms curled structures with multiple loops (Fig. 5a).

The liquid fatty acid shells surrounding the particles are also a source of high “stickiness” between particles and filaments. This stickiness can be used to assemble more complex hierarchical structures by using an external magnetic field [7–11]. When the particles enter into contact with other particles or filaments, they “stick” to each other due to the fatty acid shell. This makes it possible to make various responsive complex soft architectures. For example, microrobots made of these particles can be spatially driven by a magnetic field and, on contact, collect all the filaments and particles surrounding them (Fig. 5b). In addition, the liquid shell and bridges can be broken and re-formed on demand. We demonstrated the potential impact of this ability of capillary bridge filaments on future material design by magnetically “healing” a ruptured network. Here, we assembled a network of capillary bridged

![Figure 3. Illustration of the formation of capillary bridges between sand particles when they are wetted by water, leading to the formation of “sandcastle” structures.](image)

![Figure 4. Illustration of the formation of soft liquid-bound nanoparticle structures and filaments. Fatty acid-capped magnetic particles are formed in the first step after dispersing magnetic particles into a fatty acid soaps aqueous solution. Then, in the second step, upon application of a magnetic field, the particles align along the direction of the applied field, enter into contact, and form capillary bridges between them.](image)
magnetic filaments in aqueous solvent, and then precisely ruptured the network at a desired location using a stylus as shown in Fig. 6. An application of an external magnetic field normal to the primary axis of network damage, helped re-connect the network. This reconnection was driven by the magnetic dipole-dipole attraction between the nanoparticles, forming filament networks at the two ends of the ruptured site (Fig. 6, page 22) [2]. We found that the reconnected filaments remained in their “healed” state after the external magnetic field was turned off. This can be attributed to the formation of new capillary bridges between the damaged ends of the filaments, which allowed the connected state of the filament to be regained, thus repairing the ruptured network.

The fatty acids retained their stimuli-responsiveness while forming the capillary bridges between the magnetic nanoparticles. We used this stimuli-responsiveness to direct the on-demand disintegration of the network structures upon inducing liquid-to-solid phase transition of the fatty acid in the capillary bridges [2–11]. The temperature-driven filament disintegration of pre-assembled filaments is shown in Figure 7, page 23. Here, the dispersion temperature corresponds to the characteristic temperature of the phase transition of the fatty acid alkyl chain between the fluid and solid state. Above the phase transition, all the fatty acids have their alkyl chain under liquid state, forming a fluid shell surrounding the particles and liquid capillary bridges. However, when the alkyl chain solidified upon cooling, the bridges were not in liquid state, became brittle, and were easily broken. Thus, we can easily break and re-form on demand the capillary bridges by modifying the temperature above or below the phase transition of the corresponding fatty acid soaps. The filaments are thermoresponsive materials, where the characteristic formation/disintegration temperature of the network can be programmed in the temperature range of 10°C–80°C by tuning the nature of the fatty acid soaps (salts, alkyl chain length, side groups, and so on.) [11].

To summarize these new types of multiphasic structures, we demonstrated a simple way to assemble particles based on the use of fatty acids. The fluid fatty acid bridging provided an unusual means of particle-to-particle linkage, imparting ultrahigh flexibility and self-healing properties to the assembled structures, as well as thermo-responsiveness. The use of fatty acids to bind various types of particles in bulk could be applied in a wide range of applications such as 3D printing, food,
and cosmetic products [12–13]. A similar approach has recently been used to design a new type of toothpaste [14].

As we have just shown, our scientific approach to the design of responsive soft-materials is to find ways to use natural molecules as key components [3]. Fatty acids are the fundamental building blocks of all lipids in living matter, natural molecules that are directly available from renewable resources in large quantities. Since lipids are the main components of the skin and cellular membranes, they are also biocompatible. Large quantities of fatty acids can be extracted from agricultural and food by-products and transformed into value-added new materials [14]. Due to their intrinsic responsivity to pH, salt, and temperature, we used them to design various responsive soft materials [3]. Moreover, fatty acids can serve as solvothermic surfactants: In their protonated form (-COOH) they behave like oil (solvent), but under their deprotonated form (-COO-), called soap, they behave like a surfactant. Using innovative chemical engineering approaches to harness these characteristics has opened new pathways for making multifunctional materials, including responsive foams, magnetically manipulated structures, and self-repairing gels.

Anne-Laure Fameau works for L’Oréal company in the Physical-Chemistry Department. She received her Ph.D. in Physical Chemistry from the University of Nantes in France, in 2011. At the end of 2011, she obtained a permanent staff position at the French National Institute of Agricultural Research (INRA). In 2013, she spent 6 months as a visiting scientist in the group of Prof. Velev at North Carolina State University in the USA. In 2014, she was awarded an Agreenskills Fellowship from the European Union to spend 6 months as a visiting scientist in the group of Prof. von Klitzing at Technische Universität Berlin in Germany. In 2015, she joined L’Oréal company. In 2018, she received the European Young Lipid Scientist Award. Her past research interests were in...

References

the field of responsive soft materials based on lipids with a particular emphasis on foams and interfaces. Her research was focused on the development of these systems and their detailed structural characterization using scattering techniques such as Small Angle Neutron and X-ray Scattering (SAXS and SANS). She can be reached at anne-laure.fameau@rd.loreal.com.

Orlin Velev is a Frank and Doris Culberson Distinguished Professor at NC State University. He received a Ph.D. degree from the University of Sofia, Bulgaria (1996), while also working for one year in Japan. Velev accepted a postdoctoral position and later became research assistant professor at the Department of Chemical Engineering, University of Delaware. In 2001, he formed his new research group in the Department of Chemical and Biomolecular Engineering, NC State University, where he was promoted to chaired professor in 2009. Velev has contributed more than 200 publications and has presented more than 250 invited presentations. His awards include, among many others, NSF Career, AIChe Andreas Acrivos Award for Professional Progress, and ACS Langmuir Lecturer. Velev has been elected to an ACS Fellow and MRS Fellow. He has established a record of innovative research in the areas of colloids and nanostructures with electrical and photonic functionality, biosensors, microfluidics, and nanomanufacturing. Velev has pioneered novel nanoparticle materials, Janus and special shape particles, and responsive active materials and microstructures. Prof. Velev can be reached at odvelev@ncsu.edu.

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**SOCIETY AWARDS**

**AOCS AWARD OF MERIT**

Recognizes an AOCS Member who has displayed leadership in administrative activities, meritorious service on AOCS committees, or performed an outstanding activity or service.

Michael J. Boyer is the President of AWT Management Services, which focuses on business strategy and environmental management issues. He earned a BS and MS in civil engineering with a minor in chemistry from the University of Missouri – Columbia. He has 40 years of experience in agribusiness water, wastewater, and by-products management. Mr. Boyer has worked at 200 oilseed, vegetable oil, biodiesel, corn processing, and related facilities and developed sustainability plans for numerous private sector companies in these businesses. In addition, he has authored 50+ papers and presentations in related areas through AOCS and other public forums. Mr. Boyer’s current interests and research include managing environmental and sustainability issues for the agribusiness and related industries.

