INFORM September 2017 Volume 28 (8)

INFORM

International News on Fats, Oils, and Related Materials

KEEPING PACE WITH CLEAN LABEL

ALSO INSIDE:

Viscosity in laundry detergents

Vitamin D analysis

Structuring fibers from sugar beets

AOCS

CONTENTS

DEPARTMENTS

INFORM

www.aocs.org

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

INFORM
International News on Fats, Oils, and Related Materials
ISSN: 1528-9303 IFRMEC 28 (8)

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

INDEX TO ADVERTISERS

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

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

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

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

AOCS Advertising:
Christina Morley
Phone: +1 217-693-4901
Fax: +1 217-693-4864
Christina.morley@aocs.org

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

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

Subscriptions to *Inform* for members of the American Oil Chemists' Society are included in the annual dues. An individual subscription to *Inform* is $195. Outside the U.S., add $35 for surface mail, or add $125 for air mail. Institutional subscriptions to the *Journal of the American Oil Chemists' Society* and *Inform* combined are now being handled by Springer Verlag. Price list information is available at www.springer.com/pricelist. Claims for copies lost in the mail must be received within 30 days (90 days outside the U.S.) of the date of issue. Notice of change of address must be received two weeks before the date of issue. For subscription inquiries, please contact Doreen Berning at AOCS, doreenb@aocs.org or phone +1 217-693-4813. AOCS membership information and applications can be obtained from: AOCS, P.O. Box 17190, Urbana, IL 61803-7190 USA or membership@ aocs.org.

Clean label: the next generation

Laura Cassiday

Simple recipes. Pronounceable ingredients. No artificial flavors or colors. Foods your great-grandmother would have made. Although the definition of "clean label" varies depending on whom you ask, the clean label trend has its roots in a distrust of synthetic food ingredients with "chemical-sounding" names that consumers do not understand. Whether or not fears of these ingredients are justifiable, many manufacturers and restaurants are reformulating foods to clean up their labels. Even as they do so, the definition of clean label continues to evolve, now encompassing such far-reaching attributes as an ingredient's traceability and a company's ethics.

- **The definition of "clean label" varies, but usually includes a lack of artificial flavors, colors, and preservatives, and simple ingredient lists with no unpronounceable or "chemical-sounding" additives.**
- **The next generation of clean label also encompasses ingredient sourcing and ethical issues.**
- **In response to consumer demand, many companies are reformulating their products to have clean labels, despite a lack of scientific evidence that such products are more healthful.**

CLEAN LABEL 1.0

A recent survey of 1,300 consumers in Europe, North America, and Asia found that 76% of respondents would be more likely to buy a product that contains ingredients they recognize and trust (Ingredient Communications, http://tinyurl.com/cleanlabelsurvey, 2016). In addition, more than 52% of respondents would be willing to spend more than 10% extra for products containing these ingredients. Consumers in the United States are willing to pay the highest prices, with 44% willing to pay 75% or more extra for ingredients they recognize and trust.

These survey results highlight the increasing importance consumers place on recognizing and understanding the ingredients on a product's label. Consumers typically trust ingredients that can be found in their home kitchens, such as milk, eggs, and flour, whereas they often distrust ingredients that sound like they came from a chemistry lab, such as potassium sorbate and mono- and diglycerides—notwithstanding decades of safe use in foods.

"Consumers are demanding cleaner labels, but even the consumers themselves do not understand exactly what that means," says Jeff Hilton, chief marketing officer and co-founder of BrandHive, in a recent FoodNavigator-USA webinar. "So while the demand is being consumer-driven, it's unclear what exactly consumers are looking for." A 2015 global survey by Canadean Ltd. Research Reports found that 34% of consumers worldwide admit that they do not understand what clean label means (Fig. 1). The three most popular clean label attributes were "free from artificial ingredients" (36%), "natural/organic claims" (34%), and "no pesticides/chemicals/toxins" (31%). Surprisingly, only 11% of respondents defined clean label as "simple/short ingredient lists."

In an attempt to define their own clean label policies, some restaurants and supermarkets have compiled lists of unacceptable ingredients (so-called "no-no lists") for their foods. In 2017, the Center for Science in the Public Interest (CSPI) evaluated the clean label programs of four major chain restaurants (e.g., Chipotle Mexican Grill, Panera Bread) and nine major chain supermarkets (e.g., Kroger, Whole Foods). The report found that all of the companies excluded additives that have a small risk of serious adverse effects, such as the artificial color Red 3, the artificial sweetener aspartame, and the artificial preservative TBHQ—all of which, at very high doses, have been shown in some studies to cause cancer in laboratory animals. All of the companies also prohibited many artificial ingredients that CSPI considers safe, lacking any evidence to the con-

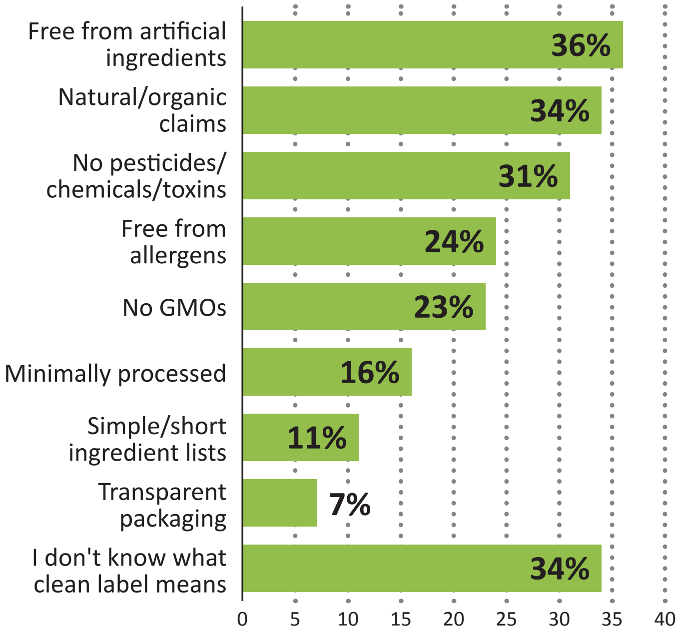


FIG. 1. Global survey: What does the term "clean label" mean to you? Credit: Canadean Ltd. Research Reports, Q4 global consumer survey, 2015.

Who really cares about clean labels?

A recent survey of 1,000 American adults (C&R Research, 2017) found that the definition and importance of "clean label" varied by generation:

Baby Boomers
The idea of clean label resonates most strongly with baby boomers (age 57 to 74 years). The top five clean-label priorities for this generation are the amount of sugar, sodium level, trans fats, artificial sweeteners, and high-fructose corn syrup. Clean-label concerns are driven primarily by age-related health issues.

Millenials
Millenials (age 21 to 35) are only slightly less concerned about clean labels than baby boomers. The top five clean-label priorities for millenials are the amount of sugar, all-natural, amount of protein, sodium levels, and preservatives. In general, millenials are young and in good health, but have the luxury of time to think about, shop, and prepare food.

Generation X
Gen Xers (age 36 to 56) are the least likely to care about clean labels. Their top priorities are whether the product is on sale, all-natural, amount of sugar, hormone-free, and trans fats. Generation Xers are juggling careers, children, and aging parents, so they have less time to worry about clean-label issues.

Source: C&R Research, 2017, http://tinyurl.com/generationclean.

trary, such as modified food starch (a thickener, stabilizer, and emulsifier) and calcium propanoate (an artificial preservative). "None of the companies' clean-label lists limit the amounts of sodium or added sugars, which cause far more harm than all other additives combined," notes the CSPI report.

None of the four restaurants' clean-label lists apply to beverages, which are a major source of some of the riskier additives, such as added sugars, artificial sweeteners, and synthetic food colors. With the exception of Whole Foods, which applies their "unacceptable ingredients" list to all foods and beverages sold in the store, the supermarkets only excluded the prohibited ingredients from their house brands.

CLEAN LABEL 2.0

The definition of "clean label" continues to evolve, making it difficult for food formulators to keep pace. "Where consumers began on the clean label movement—I call it 'clean label 1.0'—was all about less is more, simple is better. It was about what's not in the product, such as artificial colors and flavors," says Hilton. "What I think has happened now with clean label 2.0 is that consumers are going beyond that and wanting to dig down deeper into how the products are sourced, where they were sourced, more transparency regarding that sourcing, and they want to better understand the history of the product."

According to Hilton, "free-from" claims are merely expected. Now consumers want to know details about the agricultural methods used to produce the ingredients, such as GMO or pesticide usage, how the workers are treated, environmental sustainability, and fair trade practices. For animal products, consumers want to know about antibiotic and hormone use, animal welfare, and whether animals are cage-free, grass-fed, or free-range. Even a company's social mission and business ethics face scrutiny. Many consumers also look for a compelling story behind a brand—how it came to be, and the people behind the brand. Communicating all of the above, in addition to "free-from" claims, on a single package label can be challenging.

GOING CLEAN

Many food companies are reformulating their products in response to clean label concerns. However, it is often difficult to find natural replacements for tried-and-true synthetic ingredients that do not compromise efficacy, increase price, or affect sensory attributes of the food. Table 1 (page 8) shows possible clean-label replacements for some ingredients that are not considered clean-label, either because they are produced synthetically or have unfamiliar, "chemical-sounding" names.

In 2015, chocolate manufacturer Hershey pledged to remove GMOs and transition to simpler ingredients in its products. Jim St. John, senior director of chocolate product development at Hershey, told ConfectioneryNews that switching from GMO sugar (beet) to non-GMO sugar (cane) was simple because the ingredients perform the same function (Nieburg, O., 2016). Sometimes, cleaning up labels is as easy as renaming ingredients: "sodium bicarbonate" became "baking soda" on Hershey product labels to be better understood by consumers.

Much more challenging was finding replacements for tocopherols, malic acid, and ascorbic acid. Tocopherols, also

TABLE 1. Clean-label replacements

Non-Clean-Label Ingredients	Possible Clean-Label Alternatives
Artificial preservatives (e.g., TBHQ, sodium lactate, EDTA)	Botanical extracts (e.g., green tea, rosemary, citrus, chamomile, acerola cherry); tocopherols (can be labeled "vitamin E")
Emulsifiers (e.g., Mono- and diglycerides, sodium stearoyl lactylate, polysorbate 80)	Pea, faba, or lentil bean protein; Q-Naturale; phospholipids; egg yolk
Malic acid (natural flavor)	Apple juice concentrate
Texturizing and thickening agents (e.g., modified starches, hydrocolloids)	Blends of native starches (e.g., corn and tapioca); pea protein and native starch; rice flour; citrus fibers
Sodium phosphates (processed meats)	Rice starch; citrus fibers; soy protein; yeast extract; plum puree
Solvent-extracted vegetable oils	Expeller- or cold-pressed vegetable oils

known as vitamin E, are antioxidants used in Hershey's Cookies 'n' Créme chocolate to keep the milk in the white chocolate from going rancid and to prevent off-flavors. Although tocopherols are natural preservatives, their chemical-sounding names cause some consumers to view them with suspicion. In the US, tocopherols can be declared on labels as "vitamin E," but Hershey did not want to give the impression that the chocolate bar is fortified. "We haven't found a tocopherol replacement yet," says St. John. "It's a difficult one. We're also questioning whether it should be replaced."