**Arnis Kuksis** is a professor emeritus at the University of Toronto in Canada. He was born in Latvia in 1927, and came to the United States in 1949 from Baltic University, Hamburg, Germany, on an LWF scholarship. Dr. Kuksis obtained his BSc in 1951 and MSc in 1953, both in agronomy in baking systems. He moved to Bayer in 1989, moving from bench chemist to group leader to head a research group working on natural antimicrobials for food. Unfortunately, that work was cancelled after two years due to business economics, and Dr. Collison took over an analytical group supporting the citric acid division. He moved to Archer Daniels Midland to take over the research analytical group in 1997, when Bayer was selling the citric business.

Dr. Collison’s first involvement with AOCS was at the Cincinnati meeting in 2004. He became involved with the Analytical Division, helping with the completion and validation of the trans-fat method, Ce 1h-05. He became a Uniform Methods Committee (UMC) member a year later. He has attended all the ISO TC34/SC2 and SC11 meetings beginning with the Seattle meeting in 2008 and became head of the US delegation to the ISO committees in 2012. In 2011, he took over the UMC chair and held that role through 2015. In 2016, he became editor-in-chief of the Official Methods and Recommended Practices of the AOCS, working to get the 7th Edition revisions published in time for the 2017 AOCS Annual Meeting in Orlando. He continues in that position today.

**Mark W. Collison** received his PhD from Iowa State University in biochemistry, where his research was on heart metabolism related to ischemia. He took a job with Pharmacia in tech support for their biotechnology division, working on protein preparation and analysis. In 1987, he moved to the research division of Nabisco and worked on enzyme technology in baking systems. He moved to Bayer in 1989, moving from bench chemist to group leader to head a research group working on natural antimicrobials for food. Unfortunately, that work was cancelled after two years due to business economics, and Dr. Collison took over an analytical group supporting the citric acid division. He moved to Archer Daniels Midland to take over the research analytical group in 1997, when Bayer was selling the citric business.

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omy, from Iowa State College (now University), Ames, Iowa, and a PhD in biochemistry from Queen's University, Kingston, Ontario, Canada. After a postdoctoral fellowship (1956–1958) in organic chemistry at the Royal Military College, Kingston, Ontario, he returned to Queen's to join the research group of Professor J.M.R. Beveridge as a research associate in lipid biochemistry. In 1960, Dr. Kuksis was awarded a Medical Research Council (MRC) of Canada Associateship (a career investigator-ship) and was appointed assistant professor of biochemistry at Queen’s. In 1965, he transferred his laboratory to the Banting and Best Department of Medical Research (BBDMR), University of Toronto. Dr. Kuksis advanced to associate and full professor (1965–1974) with parallel cross-appointments in the Department of Biochemistry at the University of Toronto. From 1972 to 1997, he served as director of the MRC Regional Mass Spectrometry Facility at the BBDMR. He was elected professor emeritus in 1997, and is best known for original discoveries in high-temperature gas chromatography of natural triacylglycerols and for the application of a new methodology for profiling neutral glycerolipids and glycerophospholipids in health and disease.

**AOCS FELLOW AWARDS**

**Recognizes achievements in science or extraordinary service to the Society.**

**Richard D. Ashby** is a senior research scientist in the Biobased and Other Animal Coproducts Research Unit for the United States Department of Agriculture, Agricultural Research Service (ARS), at the Eastern Regional Research Center in Wyndmoor, Pennsylvania. Dr. Ashby is author or co-author of over 250 publications and presentations about bio-based production, characterization, property manipulation, and application of microbial polymers and surfactants. He is a recognized expert in the use of various fermentation techniques for the synthesis of glycolipids and bacterial polyesters (poly-hydroxyalkanoates). After obtaining his PhD from Louisiana State University in 1994, he worked as a postdoctoral fellow in the Department of Chemistry at the University of Massachusetts-Lowell under Richard A. Gross studying molecular weight control of PHA biopolymers using poly(ethylene) glycol. In 1996, he joined ARS where his focus was on the use of animal fats and vegetable oils as renewable feedstocks for PHA production.

As the biodiesel industry gained traction, his research focus shifted to the use of crude glycerol and other cheap renewable materials (lignocellulosics) as feedstocks for microbial growth and product synthesis. His work with crude glycerol resulted in his research group being awarded the 2008 ACI/NBB Glycerine Innovation Award bestowed by AOCS. Currently, he is continuing his research focus on the use of fermentation to economically produce unique microbial products designed for widespread industrial application.

**Wm. Craig Byrdwell** is a research chemist at the Food Composition and Methods Development Laboratory in the Beltsville Human Nutrition Research Center, which is a part of the United States Department of Agriculture (USDA), Agricultural Research Service (ARS). Dr. Byrdwell received his undergraduate and graduate degrees from the University of Louisville, writing his dissertation on identification of the “Unknown Phospholipid” in the human eye lens and quantification of fluorophores in normal and cataractous lenses. Dr. Byrdwell took a position at the USDA’s National Center for Agricultural Utilization Research to work on analysis of triacylglycerols (TAGs) using high-performance liquid chromatography (LC) with atmospheric pressure chemical ionization mass spectrometry (APCI-MS). Dr. Byrdwell then took a position at Florida Atlantic University, where he routinely employed dual mass spectrometers, in parallel, using both APCI-MS and electrospray ionization (ESI) MS. He re-joined ARS in 2005, and took on quantification of Vitamin D and TAGs.

Recently, Dr. Byrdwell has been analyzing fat-soluble vitamins and TAGs using triple- and quadruple-parallel mass spectrometry approaches, combining three or four mass spectrometers employing complementary ionization methods (APCI-MS, atmospheric pressure photoionization MS and ESI-MS) coupled to one, two or three liquid chromatographs in LCx/MSy techniques. Dr. Byrdwell has published more than 60 peer-reviewed articles and 10 book chapters, and has been editor/co-editor of three AOCS Press books. He is on the editorial advisory board of *Lipids*, is the lipidomics associate editor for The Lipid Library (www.LipidLibrary.com), and maintains numerous websites, including www.LipidAcademy.com. Dr. Byrdwell received the 2012 Herbert J. Dutton Award from the AOCS Analytical Division and presented the 2013 Society of Chemical Industry Julius Lewkowitsch Award Lecture.

**Mila P. Hojilla-Evangelista** was born and raised in the Philippines, where she received her BS (Cum laude) in food technology and MS in food science from the University of the Philippines at Los Baños. She earned her PhD in food technology from Iowa State University. She is presently a research chemist at the United
States Department of Agriculture, Agricultural Research Service, National Center for Agricultural Utilization Research, in Peoria, Illinois. Her research career has focused on value-added products from processing of soybeans, corn, and alternative oilseed crops (lesquerella, cuphea, pennycress, and camelina). Her recognitions include the 2018 Iowa State University Food Science and Human Nutrition Alumni Impact Award; 2018 College Distinguished Alumnus Award from the University of the Philippines at Los Baños; AOCs ADM Best Paper Award in Protein and Co-Products (four times); and 2007 Federal Laboratory Consortium (FLC) Award for Excellence in Technology Transfer (soybean protein-based foamed plywood adhesives).

Within AOCs, her primary professional society, she has been a long-serving associate editor for the JAOCs and elected to various leadership positions, including secretary/treasurer, vice-chair, and chair of the Protein and Co-Products Division and member-at-large of the Governing Board. She served as invited guest editor of the peer-reviewed special issue on bio-based adhesives for the Journal of Adhesion Science & Technology (2013) and the special issue on alternative proteins for JAOCs (2018). She has mentored a postdoctoral research associate and two PhD candidates and is actively involved in many NCAUR Student Outreach Programs for STEM activities for local area students and educators.

Eric J. Murphy has a BA in history and biology from Hastings College. His PhD in biochemistry is from The Ohio State University, where he studied brain lipid biochemistry with Lloyd Horrocks. He was an assistant research scientist at Texas A&M University, where he studied the role of cytosolic lipid binding proteins with Fred Schroeder. He served as a Senior National Research Council Fellow at the National Institutes of Health and focused on the effect of neurodegenerative disease on brain lipid metabolism with Stanley Rapoport. In 2000, he joined the University of North Dakota (UND) as an assistant professor. At UND he brought together kinetic modeling of lipid metabolism in vivo coupled with his expertise in cytosolic lipid binding proteins to study the role that these and other proteins have in brain lipid metabolism.

Dr. Murphy continues to work in the broad area of n-3 fatty acid metabolism to distinguish the metabolic differences between plant-derived and marine-derived n-3 dietary sources. In 1999, he received the Jordi Folch-Pi award for his work in brain lipid neurochemistry. In 2006, he became the editor-in-chief of Lipids. In 2005, he became chief strategy officer of Agragen, LLC, a plant science company focused on using Camelina sativa as a platform for biopharmaceutical and bioactive fatty acid production for use in humans and in animal feeds. In 2015, he and his sons founded Krampade, LLC, a company that produces anti-cramping formulations for use across a broad consumer base. From 2014 to 2016 he served on the North Dakota State Board of Higher Education.

Casimir C. Akoh is a distinguished research professor in the Department of Food Science & Technology at The University of Georgia. He has made extensive, innovative, creative, and significant contributions to the field of lipids, especially on the use biocatalysts for the modification of fats and oils for better functionality in foods and potential healthful outcome. His work is at the interface of food science and nutrition. He has designed various structured lipids as infant formula fat analogs and studied their applications in infant formula and synthesized trans-free structured lipids to replace hydrogenated fats and used them to make trans-free spreads, margarines, and shortenings. He edited eight books and his “Food Lipids” book, now in its 4th Edition (2017), is used worldwide as a textbook for graduate instruction.

Overall, Professor Akoh’s research has resulted in over 810 publications and presentations that include up to 278 refereed publications, 50 book chapters, four patents, 301 presentations, and more than 180 invited presentations at national and international conferences. He is an editorial board member or associate editor of several journals. He became a member of the AOCS Governing Board in 2001, served as chair of the Biotechnology Division (2001–2004), and secretary of AOCS (2004–2006). He was vice president of AOCS from 2007 to 2008 and president of AOCS from 2008 to 2009 during the Society’s 100-year anniversary. Professor Akoh
has received many prestigious international awards, including the Stephen S. Chang Awards (AOCS, 2004; IFT, 2008), Research and Development Award (IFT, 2008), and the AOCS Biotechnology Division Lifetime Achievement Award (AOCS, 2009).

**STEPHEN S. CHANG AWARD**
Recognizes a scientist, technologist, or engineer who has made decisive accomplishments in research for the improvement or development of products related to lipids. Provided by the Stephen and Lucy Chang endowed fund.

Dr. Xuebing Xu received his PhD in chemical engineering from the Technical University of Denmark and was a professor/honorary professor in Aarhus University, Denmark. Dr. Xu’s areas of research include lipid technology, food/lipid/ingredients functionality, and enzyme technology, among others. Dr. Xu has published more than 270 papers, edited three books, and is inventor of more than 33 patents.

Dr. Xu is the general manager of the Wilmar Global Research and Development Center. He is also a guest professor at a few universities. He was the founding president of the International Association of Rice Bran Oil (2013–2014) and is also the founding president of the International Sunflower Oil Association (2015–present). Dr. Xu won the European Lipid Technology Award in 2017, and became an AOCS Fellow in 2018.

Dr. Xu has been involved in a number of activities in AOCS. He is associate editor of JAOCS (2005–present), was chair of the Phospholipids Division (2016–2017), and is vice chairperson of the China Section (2016–present). Dr. Xu was the organization committee member for a few conferences, including the AOCS-CCOA Joint Symposium on Functional Lipids (2014) and 1st AOCS China Section Congress on Oilseeds and Co-Products (2017). He was invited as session chairs for several AOCS annual meetings.

**SUPELCO AOCS RESEARCH AWARD**
Recognizes outstanding original research in fats, oils, lipid chemistry, or biochemistry. Sponsored by MilliporeSigma, a subsidiary of Sigma-Aldrich Corp.

Dr. Richard W. Hartel, a professor of food engineering, has been with the Department of Food Science at the University of Wisconsin-Madison since 1986. Dr. Hartel has an active research program focused on phase transitions in foods—particularly chocolate (lipids), frozen desserts (ice), and confections (sugars and lipids)—funded from a variety of sources, including the United States Department of Agriculture, industry, and commodity boards. With over 75 graduated students since 1986, he currently has 14 grad students and three postdocs. His work has resulted in over 220 publications. He also authored or co-authored 15 books, including authoritative works on both ice cream and confectionery science as well as two popular interest books written with his daughter.

Dr. Hartel is well known for being a strong proponent of education and student learning. An active instructor, he teaches classes on food lipids, food manufacturing, food preservation, and food functionality, as well as a freshman seminar class and a candy science elective. He has been advisor of the Food Science Club for over 30 years and is academic advisor of 75 undergrad students. His interest in education also extends to adult learning. He is coordinator for several industry-oriented short courses: Nutritional Bars, the 2-week Candy School, and Enhanced Gummies and Jellies.

Dr. Hartel has been an active contributor to AOCS, including numerous presentations at annual meetings and many years of service with JAOCS. After working through the system from associate editor, he served as editor-in-chief of JAOCS for 10 years. He is currently back as an associate editor.

**AOCS YOUNG SCIENTIST RESEARCH AWARD**
Recognizes a young scientist who has made a significant and substantial research contribution in one of the areas represented by the Divisions of AOCS. Sponsored by the International Food Science Centre A/S.

Dr. Guodong Zhang is an assistant professor of food science and molecular and cellular biology at the University of Massachusetts-Amherst (2013–present). He obtained his BS in chemistry from Xi’an Jiaotong University in China (2003), MS in chemistry from National University of Singapore (2005), PhD in food science from the University of Wisconsin-Madison (2010,) and performed postdoctoral training at the University of California-Davis (2010–2013).

In 2013, Dr. Zhang was appointed as the foundation professor of biology at the Huaishang University of Science and Technology in China. He is a guest professor at a few universities.

Dr. Zhang has been an active contributor to AOCS, including numerous presentations at annual meetings and many years of service with JAOCS. After working through the system from associate editor, he served as editor-in-chief of JAOCS for 10 years. He is currently back as an associate editor.