In other reformulation efforts, Hershey removed the emulsifier polyglycerol polycrinoleate from its Milk Chocolate, instead increasing the cocoa butter percentage to improve the chocolate's flow properties. Artificial vanillin was replaced with a natural flavor, which required more than a year to identify. St. John notes that artificial vanillin has more consistent flavor effects than its natural replacement. "[Artificial vanillin] has a particular flavor effect on chocolate . . . it kind of mellows and smoothes chocolate flavor delivery out," he says. "We had to do a lot of testing to come up with the new flavor, and to be frank, it's a lot more expensive than vanillin." In Hershey's Brookside brand, malic acid—a natural flavor from fruit—was replaced with apple juice concentrate, a more recognizable, though less consistent, flavor ingredient. Although the reformulation efforts have decreased the efficiency of manufacturing and increased the cost, Hershey has not passed the price increase onto consumers, says St. John.

According to Marlene Schmidt, who is Nutrition, Health & Wellness Manager at Nestlé USA, Nestlé defines clean label ingredients by the "kitchen cupboard" standard. "These are ingredients that a consumer can recognize and trust, and that they may just find in their own kitchen at home," says Schmidt, in a recent FoodNavigator-USA webinar. Nestlé has committed to cleaning up the labels of many of its products, beginning with the company's best sellers. In 2017, Nestlé shortened ingredient lists and removed unfamiliar ingredients from their Skinny Cow ice cream line, and began sourcing milk from cows not treated with rBST.

Nestlé has also undertaken the challenging task of reformulating frozen dinners that are customer favorites, including Stouffer's Lasagna with Meat Sauce and Stouffer's Macaroni and Cheese. "I can't say enough that we don't want to compromise on taste for our consumers," says Schmidt. "So what we

Vegetable Juice (%)	LPI Emulsion (%)	Observation
100	0	
94	6	Undetectable change in flavor
91	9	Undetectable change in flavor. Barely noticeable bitterness
85	15	Slightly bitter

FIG. 2. When added to vegetable juice, a lentil protein isolate (LPI) emulsion induced cloudiness in the beverage, as desired, without signficantly affecting flavor. Beverage at left, 100% vegetable juice. Middle, 91% juice, 9% LPI emulsion. Right, 85% juice, 15% emulsion. Credit: Supratim Ghosh

do when we start this process is really work closely with our chefs and our food technologists. These two groups of experts work hand-in-hand to conduct multiple rounds of taste testing with our consumers. Then, once we agree upon a recipe, we take it out to even a larger group of consumers." Schmidt notes that the Macaroni and Cheese required 15 different test recipes to achieve a clean-label product that tasted the same or better than its predecessor.

At the 2017 AOCS Annual Meeting and Industry Showcases in Orlando, Florida, a Hot Topics Session focused on "Clean Label Ingredients and Processes for Food and Beverages." Several speakers discussed promising new approaches to clean-label ingredients. Supratim Ghosh, assistant professor of food and bioproduct sciences at the University of Saskatchewan, in Saskatoon, Canada, presented his research on the use of pulse proteins as natural stabilizers of oil-in-water emulsions. Pulses are edible seeds of plants in the legume family, and pulse proteins, such as pea protein isolate (PPI) and lentil protein isolate (LPI), are natural emulsifiers.

Ghosh and his colleagues investigated the ability of an LPI solution to stabilize emulsions in several commercial beverages (apple, orange, and vegetable juice; lemonade; kefir; and soy milk). The researchers found that they obtained more stable emulsions with smaller droplets if they pre-homogenized the LPI solution prior to forming an emulsion. The pre-homogenization step may expose buried functional groups in the globular LPI proteins, enhancing their emulsification properties.

Ghosh added the pre-homogenized aqueous LPI solution (95 wt%) to canola oil (5 wt%) and formed a nanoemulsion by high-pressure homogenization. They then mixed the LPI nanoemulsion with the beverages at different inclusion rates to assess their appearance and taste. The vegetable juice produced the most desirable results (Fig. 2). At the highest inclusion rate of LPI (15%), the vegetable juice retained its flavor and body, with only a slight change in bitterness, appearance, and oily mouthfeel. None of the other juices was able to mask the bitter aftertaste and oily mouthfeel of the LPI nanoemulsion.

Also during the clean label Hot Topics session, Mark Stavro, global marketing director at Bunge, discussed clean label trends for vegetable oils. Consumers are increasingly seeking minimally processed, natural, and non-GMO vegetable oils, says Stavro. Traceable, sustainable origins for the oils are important. A clean-label vegetable oil is expeller- or cold-pressed, rather than hexane-extracted, undergoes minimal refining, and contains natural antioxidants, such as rosemary or chamomile extract, rather than synthetic antioxidants such as TBHQ. Examples of milder refining conditions include using citric acid or enzymes rather than phosphoric acid for degumming, and using physical rather than chemical refining to eliminate the need for alkali neutralization with sodium hydroxide. Chemically modified oils, such as hydrogenated, partially hydrogenated, or brominated vegetable oils, are not considered clean label. According to Stavro, few vegetable oils are currently making claims of a low glycidyl ester content, but this is an area of growing importance to consumers.

WHAT'S IN A LABEL?

If a manufacturer expends the time and money to reformulate a product, they want to effectively communicate the product's clean-label attributes to consumers. However, much of the information consumers seek will not fit on the product packaging. As a result, in 2015 representatives from 90 manufacturers, retailers, trade associations, and special interest groups created SmartLabel (smartlabel.org). SmartLabel is an online platform that allows consumers to search for detailed information on the more than 7,000 products that currently carry SmartLabels. An estimated 34,000 products will have SmartLabels by the end of 2017. Consumers can search for products on the website, or scan SmartLabel QR codes on product packaging. SmartLabel provides information such as ingredients lists, allergens, "free from" claims, GMO disclosures, third-party certifications, and sourcing practices.

In 2016, a clean-label certification scheme called GoCleanLabel was launched (gocleanlabel.com). To become GoCleanLabel-certified, a brand must undergo a product evaluation, in which the ingredients are compared to "unacceptable ingredients" lists from influential supermarkets and restaurants such as Whole Foods, Panera, Kroger, and Trader Joe's. If the product meets clean-label criteria, the manufacturers may use the GoCleanLabel-certified logo on their packaging, website, and marketing materials. Because the definition of clean label changes frequently, the certification is only valid for 12 months, after which the product must be re-evaluated. Consumers and food industry professionals may search the GoCleanLabel website for specific ingredients, and the site will indicate whether or not the ingredient is considered clean label.

Other third-party certification schemes with relevance to the clean label movement include USDA Organic, Non-GMO Project Verified, and RSPO-certified (for palm oil). Recently, a company called BioChecked launched a "non-glyphosate" certification. Companies submit product samples to a partner lab for testing and provide a list of all ingredient sources. If no traces of glyphosate are detected, the product can display the Non-Glyphosate Certified seal.

THE DIRTY TRUTH ABOUT CLEAN LABELS

The clean label movement is one of the hottest trends in the food industry, and it only appears to be growing in scope and magnitude with each passing year. However, the movement is not without its critics. The American Heart Association (AHA) recently issued statements to the effect that "clean label" does not equal "healthy." Products with clean labels can still be loaded with sugar, salt, and fat. In comments submitted to the US Food and Drug Administration about the use of the term "healthy" on food products (http://tinyurl.com/AHAhealthy), AHA President Steven R. Houser notes that in surveys, many consumers define "healthy" by such criteria as organic, minimally processed, natural, non-GMO, or free of artificial ingredients. "Definitions for many of these factors are not clearly defined in law nor agreed upon by stakeholders," says Houser. "Furthermore, and perhaps more importantly, there is not sufficient scientifically sound evidence linking them to health outcomes." The AHA supports reserving the term "healthy" for foods that satisfy the 2015–2010 Dietary Guidelines for Americans.

Other critics worry that the clean label movement will cause manufacturers to stop fortifying foods with vitamins and minerals, many of which have "chemical-sounding" names. Indeed, according to Euromonitor International, between 2010 and 2015 there were declines in fortified iron, vitamin A, and vitamin K consumption in North American, and in fortified vitamin A, vitamin C, and vitamin D consumption in Western Europe (Menayang, A., 2016). Cutting fortified vitamins and minerals is an easy and cost-effective way to shorten ingredient lists. Also, because some vitamins are produced from genetically modified organisms or feedstock, manufacturers may cease fortification to obtain non-GMO certification. Because fortified foods are an important source of vitamins and minerals for many people, some dietitians worry that the clean label trend will result in negative health consequences. For example, in the United States, flour, pasta, and other grain products are routinely fortified with folic acid, a B vitamin that, when consumed by pregnant women, reduces the risk of neural tube defects in infants.

A downside of clean label claims is that they could increase litigation risks for manufacturers. Like "natural" and "healthy," "clean label" has no legal definition and is therefore open to the interpretation of the consumer. Some natural flavors contain synthetic non-flavor ingredients, such as artificial preservatives, colors, and emulsifiers. And some natural ingredients are produced by fermentation processes using genetically modified organisms. If a "reasonable consumer" could be misled by such clean label claims, then the manufacturer may be hit with costly and damaging lawsuits.

Many food formulators worry that the clean label movement unfairly demonizes safe and legal ingredients that have been used in foods for decades with no evidence of adverse health effects. Synthetic food additives have been rationally designed and improved over the years to be highly efficient at what they do, whether it be preservation, emulsification, or flavoring. Natural alternatives, if they exist, are typically less efficient and more costly. The irony of the clean label movement may be that its proponents wish to turn back the clock to great-grandmother's days, when great-grandma might have been happy to have access to time-saving, effective, safe, and inexpensive ingredients that kept her food fresh longer.