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The 112-year-old Nisshin OilliO Group is active in mainly four business domains: oil, meal, and processed food; processed oil and fat; fine chemicals; and health science. It tirelessly strives to fulfill its corporate philosophy and core commitment by acting as a responsible member of global society and maximizing its corporate value as a creative and growing business by contributing to society, the economy, and the health and happiness of every stakeholder. To realize these goals, The Nisshin OilliO Group utilizes cutting-edge technologies to produce a wide range of dietary and lifestyle staples to fulfill its promise of “good flavor, health and beauty.”

SCHROEPFER MEDAL
Recognizes a scientist who has made significant and distinguished advances in the steroid field. Originated by colleagues of George Schroepfer.

W. David Nes is a distinguished professor of chemistry and biochemistry and former director of the Center for Chemical Biology at Texas Tech University. He has published approximately 210 peer-reviewed journal articles, one patent, and eight books. He has been among the most cited scientists in sterol research. Dr. Nes is regarded as being a tireless mentor of over 75 graduate students, postdoctoral fellows, and visiting scientists.

During his career, he has been a consultant to government and industry, a question writer for the graduate record exam in biochemistry, and a sought-after lecturer. He has taken sabbatical leaves in Germany and Wales, served as associate editor of journals, and is an AOCS Fellow and former Program Director at the National Science Foundation. Professionally, he is recognized as an international expert in the chemical biology of sterols and other isoprenoids as highlighted in the 2019 Festschrift special issue in the journal *Molecules* honoring his 65th birthday.

Dr. Nes was born in Bethesda, Maryland, while his father, William R. Nes, was an organic chemist at the National Institutes of Health researching steroids. During his early life, Dr. Nes was active in sports and recruited to Gettysburg College, becoming a varsity wrestler in his sophomore year and pledging Sigma Chi Fraternity. After graduating from Gettysburg College, he attended Drexel University and received an MS under the direction of his father, with whom he collaborated on numerous projects until father’s death in 1988. After Drexel, Dr. Nes received a PhD from the University of Maryland and then pursued postdoctoral fellowships at the University of California, Berkeley, and the United States Department of Agriculture–Albany.

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Chemistry/Nutrition Category
Stability and Bioavailability of Curcumin in Mixed Sodium Caseinate and Pea Protein Isolate Nanoemulsions (JAOC 95(8): 1013–1026).
Manispuritha Yerramilli, Natalie Longmore and Supratim Ghosh

Engineering/Technology Category
Changes in Corn Protein Content During Storage and Their Relationship with Dry Grind Ethanol Production (JAOC 95(8): 923–932).
Divya Ramchandran, Mila P. Hojilla-Evangelista, Stephen P. Moose, Kent D. Rausch, Mike E. Tumbleson and Vijay Singh

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Can solidifying personal care products save the environment?

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

Rebecca Guenard

Shopping bags and soda bottles are often the focal point of the unrecyclable plastic waste that collects in the environment, but culpable containers do not just come from food. Bathrooms are stocked with personal care items in plastic containers destined to remain in landfills and oceans for centuries. Instead of thinking about ways to recycle packaging, some in the personal care industry are focused on reformulating their products to minimize volume by eliminating water as an ingredient. These new solid products are gaining popularity among beauty consumers while raising their consciousness about water scarcity. And companies like Proctor and Gamble are taking advantage of greater consumer awareness to shift the norm of personal care products away from the liquid phase.

Currently shampoo, conditioner, bodywash, and moisturizers all flow into the consumer’s hand before they are applied. Asking the consumer to accept anything different is introducing a completely new paradigm. To reap the environmental benefit of less packaging, companies will have to formulate waterless products to be a pleasing consumer experience. Those that have already accepted the challenge are finding success, as waterless beauty is currently a trend (http://tinyurl.com/y3t3cf8b). Will the world’s plastic waste problem be lessened with newly formulated personal care products?

Brianna West was inspired to reduce personal care packaging in 2012. She founded the company Ethique in Christchurch, New Zealand, after working out the kinks of waterless formulations while still a college student (https://ethiquebeauty.com). She says her company was formed out of her passion for the environment and her knowledge of cosmetic chemistry. “It seemed so obvious to remove water, and therefore plastic, and just send customers the concentrated ingredients that make up a product so they can add the water at home,” says West.

She set out to formulate a shampoo bar (Fig. 1), a solid version of shampoo that is not uncommon but is usually made from soap. The high pH of soap leaves a residue and roughens the hair cuticle. West says she wanted to avoid these undesirable effects and make a salon-quality shampoo in solid form. “Up to 75 percent of shampoo and even 90 percent of conditioner can be water,” she says. “Obviously water is an important part of the product, but I was interested in what happened if you simply combined the ingredients without water.”

After gathering feedback on her shampoo bar, West determined the optimal formulation for maximum customer satisfaction. Delivering her first product gave her confidence to try other formulations. “I branched out into conditioners, and then started replacing everything in my bathroom with solid versions,” she says. She also sells laundry and pet products.

West says one obstacle she initially faced was shipping. Ethique is an online retailer, and she needed containers that would protect her product without negating its sustainability. “It was a bit of a nightmare to develop packaging that could stand up to international shipping and look great sitting on a retail shelf,” says West. “Thankfully I have worked with some wonderful packaging companies over the years who have put lots of time into working on this for us.”

She says her current challenge is that big chemical companies are not investing research and development resources on new solid or anhydrous materials that can be used as water-free ingredients. That limits what products her company can make. Despite this, Ethique has an extensive product line, including conditioner, moisturizer, and self-tanner. “There are things we cannot do currently, but I am always looking for new innovations and developments, so this gap is closing,” says West.
Ethique surpasses its competitors in its dedication to waterless personal care and in the range of products on the market. Most companies have added a single solid product to an existing personal care line, and those products are mostly limited to haircare. In the coming months that could change, as Procter & Gamble (P&G) has announced plans for a complete brand of liquid-free home and personal care products by the end of 2019.

The Cincinnati, Ohio-based company has set self-imposed sustainability goals that it is achieving through such initiatives as the utilization of green-energy sources and the implementation of waterless products (http://tinyurl.com/yx8trrly). One of the company’s priorities is to help maintain the world’s fresh water supplies (http://tinyurl.com/y598ty18).

According to a 2015 report by the market analysis firm Mintel, water use will outweigh water supply by 2025, elevating freshwater from a ubiquitous resource to a luxury item (http://tinyurl.com/y56vumpq). Climate change, population growth, and changing consumption patterns have put freshwater sources at higher risk that any other ecological system (http://worldwildlife.org). The Mintel report predicts that personal care brands will have to change how they formulate their products to accommodate for fewer sources of fresh water.

P&G is heeding the warning. In March 2019, they announced their new water-free haircare brand, Waterless (http://tinyurl.com/y5aeq7kv). The products are intended for use in areas like South Africa, where water supplies have dwindled. Unlike solid personal care products, the Waterless line is intended to clean and condition with no water at all. In addition, P&G will launch a line of solid personal care products are part of the D53 brand which will include facial cleanser, body wash, laundry detergent, and toilet cleaner (http://tinyurl.com/y2959ath).