Laura Cassiday is an associate editor of Inform *at AOCS. She can be contacted at laura.cassiday@aocs.org.*

Information

Center for Science in the Public Interest (CSPI). (2017) "Clean labels: public relations or public health?" http://tinyurl.com/CSPI-cleanlabel

FoodNavigator-USA. "Where next for clean label?" Editorial webinar, May 23, 2017. Free registration, http://tinyurl.com/ceanlabelwebinar

Menayang, A. (2016) "In the age of clean label, are consumers in developed markets losing interest in fortified foods?" FoodNavigator-USA.com, September 21, 2016. http://tinyurl.com/FoodNav-fortification

Nieburg, O. (2016) "Removing GMOs easy; ditching chemical-sounding ingredients the challenge, says Hershey R&D chief." ConfectioneryNews.com, December 15, 2016. http://tinyurl.com/Hershey-cleanlabel

How to choose drop-in replacements for active ingredients in existing liquid laundry detergent formulations

Thu Nguyen

- Formulation viscosity is an important property of liquid laundry detergents. Due to the complex makeup of such formulations, selecting a drop-in replacement for one or both of the main active ingredients in existing liquid laundry detergents while maintaining the formulation viscosity requirement can be challenging.

- Our research and development team recently investigated how surfactant parameters affect formulation viscosities.

- This article summarizes our results, which formulators can use to identify surfactants as direct drop-in replacements for alcohol ethoxylate (AE) and/or alcohol ether sulfate (AES) in existing liquid laundry detergent formulas.

Viscosity is an important consideration in formulating liquid laundry detergents, as it can affect consumer perceptions and formulation stability (Drozd and Gorman, 1988). Detergents with viscosities that are too low are often viewed by consumers as weak and insufficient for cleaning performance, while detergents with viscosities that are too high may be difficult to pour and leave significant amounts of residual detergent stuck in the measuring cup, especially at cold temperatures. Viscosities that are too high can also cause formulation stability problems. In some markets, liquid laundry detergents have a typical viscosity range of 100–500 cPs, whereas in other markets where consumers like much thicker products, the viscosity can be in the range of 2000–3000 cP (Lai, 1997).

Liquid laundry detergents are composed of active ingredients (surfactants) and inactive ingredients (solvents, stabilizers, and carriers—often referred to as hydrotropes). Such formulations usually use anionic and nonionic surfactants. The nonionic surfactant is typically an alcohol ethoxylate (AE). In the past, linear alkylbenzene sulfonate (LAS) was almost the only anionic used in liquid laundry detergents, but current formulations also include the use of alcohol ether sulfate (AES). Matson and Berretz published a series of papers discussing the effect of hydrotropes, the choice of nonionic surfactant, and the nonionic/anionic surfactant ratio on the viscosity, solubility, and detergency performance of liquid laundry detergents (Matson and Berretz, 1979 and 1980). These papers show that the effect of hydrotrope and surfactant properties on the viscosity and solubility is not uniform among different liquid formulations. Also, each detergent manufacturer has its own viscosity standard for its formulations.

There are a wide variety of chemicals that can be used to adjust the physical properties (viscosity and stability) of liquid laundry detergents. When developing a new formulation, there is more freedom in selecting the group of appropriate chemicals that produce the desirable formulation properties. However, if one or more active ingredients in an existing liquid laundry detergent formulation must come from an alternative feedstock, selecting a drop-in replacement that will not require other formulation modifications can be challenging. Consequently, this article focuses on how to effectively select a drop-in replacement for currently used nonionic AE or anionic AES in existing liquid laundry detergent formulations. The goal of our investigation was to identify one drop-in replacement surfactant for as many existing formulations as possible. Since even one liquid laundry detergent manufacturer can have hundreds of formulations that have completely different makeups, being able to identify one surfactant as a drop-in replacement for an active ingredient in all of the formulations will require minimum changes to the existing manufacturing process.

Previous studies (Dillan, 1985 and Cox, 1989) have shown the effect of alcohol molecular weight and the degree of ethoxylation on the viscosity of AE and AES. Our study looked at the effect of surfactant parameters, including the degree of alcohol branching, the alcohol molecular weight (MW), and the degree of ethoxylation, on formulation viscosities.

The formulas used for this study are listed in Table 1. The formulas are categorized as high-viscosity formulas (H1 to H4) and low-viscosity formulas (L1 to L5). The value of the targeted viscosity for each formula was obtained from the complete existing formulas, also referred to as control formulas. The "HOLE" formula of each formula listed in Table 1 was used for the evaluation. The "HOLE" formula is the complete formula without the active ingredient (AE or AES).

Viscosity was measured using a Brookfield DVT2 viscometer at room temperature (21±0.5°C) after the formulas were prepared and equilibrated for 24 hours.

TABLE 1. Liquid laundry detergent formulas studied

Formulation #	% 7 mole AE	% 3 mole AES	Target viscosity, cP
H1	4.9	10.5	1268
H2	1.66	5.8	1172
H3	0	4.1	1024
H4	10	N/A	1000
L1	12.82	N/A	544
L2	0.6	N/A	298
L3	16	N/A	297
L4	5.4	N/A	288
L5	23.07	N/A	235

DROP-IN REPLACEMENT FOR NONIONIC SURFACTANTS

Figure 1 (page 14) shows the effects of the degree of ethoxylation, the alcohol MW, and the degree of alcohol branching of the AE on the viscosities of five low-viscosity formulas. All three parameters show no significant effect on the viscosities of Formulas L1 and L5, while the effect is more pronounced for Formulas L2, L3 and L4. Among the three parameters, the degree of alcohol branching shows no effect on the formulation viscosity. The effects are more obvious with changes in the degree of ethoxylation and alcohol MW, in which the formulation viscosities increase with decreasing ethoxylation (EO) number and increasing alcohol MW. However, it should be noted that AE with too low of an EO number or too high of an alcohol MW could create formulation stability issues due to the low solubility of the surfactant.

None of the studied AE with 7 moles of EO was able to produce the desirable viscosities (indicated by the control formulas) for formulas L3 and L4. For the AE with 6.5 moles of EO, some with a low MW of 199 produced viscosities that were

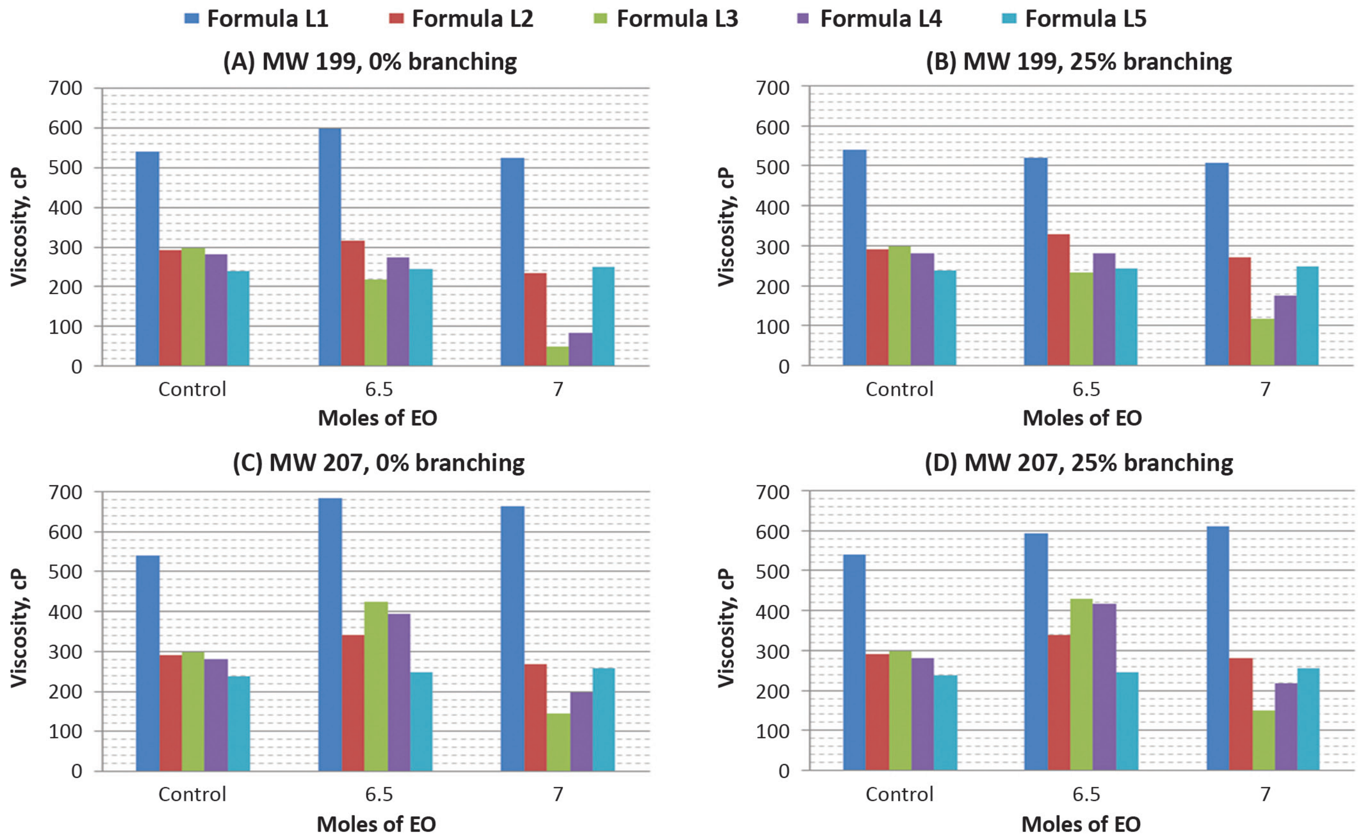


FIG. 1. The effects of ethoxylation degree, alcohol MW, and degree of alcohol branching on the viscosities of low-viscosity formulas

too low, while some with a high MW of 207 produced too viscosities that were higher than the desired formulation viscosities. Due to the insignificant effect of the degree of alcohol branching, two linear AE with 6.5 moles of EO and MW of 205 and 206 were further evaluated (Fig. 2). The results show that the AE with MW of 205 resulted in slightly lower viscosities for formulas L3 and L4 than the AE with MW of 206. In general, within allowable variations of viscosities, all three AE with 6.5 moles of EO from the alcohols with MW of 205, 206, and 207 would potentially produce desirable viscosities for all five of these low-viscosity formulas. The cloud point tests show no physical instability of these formulas at 50°C, even for formulas using AE of alcohol MW of 207. It should be noted that these alcohol MW were obtained by blending different alcohols.

For high-viscosity formulas H1 and H2, AE of alcohol blends with high MW created phase separation in the formulas at both 4°C and 50°C, especially at 6.5 moles of EO. The effect of all surfactant parameters on the formulation viscosities was similar to that observed for the low-viscosity formulas. The formulation viscosity increased with decreasing degree of ethoxylation, decreasing degree of alcohol branching, and increasing alcohol MW (Fig. 3). However, the effect of alcohol MW for AE with 6.5 moles of EO was not as significant as the effect of the degree of ethoxylation and the degree of alcohol branching for these high-viscosity formulas. As seen in Fig. 3B, some degree of alcohol branching is required to lower the formulation viscosity to the desired value. Any AE made from alcohol blends of MW ranging from 195 to 203 was able to produce the desirable formulation viscosities for both formulas H1 and H2 without creating the formulation instabilities at 4 and 50°C, while phase separation was encountered in formulations using AE made from alcohol blends of MW higher than 203.

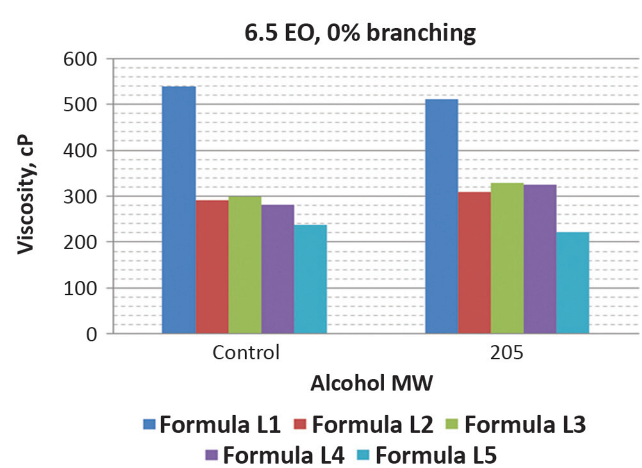


FIG. 2. Formulation viscosities using AE with 6.5 moles of EO of two linear alcohols with MW of 205 and 206

In other high viscosity formulas such as formula H4, a salt-viscosity response for the formulation using different AE was evaluated. As shown in Fig. 4, alcohol MW had a significant effect on the salt-viscosity response of the formula. Similar effects of the three surfactant parameters were observed, in which the formulation viscosity increased with decreasing moles of EO, decreasing the degree of alcohol branching, and increasing alcohol MW.

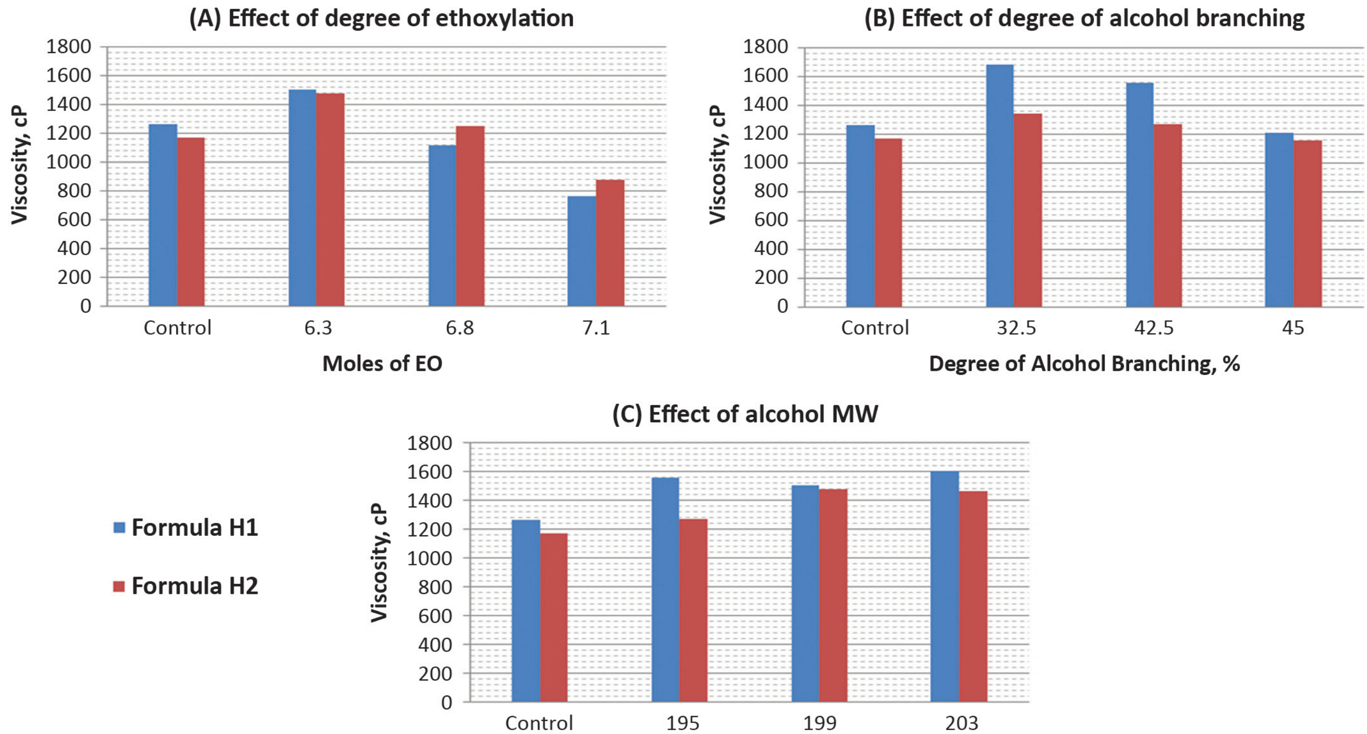


FIG. 3. The effects on formulation viscosities for high-viscosity formulas H1 and H2 by (A) the degree of ethoxylation (alcohol MW 200), (B) the degree of alcohol branching (6.5 EO, alcohol MW 195), and (C) the alcohol MW (6.5 EO)

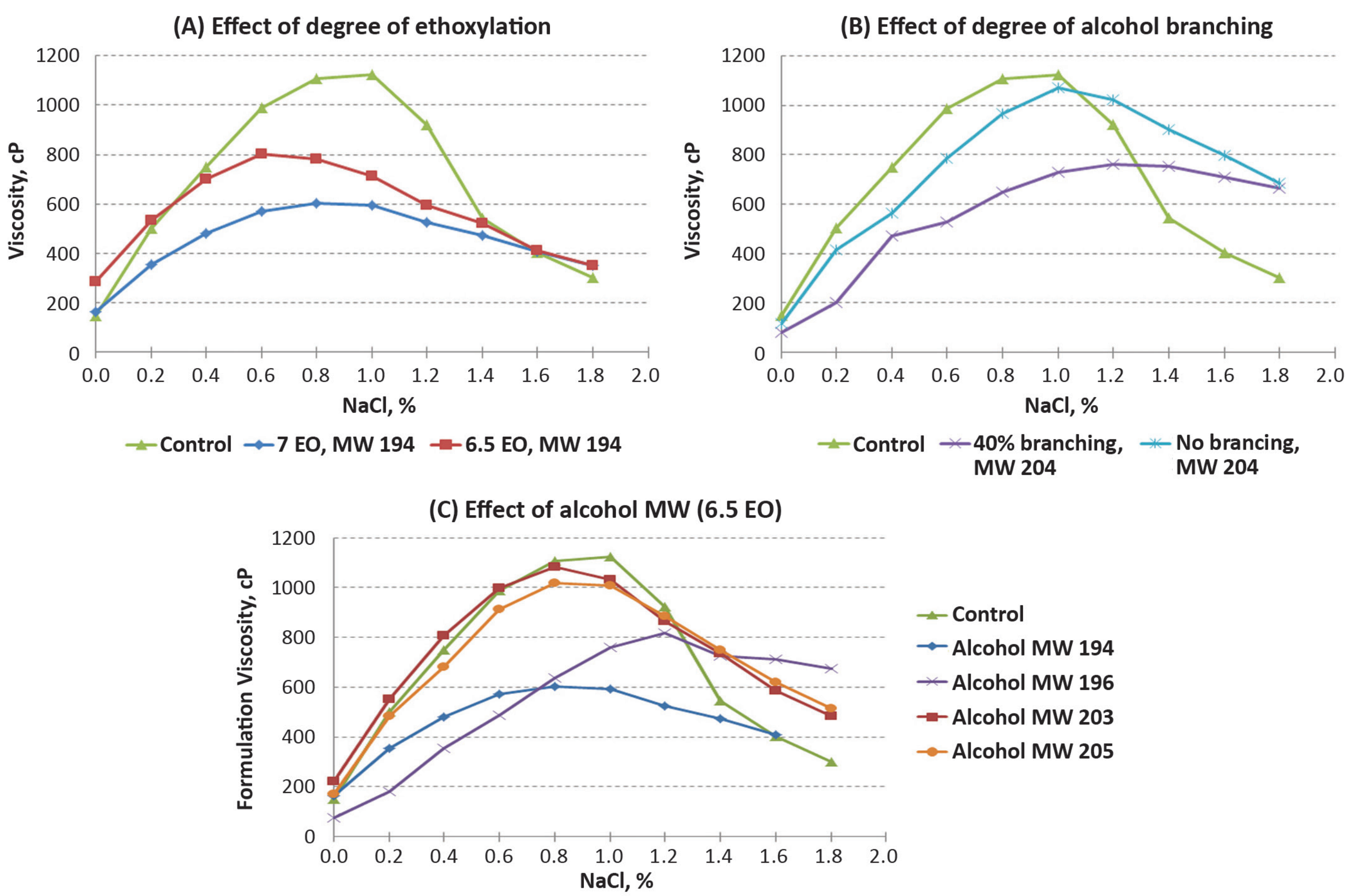


FIG. 4. The effects on the salt-viscosity profile of formula H4 by (A) the degree of ethoxylation, (B) the degree of alcohol branching, and (C) the alcohol MW