According to P&G, proprietary technology allows them to design different formulations for a variety of waterless personal care products. The company says that taking water out of their formulas reduces the weight of the products by 80% and the volume by 70%. These reductions result in less packaging use and lower shipping costs, both of which lead to a more sustainable use of environmental resources.

Until the D53 brand makes it to stores, the instore options for consumers—at least in the United States—are limited. Kelly Dobos, technical manager of cosmetics at Sun Chemical in Parsippany, New Jersey, has tried the available products in her area as part of a column she writes for Cosmetics & Toiletries. She found the products similar to powdered soaps that were common in the 1980s and 90s.

“When you think about waterless cosmetics products you think of old-school formulations like soap,” says. “But just because soap is seen as old-school and not technologically forward, that doesn’t mean that it’s not good.” Dobos says personal care companies should reconsider these old formulations as a starting point to making products that reduce water consumption.

OWA Haircare in New York City began selling powdered shampoo in 2018 (https://owahaircare.com). The shampoo’s texture is reminiscent of the older powdered soap formulations. According to Dobos, these types of powdered shampoos need to go a step future if they want to win over customers. “A lot of soap formulations are just cleansing and they are not conditioning,” she says. “Effort needs to be put into adding conditioning benefits to the formulations.”

Though she didn’t name Ethique specifically, Dobos found the waterless conditioners she sampled inferior to their water-centric counterparts. She says they have a rough texture and lack the performance of traditional conditioners. However, she believes the motivation for more environmentally friendly personal care products will prompt companies to find ways to make water-free formulations more appealing to the consumer. “We will see more innovation coming,” says Dobos.

The question that remains is: Will consumers give up the sensory experience they have come to expect from their personal care products in place of a more environmentally responsible choice? West admits that application is an issue and solid products are an obstacle for some consumers. “Although people really resonate with our “why” very quickly, a lot of people do not understand how to use a solid as they do a liquid,” she says, “so there is a high barrier to a customer’s first purchase.” She says she usually hooks customers with the shampoo bar since many consumers are familiar with this type of product. After that, consumers will try the conditioning bar and then move on to more unusual solid products like moisturizing bars.

Most importantly, as Dobos points out, consumers are not going to sacrifice performance. Personal care companies trying waterless formulations will have to be aware of product quality as well as environmental sustainability. “People who do not normally make decisions based on the environmental credentials of a product will still buy them over bottled products if they love the way they make their hair feel,” says West.

Olio is produced by Inform’s associate editor, Rebecca Guenard. She can be contacted at rebecca.guenard@aocs.org.

References


USDA and FDA announce a formal agreement to regulate cell-cultured food products from cell lines of livestock and poultry

The US Department of Agriculture’s (USDA) Food Safety and Inspection Service (FSIS) and the US Department of Health and Human Services’ (HHS) Food and Drug Administration (FDA) announced on March 7, 2019, that they have reached a formal agreement to jointly oversee the production of human food products derived from the cells of livestock and poultry.

The agreement describes the oversight roles and responsibilities for both agencies and outlines how the agencies will collaborate to regulate the development and entry of these products into commerce. This shared regulatory approach will encourage innovation while ensuring that cell-cultured products derived from the cell lines of livestock and poultry are produced safely and are accurately labeled.

“Consumers trust the USDA mark of inspection to ensure safe, wholesome, and accurately labeled products,” said USDA Deputy Under Secretary for Food Safety Mindy Brashears. “We look forward to continued collaboration with FDA and our stakeholders to safely regulate these new products and ensure parity in labeling.”

“We recognize that our stakeholders want clarity on how we will move forward with a regulatory regime to ensure the safety and proper labeling of these cell-cultured human food products while continuing to encourage innovation,” said Frank Yiannas, FDA Deputy Commissioner for Food Policy and Response. “Collaboration between USDA and FDA will allow us to draw upon the unique expertise of each agency in addressing the many important technical and regulatory considerations that can arise with the development of animal cell-cultured food products for human consumption.”

Under the formal agreement, the agencies agree upon a joint regulatory framework wherein FDA oversees cell collection, cell banks, and cell growth and differentiation. A transition from FDA to FSIS oversight will occur during the cell harvest stage. FSIS will oversee the production and labeling of human food products derived from the cells of livestock and poultry.

The shared responsibility makes sense, as cell-cultured food products cross the boundaries of medical and food technology.

The FSIS, an agency within the U.S. Department of Agriculture, is the public health agency responsible for ensuring that nation’s meat, poultry, and egg products are safe, wholesome, and accurately labeled.

The FDA, an agency within the U.S. Department of Health and Human Services, protects the public health by assuring the safety, effectiveness, and security of human and veterinary drugs, vaccines and other biological products for human use, and medical devices. The agency also is responsible for the safety and security of our nation’s food supply, cosmetics, dietary supplements, products that give off electronic radiation, and for regulating tobacco products.

To view the Formal Agreement, visit the FSIS website at www.fsis.usda.gov/formalagreement or the FDA website at www.fda.gov/Food/InternationalInteragencyCoordination/DomesticInteragencyAgreements/UCM632752.htm.
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Recently, I read an article in the February 2019 edition of Analytical Scientist magazine that illustrated the state of analytical chemistry in Brazil, Chile, and Colombia [1]. I was intrigued by how the local economy shapes the development of the discipline. Below, I have adapted parts of the article into a Q&A format, and incorporated other relevant information.

Q: What is the state of analytical chemistry in Brazil? Which areas within this field are experiencing growth?

Analytical chemistry is a growing field in Brazil; over 3,000 Ph.D. students defended analytical chemistry theses during the 2016–2018 period. The most traditional applications of analytical chemistry are petrochemicals. However, there are many other applications that relate to local economies. Some examples are ethanol and other derived products, such as essential oils. Since Brazil is a country with one of the most extensive botanical reserves in the world, there is a focus on extraction, purification, and separation of active ingredients arising from natural products. In that sense, many laboratories across the country are equipped with WCOT GC columns, UHPLC, high resolution, and tandem MS systems, as well as other superficially porous technologies. Separation science is quickly growing in this region, and other techniques could arise from research in this area. In this sense, in Brazil (and in much of Latin America), research funding comes mostly from the government. Although this type of funding has many advantages,
there is room for improvement to foster industry-academia projects that lead to mutual benefit. Some of these industry-academia relationships could be forged in symposiums, which Brazil is also active in sponsoring. In 2018, the Brazilian National Symposium on Analytical Chemistry (ENQA) united over 1,200 participants, and the Latin American Symposium on Chromatography and Related Techniques (COLACRO) also had increased attendance [1]. Brazil has the “Sociedade Brasileira de Química” (Brazilian Chemical Society), which is a non-profit organization established in 1977 that publishes the Journal of the Brazilian Chemical Society [2].

Q: What type of academic research in analytical chemistry is of interest in Columbia? How are industry and academia connected?