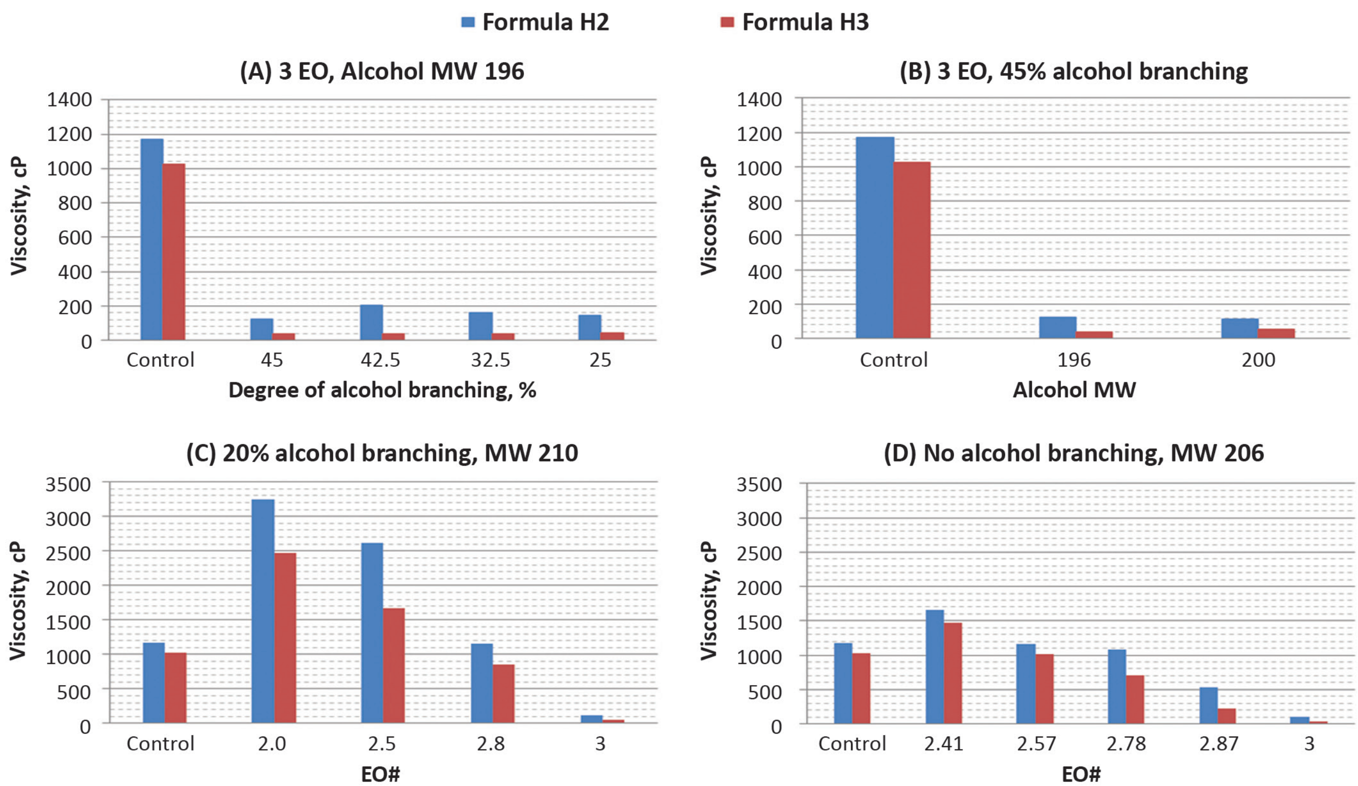


FIG. 5. The effects on formulation viscosities of formulas H2 and H3 by (A) the alcohol branching degree, (B) the alcohol MW, and (C) & (D) the ethoxylation degree of different alcohol MW

DROP-IN REPLACEMENT OF ANIONIC SURFACTANTS

The evaluation of the anionic surfactant was based on formulas H2 and H3. The evaluation also focused on the effects of the three surfactant parameters discussed above.

Figures 5A & B clearly indicate an insignificant effect of degree of alcohol branching and alcohol MW at 3 mole EO on the formulation viscosity. All the 3 mole AES of different alcohol MW and degree branching studied were not able to produce the desired viscosities for both formulas H2 and H3. However, figures 5C & D demonstrate that a small decrease in the number of moles of EO from 3 to 2.8 could significantly increase the viscosities of both formulas. A further decrease in the EO number could, however, make the formulas too thick. The results in Figs. 5C & D also indicate that AES with lower

References

Drozd, J.C. and W. Gorman, Formulating characteristics of high and low 2-phenyl linear alkylbenzene sulfonates in liquid detergents, *JAOCS 65*: 398–404, 1988.

Lai, K.Y. (Ed.) *Liquid Detergents*. Marcel Dekker, Inc. (1997).

Matson, T.P. and M. Berretz, The formulation of non-built heavy-duty liquids, Part I(A): The effect of hydrotropes on physical properties (viscosity), *Soap/Cosmetics/Chemical Specialties,* pp. 33 et. seq. (1979).

Matson, T.P. and M. Berretz, The formulation of non-built heavy-duty liquids, Part I(B): The effect of hydrotropes on physical properties (solubility), *Soap/Cosmetics/Chemical Specialties,* pp. 41 et. seq. (1979).

Matson, T.P. and M. Berretz, The Formulation of Non-Built Heavy-Duty Liquids, Part II: The Nonionic Hydrophile and Its Effect on Non-Built Heavy-Duty Liquids," *Soap/Cosmetics/Chemical Specialties,* pp. 36 et. seq. (1980).

Matson, T.P. and M. Berretz, The formulation of non-built heavy-duty liquids, Part III: Physical properties and detergency performance with changes in surfactant type and concentration, *Soap/Cosmetics/Chemical Specialties,* pp. 41 et. seq. (1980).

Dillan, K.W., Effects of the ethylene oxide distribution on nonionic surfactant properties, *JAOCS 66*: 1144–1151, 1985.

Cox, M.F., Effects of alkyl carbon chain length and ethylene oxide content on the performance of linear alcohol ether sulfates, *JAOCS 66*: 1637–1646, 1989.

alcohol MW would require a lower mole of EO (MW 206 and 2.6 EO), while AES with higher alcohol MW would require a higher mole of EO (MW 210 and 2.8 EO) to produce desired viscosities for both formulas.

These results suggest that the makeup of every liquid laundry detergent formula is different. However, the selection process of a nonionic AE and/or an anionic AES surfactant replacement for currently used surfactants in existing formulas can be narrowed down to the evaluation of three surfactant parameters: degree of ethoxylation, alcohol MW, and degree of alcohol branching. In the case of most nonionic surfactants, a decrease in the degree of ethoxylation and an increase in the alcohol MW increased the formulation viscosity, while the effect of the degree of alcohol branching was insignificant. In some cases, where the degree of alcohol branching affects the formulation viscosity, the nonionic AE with a lower degree of alcohol branching produced higher formulation viscosity. For anionic surfactants, the degrees of ethoxylation and alcohol branching showed a more pronounced effect than the alcohol MW. Therefore, having a wide portfolio of alcohol structures can make this process more efficient and successful both technically and economically.

Thu Nguyen is a senior research engineer and Surfactant Applications group leader at Sasol Performance Chemicals, Westlake, Louisiana, USA. She can be contacted at Thu.Nguyen@us.sasol.com.

Acknowledgment: The author acknowledges the R&D team and the management at Sasol for the contribution and the support for this study.

AOCS MEETING WATCH

September 11–14, 2017. 17th AOCS Latin American Congress and Exhibition on Fats, Oils, and Lipids, Grand Fiesta Americana Coral Beach Hotel, Cancun, Mexico. http://lacongress.aocs.org

May 6–9, 2018. AOCS Annual Meeting & Expo, Minneapolis Convention Center, Minneapolis, Minnesota, USA.

September 6, 2018. JOCS AOCS Joint Symposium, Kobe Gakuin University, Arise Campus, Kobe, Japan

October 28–31, 2018. Fabric and Home Care World Conference, Boca Raton Resort & Club, Boca Raton, Florida, USA.

For in-depth details on these and other upcoming meetings, visit http://aocs.org/meetings or contact the AOCS Meetings Department (email: meetings@aocs.org; phone: +1 217-693-4821; fax: +1 217-693-4865).

Unraveling the power of microcellulosic fibers

Robert Nolles

Sugar beet pulp is a major residual stream from the sugar industry (Fig. 1). Approximately 4 to 5 million tons are generated annually in the United States alone, and this biomass is underutilized as low-value animal feed, feedstock for green gas production, or fertilizer.

- Better use of biomass side streams from food ingredient production is key to a biobased or circular economy.
- The production of high-performance microcellulosic fiber material from sugar beet pulp is an example of how biorefining can turn a low-value side stream into a product that is valuable to end users.
- The result is a suspension-stabilizing additive with fascinating rheological and structuring properties that is finding its way into home, fabric, and personal care products.

To make better use of sugar beet pulp, Royal Cosun, a Dutch agro-industrial cooperative that uses vegetable raw materials to manufacture ingredients for food, non-food applications, and the chemical industry, invested in an integrated and cost-effective cascading biorefinery to refine sugar beet pulp and isolate high-value components for use in a broad variety of end products. The overall objective of the biorefinery initiative is to establish value chains based on microcellulose fibers, arabinose, and galacturonic acid in high-value markets such as detergents, paints, coatings, and composites—as well as in personal care and the oil and gas industries (Fig. 2). Cumulatively, the three main constituents of sugar beet pulp account for 65% of its mass, and the residuals of the biorefinery are suitable to generate biogas.

MICROCELLULOSIC FIBERS FROM SUGAR BEET PULP

One of the main fractions obtained from the sugar beet pulp refinery is parenchymal cellulosic material. The root vegetable origin of the cellulose in combination with the patented process for extraction, purification, and morphological transition of the cellulose results in rheological functionalization of this non-lignin based cellulose. The microcellulosic fiber product obtained after processing is a 100% natural biopolymer that has not been chemically modified and is completely biodegradable. The fibers are derived from renewable vegetable resources that do not compete with the food chain.

The parenchymal cellulose-based materials are called microcellulosic fibers, and they are marketed under the brand name Beta*fib*® MCF. These fibers provide unique opportunities to develop novel solutions for particle-carrying compounds in fluid water-based compositions [1]. The first manufacturing line for this innovative microcellulosic product, built in The Netherlands, was commissioned in late 2016.

COMPOSITION AND DIMENSIONS

The composition of Beta*fib*® is rather complex. The material is a particulate cellulose material containing at least 60% cellulose, 0.5–10%

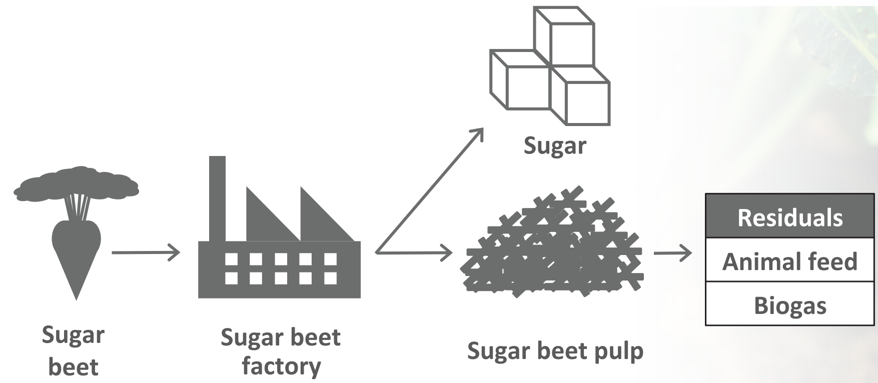


FIG. 1. Traditional value chain for sugar beet

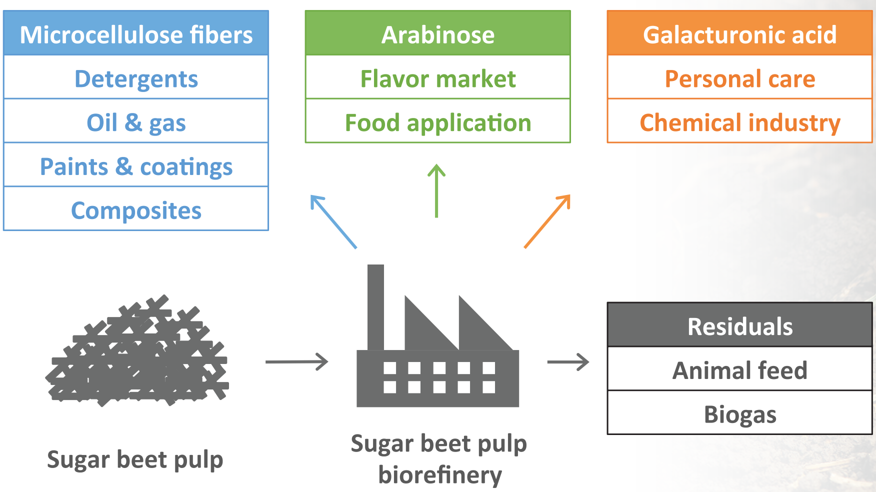


FIG. 2. Biorefining of sugar beet pulp

pectin, and 1–15% hemicellulose, and it has a particle dimension within the range of 25–75 µm. The diameter of the particles is typically in the magnitude of tenths of microns. It is assumed that within the cellulose particles the organization of the cellulose fibrils, as it exists in the parenchymal cell walls, is at least partly retained—even though some of the pectin and hemicellulose is removed during the manfacturing process. Furthermore, the cellulose-based nanofibrils are not completely unraveled (i.e., the material is different from cellulose nanofibers).

Studies have shown that at least some hemicellulose or pectin must be retained in the material to support the structural organization of the cellulose in the particles (e.g., by providing an additional network). Such hemicellulose networks would hold the cellulose fibers together, thereby providing structural integrity and strength to the cellulose particle.

THE UNIQUE PROPERTIES OF MICROCELLULOSIC FIBERS

Once introduced into an aqueous environment, the microcellulosic fibers form a particle gel and create a physical 3D network. This leads to viscosity built up, delivers high zero shear viscosity, and results in strong shear thinning behavior of the gel that is formed. Figure 3 (page 20) shows a high viscosity value (η) under no and low shear (γ) that the microcellulosic fibers establish, next to a strong shear thinning character. The physical network formed by the microcellulosic fibers tends to be stronger than the chemical network typically built by nanofibers. Hence, the measured yield point of a gel structured by the microcellulosic fibers exceeds the yield point of gels structured by well-known structurants or viscosifiers like CMC, xanthan gum, and guar gum.

Beta*fib*® microcellulosic fibers have a very distinct character compared to, for instance, xanthan gum (Fig. 4, page 20). The storage modulus (G') shows an elevated value, indicating that the structure built with the microcellulosic fibers is firmer. This is a clear sign of superior suspension properties. As a result of the rheological properties, microcellulosic fibers are remarkably effective in preventing migration of suspended solid particles, encapsulated actives, or gas bubbles. For example, the structure can prevent sedimentation of non-colloidal suspended particles in water-based fluids.

The microcellulosic fibers have been found to retain their performance as a suspension-stabilizing additive under harsh conditions, including temperatures up to 350°F, at high

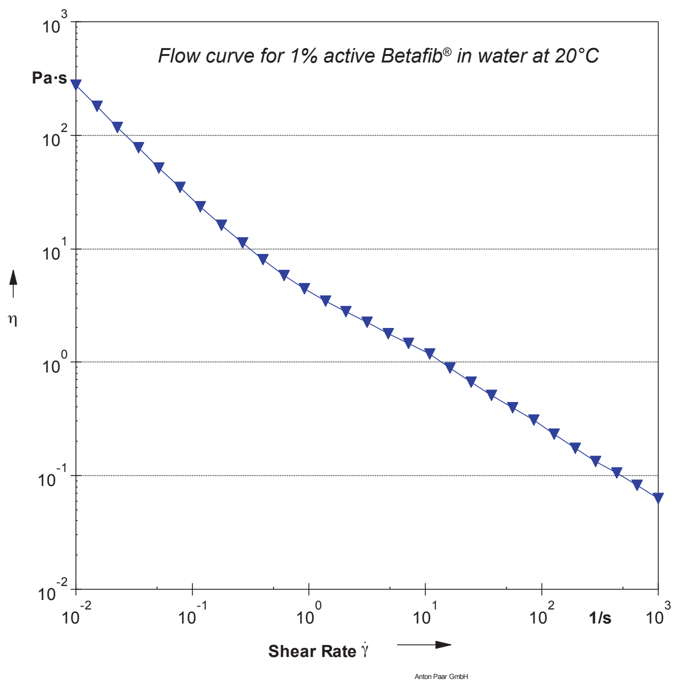


FIG. 3. Flowcurve for 1% active Beta*fib*® microcellulosic fibers in water at room temperature. Rheometer: Physica MCR 301, Anton Paar.

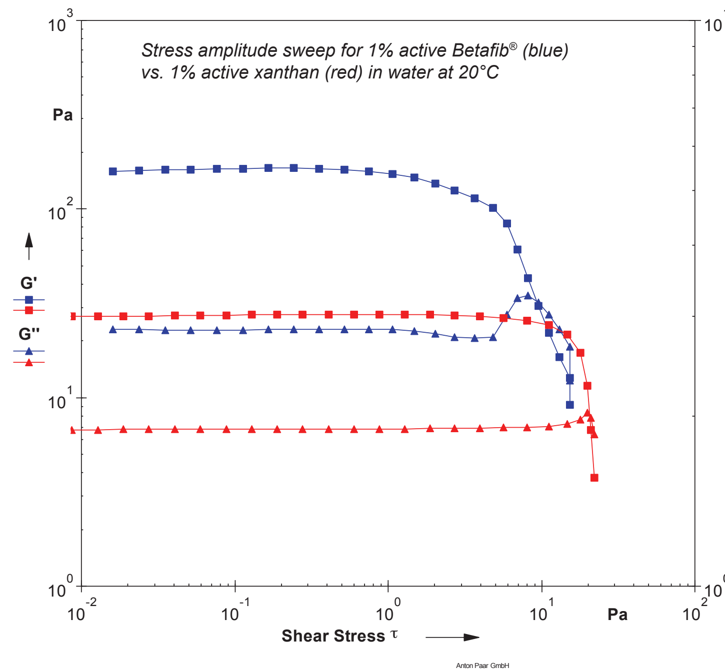


FIG. 4. Stress amplitude sweep of 1% active Beta*fib*® (blue) vs. 1% active xanthan (red) in water at room temperature. Rheometer: Physica MCR 301, Anton Paar.

and low pH values, and in the presence of certain oxidizing or reducing agents. Moreover, test compositions structured with the microcellulosic fibers and containing high concentrations of surfactant or electrolytes showed remarkable stability compared to many known structuring agents. Since the fibers are nonionic to slightly anionic they show a significantly lower water holding capacity than many other structurants.

APPLICATION BENEFITS

The free space within the physical network built by the microcellulosic fibers enables the ability to "entrap" particles, such as beads and encaps, or gas bubbles. Consequently, the density of the materials that must be suspended compared to the density of the liquid is of little importance. In addition, physical networks have the capability to carry more "weight."

TABLE 1. Overview on applications and functionalities of Beta*fib*® in home, fabric, and personal care

Application benefit	Liquid laundry	Other home care	Oral care	Rinse off personal care	Leave on personal care	Examples
Vertical cling of cleaners		●				Toilet bowl / oven / drain / bathroom
Suspension of actives	●	●		●	●	Hand dish / fabric conditioner / abrasive cleaner / sun screen / liquid detergent / cream and gel
No issues with density differences of actives	●			●	●	Prevention of creaming / prevention of settling
Foam stability				●		Shower gel / shaving gel
Ease of dosing			●	●		Tooth paste / shampoo / shower gel
Improving shelf life	●			●	●	Fabric conditioner / oil-in-water emulsion
Skin feel				●	●	Body lotion / sun screen / shower gel
Rinsability		●		●	●	Various cleaners / shampoo / scrub / gel

This property is relevant for formulations that must carry particulates such as abrasives, TiO_2, or ZnO. Given the "virtual fixation" by the 3D network, suspended materials neither sink nor float during rebuilding of the network. As a consequence, fluids remain homogeneous during the application of shear and after shear is released.

Based on the intrinsic properties of the microcellulosic fibers, numerous potential application areas have been identified. An overview of applications and corresponding functionalities is given in Table 1.

Categories within the home care space for application of the microcellulosic fibers are abrasive cleaners, toilet bowl cleaners, and automatic dishwasher detergent gels. These are all products with known formulation challenges. In toilet bowl cleaners, for example, the strong shear thinning behavior of the fibers allows easy application using a spray bottle, and high zero shear viscosity supports vertical cling. Moreover, because purified cellulose is a rather inert material, it can function with the low pH of a typical toilet bowl cleaner.

Beta*fib*® is also suitable for use in liquid laundry detergents and softeners. The structure built by the microcellulosic fibers allows encapsulated fragrances to be effectively suspended for long-lasting freshness uses. Other catagories within personal care in which the microcellulosic fibers can resolve formulation challenges are anti-dandruff shampoo, scrubs, and sunscreens. All of these products are typically difficult to stucture or have potential stability issues that can be overcome by Beta*fib*®.

Since the fibers are derived from renewable resources, are not modified or derivatized, and are 100% biodegradable, the sustainability profile is excellent. Hence, there is a natural fit in product catagories with natural or sustainability claims. Last but not least, due to the temperature resistance of the material, it could improve shelf life and be beneficial in dealing with challenging transportation and warehousing conditions for sensitive formulations.

Robert Nolles is marketing and sales director at Cosun Biobased Products, San Jose, California, USA. He can be contacted at robert.nolles@cosun.com.