At the CIBIMOL Research Center of Excellence, Universidad Industrial de Santander Bucaramanga, an area of focus in analytical chemistry is that of separation science. This is particularly applied to extracts of aromatic plants, starting from the field and passing through the complete essential oil value chain. This comprises the complete characterization of secondary metabolite mixtures, biological activity determination, and antioxidant capacity of components. Because of the nature of this work, high-mass spectrometric and high-chromatography resolution are techniques of most interest. However, generally speaking, equipment is insufficient in many universities in Colombia, and lack of government funding to improve research is a cause of concern. The lack of research funding is causing many chemists who return to the country after obtaining expertise abroad, to reconsider their options. Unfortunately, there are discrepancies between industry and academia. Industry has very specific concerns that need to be resolved quickly, but industry does not invest in academia to facilitate resources that would allow for the fast turnaround they require. Despite these challenges, some sectors are forming great industry-academia partnerships that are addressing such challenges as the determination of pesticide residues in coffee and other food items for export [1]. Colombia has the “Sociedad Colombiana de Ciencias Químicas” (Colombian Society of Chemical Sciences), which is a non-profit agency created in 1941 with the objective of promoting and sharing discovery related to basic and applied chemistry [3].

Q: What is the state of analytical chemistry in Argentina?

Since 1999, Argentina has the “Asociación Argentina de Químicos Analíticos (Argentinean Association of Analytical Chemists), AAQA, which unites researchers, professionals, and students that specialize in the analytical chemistry field. AAQA also provides various opportunities for continuing education in the field, as well as congresses to share expertise and develop projects. Many universities across the country have expertise in analytical chemistry [5]. However, the expertise may be integrated as an application in another field. For example, the “Instituto de Investigaciones Bioquímicas de La Plata (INIBIOLP), Universidad Nacional de La Plata, in Buenos Aires, (Institute of Biochemical Research of La Plata, National University of La Plata) is a nonprofit institution that aims to understand biochemical processes related to lipids (lipid metabolism and cancer, natural products and metabolism of lipids, lipids and proteins biophysics, and others). INIBIOLP counts with expertise in analytical chemistry, and also offers high-level analytical chemistry services to the public (fatty acid profiles by GC, identification of organic contaminants by GC and MS, protein purification by ionic exchange and/or molecular exclusion, and others). Although the analytical chemistry tools are integrated within other areas of research, they are also open for hire to those that need specialized testing [6].

**Further reading**


“Sociedad Chilena de Química” (Chilean Chemistry Society), which groups professionals in the fields of organic, inorganic, and analytical chemistry, as well as natural products, environmental, catalysis, adsorption chemistry, and education. Since 1951, it has published the trimestral “Journal of the Chilean Chemical Society” which compiles distinguished research from Chile and abroad [4].

Q: What is the state of analytical chemistry in Chile?

In Chile, analytical chemistry developed as an independent field in the 1980s, with the first Ph.D.s formed in the country in the 1990s. In that sense, it is a new field for the region and it is a popular choice for Ph.D. theses. However, government funding is challenging for researchers in Chile, since the budget for science, technology, and innovation is 0.38% of the GDP, the same as it has been since 2011 (this is approximately three times lower than in Brazil, and seven times lower than in the United States). Industry funding is also a challenge, and the government is trying to promote it by implementing policies that favor industries on short-term projects. Ideally, these policies should promote long-term partnerships that benefit both parties and have deeper scientific value [1]. Chile has the...
Leon Espinosa trained as a chemical engineer and came to the fats and oils industries through his first job with Lloreda SA, a consumer products company based in Colombia that produces and sells food and cleaning products derived from oils. There, he worked on the design, manufacture, and startup of processing plants for tallow and soybean, sunflower, palm, palm kernel, and cottonseed oils.

“For the past 10 years, I have been with Desmet Ballestra,” he explains, “where my work is more related to the search for solutions for our customers.”

Espinosa became involved in AOCS in 2009, when his friend and fellow AOCS member Roberto Berbesi invited him to participate in the 14th AOCS Latin American Congress and Exhibition held in Cartagena, Colombia, in 2011. Ten years later, Espinosa is himself now chair of the Latin American Section and is working to organize and promote the 17th AOCS Latin American Congress and Exhibition, which will be held in Foz do Iguaçu, Brazil, on October 8–11, 2019.

“Organizing a large meeting like this involves putting many puzzle pieces together,” he notes, “including the organizers, the presenters, and the logistics. Making each piece fit is like tuning a philharmonic orchestra.”

AOCS is important to the region, he suggests, because it provides access to the most recent information on topics of interest to those in the fats and oils industries. “A great advantage of a Congress like this is that it brings together a select group of people with in-depth knowledge of the subjects and allows participants to interact with people in similar businesses. Attending the Congress is important for all levels of employees within related organizations. New hires can start with the short courses and then participate in the main sessions and visit the booths to know what is available to industry.”

Espinosa echoes the sentiments of many AOCS volunteers when he says that belonging to AOCS helps members to know and relate to more people. “Expanding your network of knowledge and people helps you climb the ladder of your career,” he concludes.
Acyltransferases and uses thereof in fatty acid production
The present invention relates to the recombinant manufacture of polyunsaturated fatty acids. Specifically, it relates to acyltransferase polypeptides, polynucleotides encoding said acyltransferases as well as vectors, host cells, non-human transgenic organisms containing said polynucleotides. Moreover, the present invention contemplates methods for the manufacture of polyunsaturated fatty acids as well as oils obtained by such methods.

Polypeptides with lipase activity and polynucleotides encoding same for cleaning
Isolated polypeptides with lipase activity; polynucleotides encoding the polypeptides; nucleic acid constructs, vectors, and host cells comprising the polynucleotides; and methods of using the polypeptides for cleaning are disclosed.

Method for producing stabilized whole wheat flour
Zhao, B., et al., Intercontinental Great Brands LLC, US10212958, February 26, 2019
A stabilized flour, such as stabilized whole grain wheat flour, exhibiting unexpectedly superior extended shelf life and superior biscuit baking functionality, may be produced with or without heating to inhibit lipase by subjecting whole grains or a bran and germ fraction or component to treatment with a lipase inhibitor, such as an acid or green tea extract. Treatment with the lipase inhibitor may be performed during tempering of the whole grains or berries or during hydration of the bran and germ fraction or component.

Solid solution compositions and use in chronic inflammation
Bannister, R.M., et al., Infirst Healthcare Ltd., US10213381, February 26, 2019
The present specification discloses pharmaceutical compositions, methods of preparing such pharmaceutical compositions, and methods and uses of treating a chronic inflammation and/or an inflammatory disease in an individual using solid pharmaceutical compositions comprising 34% to 40% by weight of a hard fat that includes a triglyceride mixture; c) 22% to 28% by weight of a liquid lipid mixture that includes monoglyceride; and d) 7% to 13% by weight of a liquid glycol polymer.

Combinational liposome compositions for cancer therapy
Yang, J., et al., Mallinckrodt LLC, US10213385, February 26, 2019
The present invention provides methods for delivery of therapeutic agents to a subject using multi-component liposomal systems. The methods include administration of a therapeutic liposome containing an active agent, followed by administration of an attacking liposome that induces release of the agents from the therapeutic liposome.

Multi-step separation process
Kellihor, A., et al., BASF Pharma (Callanish) Ltd., US10214475, February 26, 2019
The present invention provides a chromatographic separation process for recovering a polyunsaturated fatty acid (PUFA) product from a feed mixture, which comprises: (a) purifying the feed mixture in a first chromatographic separation step using an eluent a mixture of water and a first organic solvent, to obtain an intermediate product; and (b) purifying the intermediate product in a second chromatographic separation step using as eluent a mixture of water and a second organic solvent, to obtain the PUFA product, wherein the second organic solvent is different from the first organic solvent and has a polarity index which differs from the polarity index of the first organic solvent by between 0.1 and 2.0, wherein the PUFA product is other than alpha-linolenic acid (ALA), gamma-linolenic acid (GLA), linoleic acid, an ALA mono- di- or triglyceride, a GLA mono- di- or triglyceride, a linoleic acid mono- di- or triglyceride, an ALA C.sub.1-C.sub.4 alkyl ester, a GLA C.sub.1-C.sub.4 alkyl ester or a linoleic acid C.sub.1-C.sub.4 alkyl ester or a mixture thereof.

Method and device for processing an organic oil in steps
A method is provided for processing an organic oil in steps, including the following: A) providing a raw oil; B) degumming the raw oil by adding water or acid to the raw oil and forming at least two phases, an aqueous phase and an oil phase, and separating the aqueous phase enriched in phospholipid from the oil phase; C) adding sodium hydrogen carbonate and/or sodium acetate to the oil phase from step B, and removing alkaline-earth compounds and/or phospholipids and/or stearyl glycosides, in solution or suspension in an aqueous phase, from the oil phase.
Congratulations and welcome to this year’s Division leadership! We look forward to working with you.

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AOCS Journals: 2018 Editor-in-Chief picks

Once a month, AOCS’s weekly news digest, Inform SmartBrief, highlights a paper from one of AOCS’ three journals. These papers are selected by that journal’s respective Editor-in-Chief, who explains the significance and relevance of that particular paper. Here, we have assembled all of the Editor-in-Chief picks from 2018 in one place. To follow the 2019 EIC picks as they are released in real time, sign up for a free subscription to Inform SmartBrief at https://bit.ly/2HlEQue.

**Journal of the American Oil Chemists’ Society**

**Extraction of surface wax from whole grain sorghum**
Hums, M.E., et al.
https://doi.org/10.1002/aocs.12088

**Protein solubilization**
Sathe, S.K., et al.
https://doi.org/10.1002/aocs.12058

**31P NMR method for phospholipid analysis in krill oil: proficiency testing—a step toward becoming an Official Method**
Zailer, E., Y B. Monakhova, B.W.K. Diehl
https://doi.org/10.1002/aocs.12153

**Human liver fatty acid binding protein-1 T94A variant, nonalcohol fatty liver disease, and hepatic endocannabinoid system**
https://doi.org/10.1002/lipd.12008

**Plasma unesterified fatty-acid profile is dramatically and acutely changed under ischemic stroke in the mouse model**
Golovko, S.A. and J.Y. Golovko
https://doi.org/10.1002/lipd.12073

**Berberine inhibits oxygen consumption rate independent of alteration in cardiolipin levels in H9c2 cells**
Chang, W., et al.
https://doi.org/10.1007/s11745-017-4300-z

**Recent advances in the synthesis of sulfonate Gemini surfactants**
Zhou, M., et al.
https://doi.org/10.1002/jsde.12046

**Advantage of sodium polyoxyethylene lauryl ether carboxylate as a mild cleansing agent. Part 2: effects on skin functions and conditions**
Endo, K., et al.
https://doi.org/10.1002/jsde.12177

**Interactions between lipases and amphiphiles at interfaces**
Reis, P., et al.
https://doi.org/10.1002/jsde.12254
The 7th Edition was revised by academic, corporate, and government experts to ensure the most technically accurate methods are presented. Reviewers harmonized the methods with other leading scientific organizations, including AOAC International, AACC International, FOSFA International, IOC, and ISO. Procedures were updated to include new apparatus, equipment, and supplier information, including current locations, mergers, and business closures. The 7th Edition includes all additions and revisions of the 6th Edition.

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Coalescence of small bubbles with surfactants

Bubble coalescence is central to many important technological processes, such as separations, cleaning of oil spills, microfluidics, emulsification, and foaming. It is well known that surfactants, which are frequently present as additives or contaminants, delay coalescence by slowing the drainage of the liquid film separating the approaching bubbles before they make contact. However, the coalescence and surfactant transport mechanisms developed after surfactant-laden bubbles make initial contact remain poorly understood. Here, we characterize these mechanisms using high-fidelity numerical simulations to predict the evolution of bubble interfaces, surfactant spreading, and induced Marangoni flows. Our results show that the surfactant initially accumulates on the tiny meniscus bridge formed between the coalescing bubbles due to the rapid and highly localized contraction of meniscus area. At the same time, a Marangoni-driven convective flow is generated at the interface, which drags the accumulated surfactant away from the joining meniscus and toward the back of the bubbles. Together, these transport mechanisms affect the rate bubble coalescence by dynamically modifying the local pull of surface tension on the bubble interfaces.

High-resolution visualization of cosmetic-active compounds in hair using nanoscale secondary ion mass spectrometry

A wide range of small molecules are used in our daily hair products to improve the appearance of hair and to protect it from damage from the environment. To better design formulations of these products, an understanding of the partitioning and distributions of these small molecules in hair is critical. In this study, we used preferential extraction methods to measure the partitioning of active compounds commonly found in hair cosmetic products on the hair surface and inside hair, and investigated the use of stable isotope labelling, cryosample preparation, and nanoscale secondary ion mass spectrometry (NanoSIMS) for high-resolution visualization of distributions of these compounds. With this approach, we quantified partitioning and directly visualized distributions at high-resolution of four molecules (e.g., resorcinol, salicylic acid, pentadecyl alcohol, and pentadecanoic acid) in hair. This has not been achieved before and revealed distributions of high lipophilicity active compounds in the lipid-rich and hydrophobic cell membrane complex network in hair, while low lipophilicity ones distributed dispersedly.

Wettability manipulation of overflow behavior via vesicle surfactant for water-proof surface cleaning

Efficient superhydrophobic surface cleaning strategies are of paramount importance for water-proof wearable electronics applications. Herein, a dynamic, time-dependent cleaning approach on a superhydrophobic surface is presented, which uses vesicle surfactants (Aerosol OT) to manipulate the surface wettability transition from non-wetting to ultra-wetting within milliseconds and fluid overflow behavior. Contaminants such as dust and grease on water-proof surfaces can be easily removed under the ultra-wetting state. In addition, a water wash can remove the surfactant in milliseconds to achieve a low surfactant retention and recover the superhydrophobicity of wearable devices. This washing strategy achieves stable wettability transitions for more than 20 cycles and does not damage the electronic cloth. This cleaning strategy can inspire the next explosion in the development and application of advanced materials.