References

[1] Stabilization of suspended solid particles and/or gas bubbels in aqueous fluids; G.P.F.M. van Engelen, G.A. van Ingen, C. Meeuwissen and R. Nolles; Patent WO 2014142651-A, Sep 2014

[2] PULP2VALUE, Processing Underutilised Low value sugar beet Pulp into VALUE added products, http://pulp2value.eu/, Mar 2015

Analyzing **vitamin D** and **previtamin D** in foods by **UPLC-MS and PTAD derivatization**

Jinchuan Yang and Gareth Cleland

- **Recent developments in analyzing vitamin D in foods by liquid chromatography (LC) include the incorporation of a derivatization reaction using 4-Phenyl-1,2,4-triazoline-3,5-dione (PTAD), and the adoption of mass spectrometry (MS) for a simpler sample preparation process and greater sensitivities.**

- **However, the previtamin D in foods is not measured in LC-MS methods that use PTAD derivatization. This could lead to big errors since a significant amount of vitamin D can exist as previtamin D.**

- **Our research group recently developed and tested a new LC-MS with PTAD derivatization method for directly measuring both the previtamin D and the vitamin D in foods. The total vitamin D was the sum of these two vitamin D forms. This method enables accurate determination of the vitamin D in foods.**

Vitamin D is a fat-soluble vitamin that promotes calcium absorption and maintains adequate serum calcium and phosphate concentrations [1]. The most common vitamin Ds are vitamin D_3 (also known as cholecalciferol) and vitamin D_2 (ergocalciferol). Their structures are shown in Figure 1. Vitamin D can be produced endogenously when ultraviolet (UV) light strikes the skin and triggers vitamin D synthesis. Recent studies revealed that certain populations still rely on diets to get adequate supplies of vitamin D [2]. The US Food and Drug Administration (FDA) requires vitamin D concentration to be listed on nutrition or supplement facts sheets for all food and dietary supplement products [3].

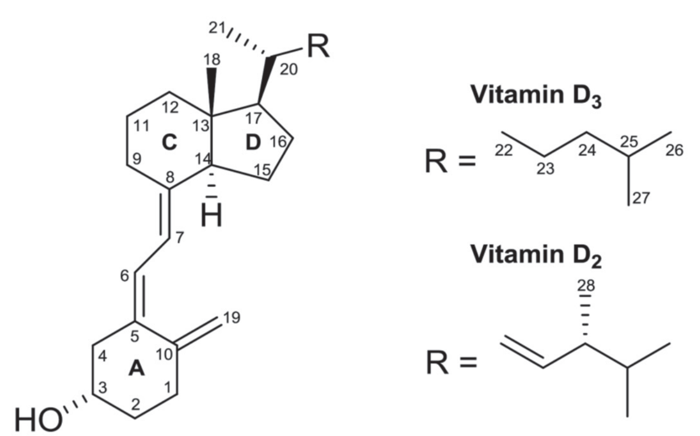


FIG. 1. Structures of vitamin D_3 (cholecalciferol) and vitamin D_2 (ergocalciferol)

International standards for vitamin D analysis often involve saponification, liquid-liquid extraction (LLE), clean-up, LC, and UV/Vis spectroscopy or MS detection. The greatest challenge of vitamin D analysis in foods is the diverse interferences from sample matrices. A large number of lipid-like compounds are co-extracted with vitamin D. Even after extensive sample extract clean-up, there are still numerous interferences that co-elute and interfere with vitamin D quantitation. To simplify sample preparation and improve MS detection, a derivatization reaction using PTAD was adopted as a new standard method [4]. Better analytical performance has been obtained by this new method. However, previtamin D is not measured in this new standard.

Vitamin D can undergo thermal isomerization to reversibly change to previtamin D (Fig. 2), and both forms are biologically active. It has been reported that the relative content of previtamin D could represent as much as 22% of the total vitamin D at 80°C [5]. Therefore, it is prudent to determine directly the previtamin D and the vitamin D contents in foods.

We recently developed such a method that can directly measure the previtamin D and the vitamin D in foods [6]. Details of the method can be found in the application note [6]. To briefly describe the method, the sample is saponified first (75°C for 1 hour), then the analytes are extracted in hexanes. The hexane extract is blow-dried before it is reacted with the PTAD at room temperature for 40 min. The derivatization reaction is then quenched with water and filtered. The filtered solution is injected in an ACQUITY UPLC system, and the analytes are eluted on an ACQUITY UPLC BEH C_{18} 2.1 x 50mm 1.7 um column. The mobile phase A is water, the mobile phase B is acetonitrile (ACN). A 0.1% concentration of formic acid is used as the additive in both mobile phases. The flow rate is set at 0.60 mL/min. The elution program includes an initial hold at 20% mobile phase B for 0.25 min, a linear gradient from 20% B to 100% B in 2.5 min, and a hold at 100% B for 3.75 min. Stable isotope labeled vitamin D_3 (SIL-D_3) is the internal standard (IS). The MS detection is in electrospray mode (positive polarity). There are two multiple reaction monitoring (MRM) transitions for each vitamin D PTAD derivative. The MRM parameters are shown in Table 1. Figure 3 (page 24) shows the typical MRM chromatograms of vitamin D and previtamin D in standard solutions and in sample solutions. Figure 4 (page 24) shows the linear calibration curves that were used in a vitamin D analysis.

There is no pure standard for previtamin D. To quantify the previtamin D, the relative response factors of the vitamin D over the previtamin D in LC-MS were determined in a simple experiment. A solution that contained certain levels of vitamin D_3, vitamin D_2, and SIL-D_3 was split into two portions. One portion was kept in room temperature, while the other portion was heated and maintained at 75°C for 1 hour. Because of the thermal isomerization equilibration, higher previtamin D concentrations and lower vitamin D concentrations were formed in the heated portion than those in the unheated portion. These two portions were analyzed by this method. The relative response factors in LC-MS were obtained by comparing the response differences (peak area differences) in the previtamin D and the vitamin D in the heated and the unheated portions. The relative response factors for the vitamin D_3, the vitamin D_2, and the IS (SIL-D_3) were determined each time the sample was analyzed.

The vitamin D concentration in the National Institute of Standards and Technology (NIST) reference material 1849a was measured by this method. Excellent accuracy (102.6%) and

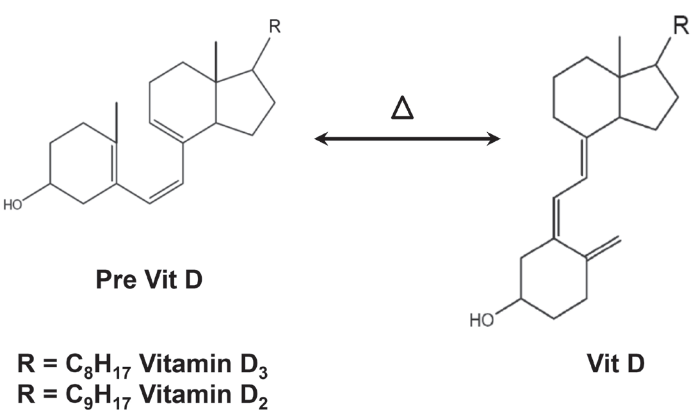


FIG. 2. Reversible thermal isomerization of previtamin D to vitamin D. The equilibration constant and equilibration time change with temperature [5].

TABLE 1. The MRM parameters used in the new LC-MS vitamin D analysis method for foods using PTAD derivatization

	MRM	Dwell (secs)	Cone Volt	Col. Energy	Delay (secs)	Compound	Note
1	560.3>161.0	0.032	43	36	Auto	D3:PTAD	Qualifier
2	560.3>298.1	0.032	43	19	Auto	D3:PTAD	Quantifier
3	560.3>365.3	0.032	43	21	Auto	preD3:PTAD	Qualifier
4	560.3>383.3	0.032	43	13	Auto	preD3:PTAD	Quantifier
5	563.2>301.2	0.032	43	16	Auto	SIL-D3:PTAD	Quantifier
6	563.2>386.3	0.032	43	11	Auto	preSIL-D3:PTAD	Quantifier
7	572.3>311.8	0.032	43	15	Auto	D2:PTAD	Qualifier
8	572.3>377.3	0.032	43	19	Auto	preD2:PTAD	Qualifier
9	572.3>395.3	0.032	43	9	Auto	preD2:PTAD	Quantifier
10	572.3>448.2	0.032	43	9	Auto	D2:PTAD	Quantifier

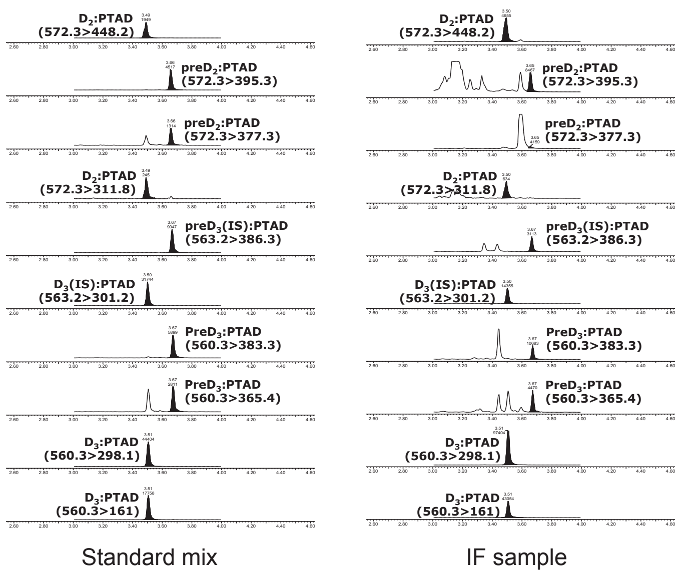


FIG. 3. Typical MRM chromatograms of vitamin D and previtamin D in standard mix solutions and infant formula samples. The vitamin D (RT 3.50 min) is separated from the previtamin D (RT 3.67 min). Column: ACQUITY C18 2.1 x 50 mm, 1.7 µm.

repeatability (2.4% RSD) were obtained. Spiking experiments on an infant formula and an oatmeal sample at two different spiking levels yielded recoveries between 98% and 117%. The limit of quantitation (LOQ) values for the vitamin D_3 and the vitamin D_2 were estimated at 0.01 mg/kg and 0.02 mg/kg in oatmeal, and 0.0003 mg/kg and 0.002 mg/kg in solvent, respectively. These values are comparable to the existing standard.

Besides infant formula and oatmeal, other types of foodstuffs, such as non-fat dry milk powder fortified with vitamin A and vitamin D, soy-based infant formula, chocolate fortified with vitamin D, and fish oil were tested. The vitamin D concentrations determined for the milk and the oatmeal were in agreement with their label vitamin D values (within 9% difference). The vitamin D result for soy-based infant formula was 52% higher than its label value, which was not uncommon in food products. The result for the fortified chocolate was high (70% higher than the label value), and this needed to be further investigated. The fish oil sample's nutrition facts were not available for comparison.