Quantifying the effect of solution formulation on the removal of soft solid food deposits from stainless steel substrates

The role of detergent formulation on the cleaning of a complex carbohydrate-fat food soil from stainless steel surfaces was studied using a modified version of the millimanipulation device described by Ali et al. (2015b) which allowed the force required to scrape the soil from the surface to be measured as the soil is immersed, in situ and in real time. This allowed the influence of temperature, solution chemistry, and time on the mechanical forces (theology) and removal behavior of the soil to be studied—in effect quantifying the relationships in Sinner’s cleaning circle. The soil simulated a burnt-on baked-on deposit and featured regular cracking in the 300 gm thick layer. The removal force decreased noticeably on hydration: the cleaning mechanism was then determined by the agents present. At 20°C, below the temperature at which the fat phase was mobile, removal was characterized by cohesive failure except in the presence of the cationic surfactant CTAB, which promoted adhesive failure and fast decay in removal force. At 50°C, when the
fat was mobile, a transition between cohesive and adhesive failure was observed at pH 7 which was inhibited at higher pH. Adhesive failure and fast decay in removal force was observed at higher pH and 50°C in the presence of the anionic and non-ionic surfactants, SDBS and TX-100, respectively.

From well-entangled to partially entangled wormlike micelles


We combine mechanical rheometry, DWS, and SANS with a simulation model, the "pointer algorithm," to obtain characteristic lengths and time constants for WLM solutions over a range of salt concentrations encompassing the transition from unentangled to entangled solutions. The solutions contain sodium lauryl ethylene glycol sulfate (SLE15), cocamidopropyl betaine (CAPB), and NaCl. The pointer algorithm is extended to include relaxation of unentangled micelles, allowing micelle parameters to be extracted from the rheology of partially entangled solutions. DWS provides the data at high frequency needed to determine micelle persistence length accurately. From pointer algorithm fits to rheology, we observe a salt-induced rapid change in micellar length as the solution enters the well-entangled regime and a weaker growth with surfactant concentration consistent with mean-field theory. At a lower surfactant concentration, micelle length and persistence length from SANS are roughly consistent with values from rheology once the lower surfactant concentration used in SANS is accounted for. This is, to our knowledge, the first time that quantitative comparisons of structural features including micelle length are made between rheology and SANS. Finally, scaling laws for micelle diffusion and recombination times indicate that micelle kinetics are reaction controlled leading to mean-field recombination with surrounding micelles over the entire range of concentration of interest except at very low and very high surfactant concentrations where either short micelles or branched micelle clusters are dominant.

Triacylglycerol composition of breast milk during different lactation stages


Triacylglycerol (TAG) composition of breast milk plays an important role in improving digestion, absorption, and metabolism when consumed by infants. This study characterized the TAG profile of human colostrum, transitional, and mature milk samples from 103 women. Significant differences in the TAGs composition of breast milk fat from three lactation stages were observed. The TAGs with high molecular weight and unsaturated fatty acid (such as 1,3-olein-2-palmitin (OPO) and 1(3)-olein-2-palmitin-3(1)-linolein (OPL)) were enriched in colostrum, while the TAGs containing medium-chain fatty acids were more abundant in transitional and mature milk than that in colostrum. Of note, OPL was the most common TAG in breast milk of Chinese women while the most common TAG in breast milk of Western women was OPO. This data will promote the development of infant formulas in terms of the TAG composition more suitable for infants.
Rapid assessment of deep frying oil quality as well as water and fat contents in French fries by low-field nuclear magnetic resonance


Most of the health hazards in fried foods are related to unqualified frying oil and excessive oil content. In this study, the feasibility of using low-field nuclear magnetic resonance techniques (LF-NMR) for analysis of the water and oil contents in French fries, as well as simultaneous evaluation of frying oil quality during deep frying, was investigated. Three proton populations were identified and successfully assigned to water and oil relaxation signals. Significant correlation between the T2 relaxation parameters (Awater and RCoil) and the water and oil content was acquired. MRI could visualize the changes of signal intensity and spatial distribution, as well as the internal structural changes during frying. Using the correlation model built by multiple regression analysis, the total polar compounds content of the frying oil could be successfully predicted by LF-NMR relaxation characteristics, which indicates that LF-NMR was an effective method to monitor the quality of frying oil.

Association genetics identifies single nucleotide polymorphisms related to kernel oil content and quality in *Camellia oleifera*


*Camellia oleifera*, as an important nonwood tree species for seed oil in China, has received enormous attention owing to its high unsaturated fatty acid contents benefited to human health. It is necessary to examine allelic diversity of key genes that are associated with oil production in *C. oleifera* cultivars with a large variation of fatty acid compositions. In this study, we performed the association analysis between four key genes (two CoSAD and two Cofad2) coding fatty acid desaturases and traits including oil content and fatty acid composition. We identified two single nucleotide insertion–deletion (InDel) and 362 single-nucleotide polymorphisms (SNPs) within the four candidate genes by sequencing an association–deletion (InDel) and 362 single-nucleotide polymorphisms related to kernel oil content and quality in *Camellia oleifera*. We successfully predicted the function of allelic variations significantly associated. In all, 90 single marker-trait and one haplotype-trait associations were significant in association population, and these loci explained 1.87–17.93% proportion of the corresponding phenotypic variance. Further, six SNP marker–trait associations (Q < 0.10) from Cofad2-A, CoSAD1, and CoSAD2 were successfully validated in the validation population. The SNP markers identified in this study can potentially be applied for future marker-assisted selection to improve oil content and quality in *C. oleifera*.

Highly efficient and enzyme-recoverable method for enzymatic concentrating omega-3 fatty acids generated by hydrolysis of fish oil in a substrate-constituted three-liquid-phase system


A novel three-liquid-phase system which contained fish oil as the nonpolar phase was developed for the lipase-based hydrolysis of fish oil and subsequent enrichment of the omega-3 polyunsaturated fatty acids (n-3 PUFA) in the glyceride fraction of the fish oil. In comparison with the traditional oil/water system, the enrichment factor of n-3 PUFA in this system was increased by 363.4% as a result of a higher dispersity, higher selectivity of the lipase for the other fatty acids except for n-3PUFA, and relief of product inhibition. The content of n-3 PUFA in the glyceride fraction could be concentrated to 67.97% by repeated hydrolysis after removing the free fatty acids. Furthermore, the lipase could be reused for at least eight rounds. This method would be an ideal approach for enriching n-3 PUFA because it is cost-effective, low in toxicity, and easily scaled up.

Droplet-stabilized oil-in-water emulsions protect unsaturated lipids from oxidation


Droplet-stabilized emulsions use fine protein-coated lipid droplets (the shell) to emulsify larger droplets of a second lipid (the core). This study investigated the oxidation resistance of polyunsaturated fatty acid (PUFA) oil within droplet-stabilized emulsions, using shell lipids with a range of melting points: olive oil (low melting), tristearin (high-melting), and palmolein oil (intermediate melting point). Oxidation of PUFA oil was accelerated with a fluorescent lamp in the presence of ferrous iron (100 micrometer) for 9 days, and PUFA oxidation was monitored via conjugated dienes, lipid hydroperoxides, and hexanal levels. Oxidation was slower in droplet-stabilized emulsions than in conventional emulsions or control emulsions of the same composition as droplet-stabilized emulsions but different structure, and tristearin gave the greatest oxidation resistance. Results suggest the structured interface of droplet-stabilized emulsions limits contact between pro-oxidants and oxidation-sensitive bioactives encapsulated within, and this antioxidative effect is greatly enhanced with solid surface lipids.
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