The key improvement in this new LC-MS method is that the previtamin D is detected and quantified along with the vitamin D. The relative content of the previtamin D in the total vitamin D can vary a lot in different brands and kinds of food products due to different manufacturing, transportation, and storage conditions. Without counting the previtamin D concentration in the vitamin D analysis, the results always carry errors originating from the previtamin D. These errors could be huge when accidental situations occur during the manufacturing, transportation or storage of food products. This new LC-MS method ensures that any previtamin D in foods, whether small or large, is counted in the vitamin D analysis. This eliminates the source of error from the previtamin D and improves the accuracy and precision of the vitamin D analysis.

Jinchuan Yang and Gareth Cleland are principal scientists at Waters Corporation, Milford, Massachusetts, USA. Yang can be reached at Jinchuan_Yang@waters.com.

References

Vitamin D Fact Sheet for Health Professionals, National Institutes of Health. Website: https://ods.od.nih.gov/factsheets/VitaminD-HealthProfessional/, visited May 1st, 2017

Holden, J.M. and L.E. Lemar, *Am J Clin Nutr 88*: 551S–553S, 2008

Food labeling: Revision of the nutritional and supplement facts labels, FDA, Federal Register/Vol. 81, No. 103/Friday, May 27, 2016/Rules and Regulations

AOAC Official Method 2016.05, *Official Methods of Analysis of AOAC INTERNATIONAL* (2016) 20th Ed., AOAC International, Gaithersburg, MD, USA

Keverling-Buisman, J.A., K.H. Hanewald, F.J. Mulder, J.R. Roborgh, and K.J. Keunig, *J. Pharm. Sci. 57*: 1326–1329, 1968

Yang, J., G. Cleland, and K. Organtini, Waters Application Note 72000xxxx (in press), Waters Corporation, 2017

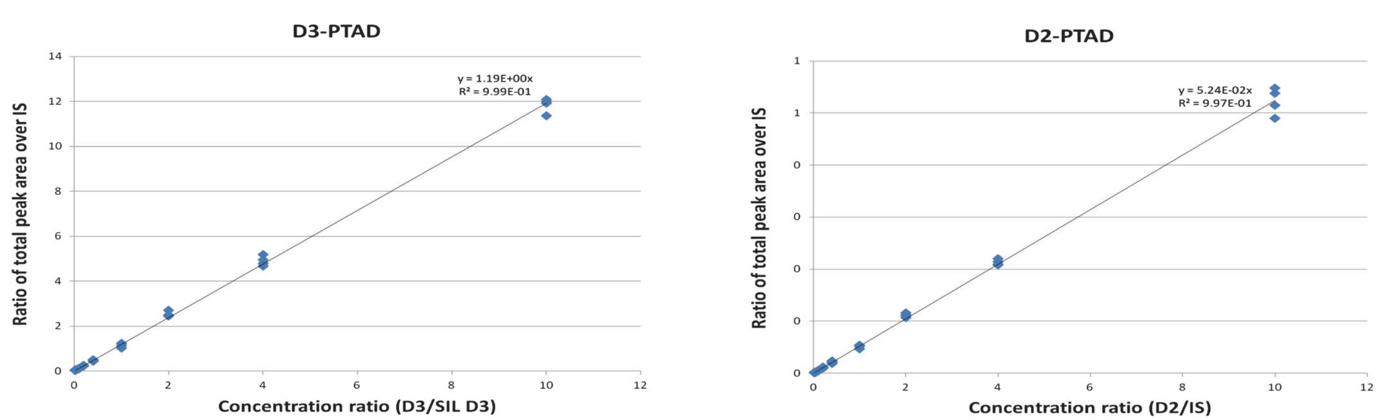


FIG. 4. Calibration plots for vitamin D_3 and vitamin D_2. In these plots, the ratios of the analyte's total vitD peak area over the internal standard's total vitD peak area are plotted against the total vitamin D concentration ratio of analyte over that of the IS. The data were fitted with a linear regress through zero. R2 of 0.999 and 0.997 were obtained for the vitamin D_3 and D_2, respectively.

Developmental programming of the autonomic nervous system: roles for nutrition and inflammation

M.L. Drewery and C.J. Lammi-Keefe

- In this article, we discuss the inspiration behind our current work: investigation of the developing autonomic nervous system's sensitivity to maternal nutrition and inflammation during pregnancy, as evidenced by heart rate (HR) and heart rate variability (HRV) during infancy.
- Specific focus is given to the n-6 and n-3 long chain polyunsaturated fatty acids given the documented contrast in their roles *in vivo*.
- We provide a compelling position for the hypothesis that there is a link between intrauterine exposure to nutrition and inflammation and early life HR/HRV.

The autonomic nervous system contains two arms, the sympathetic and parasympathetic nervous systems, which work antagonistically: The sympathetic nervous system, often referred to as the fight-or-flight response, prepares the body for intense-physical activity, while the parasympathetic nervous system relaxes the body and inhibits or slows high-energy functions so the body can "rest and digest." The interaction of sympathetically and parasympathetically innervated inputs to the heart with the sinoatrial node reflects autonomic activity which can be assessed non-invasively as HR and HRV.

HEART RATE VARIABILITY AS A PROXY OF AUTONOMIC DEVELOPMENT

In early life (fetus, neonate, infant), HR and HRV are developmental expressions of autonomic maturation and reflect cardiac-autonomic integration. HR and HRV are established during fetal life and related to measures through early childhood; the trajectory into adolescence and adulthood has not been assessed, to the best of our knowledge. However, the relationship of fetal and early infancy HR and HRV to childhood HR and HRV supports the hypothesis that these measures in early life have relevance for autonomic functioning in later life.

In general, low HR and high HRV are attributes of healthful cardiac and autonomic function, although there is a threshold beyond which further HR reductions and/or HRV rises would indicate abnormalities. In the current discussion, low HR and high HRV are considered hallmarks of health.

CLINICAL APPLICATIONS OF HEART RATE VARIABILITY

As early as the mid-1960s, clinicians noted that fetal stress was preceded by a reduction in HRV even before HR changes were detected. Today, fetal HR monitoring throughout pregnancy and labor/delivery is standard practice and attributed with lowering morbidity that is associated with fetal distress.

Beyond early life, HRV has clinical relevance for the detection of diabetic neuropathy and assessment of sudden death risk following myocardial infarction (stroke). A "real-life" example of the importance of a healthy, responsive autonomic nervous system lies in the "recovery HR", or the heart's ability to return to a normal rate following physical activity. Physical activity is accompanied by and requires an elevated HR; however, once at rest, HR must return to basal values. An individual with higher HRV is more efficient at lowering HR to normal and, thus, recovers from physical exertion more quickly than an individual with lower HRV.

HEART RATE VARIABILITY AND NUTRITION

Intervention studies indicate that intake of docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA), n-3 LCPUFAs, affects HR and HRV throughout life and in healthy and diseased states (Table 1). Specifically, n-3 LCPUFA supplementation beneficially decreases HR and increases HRV. Autonomic modulation by n-3 LCPUFAs is thought to underlie these observations.

N-3 LCPUFAs increase brain levels of the primary parasympathetic neurotransmitter, acetylcholine, without affecting activity of the acetylcholine-degrading enzyme, resulting in an overall increase in acetylcholine. Further, α-linolenic acid, the n-3 shorter chain precursor of DHA and EPA, induces long-term enhancement of acetylcholine receptors. This effect has not been explored with n-3 LCPUFAs but would presumably mimic that of α-linolenic acid.

The relationship between n-3 LCPUFAs, HR, and HRV in early life mirrors that of adults (Table 2). Those early life-specific studies have investigated the effects of maternal n-3 LCPUFA intervention on fetal HR and HRV and postnatal n-3 LCPUFA intervention on infant HR and HRV. However, the potential for developmental programming of infant HR and HRV by maternal factors, including nutrition status, has yet to be explored. As there is significant fetal autonomic maturation during the third trimester, supply of n-3 LCPUFAs during this time is critical and can exert long-term programming effects. The primary determinant of fetal availability of n-3 LCPUFAs is maternal status with respect to these fatty acids. As humans are inefficient at synthesizing n-3 LCPUFAs from precursor fatty acids, pregnant women must consume dietary or supplemental n-3 LCPUFA to support fetal development.

With this information in hand, we generated the hypothesis that maternal fatty acid status during pregnancy is related to infant HR and HRV. Maternal status with respect to n-3 and n-6 LCPUFA, as well as the n-6:n-3 ratio, are of specific interest as these fatty acids families share an enzymatic pathway and are, thus, metabolically competitive.

HEART RATE VARIABILITY AND INFLAMMATION

The fetal origins hypothesis is rooted in the observation that the intrauterine environment can program infant outcome with lifetime health and developmental consequences. First noted by the physician and epidemiologist David Barker in the early 1990s, it is now widely accepted and investigated in the biomedical community. The premise is that alterations in the fetal environment can result in structural, metabolic, and/or physiological changes, potentially predisposing the fetus to adverse outcome(s) in postnatal life, including type 2 diabetes and cardiovascular disease.

As shown in Table 2, the fetal autonomic nervous system is sensitive to programming by maternal nutrition. Related research indicates maternal exercise during pregnancy affects fetal HR and HRV in a fashion analogous to n-3 LCPUFA intervention. It was hypothesized that the developing autonomic nervous system can be programmed by inflammatory stress and fetal autonomic sensitivity to inflammation may underlie previous observations evidencing a role for maternal factors in programming fetal HR and HR.

It is well established that inflammatory mediators are required for proper central nervous system function during development. However, over-abundances of some pro-inflammatory molecules can be toxic. Thus, prolonged or exaggerated exposure to these molecules during critical developmental periods could alter autonomic outcome.

Early life HR and HRV have not been assessed in relation to intrauterine and/or postnatal exposure to inflammation. However, inflammation-related diseases (i.e., rheumatoid

TABLE 1. Intervention studies showing a benefit for n-3 LCPUFA and HRV[1,2]

Population	Dose	Duration
Coronary artery disease	5.2 g	12 weeks
Dialysis patients	5.2 g	12 weeks
Healthy adults	2.0 g, 6.6 g	12 weeks
Nursing home residents	2.0 g	20 weeks
Infants (2.5 y)	4.5 g	Mother; 20 weeks during lactation
Prison inmates	1.7 g	23 weeks
Overweight adults	6.0 g	12 weeks
Epileptic adults	2.0 g	12 weeks (cross-over)

[1]LCPUFA: long chain polyunsaturated fatty acid; HRV: heart rate variability

[2]"Benefit" defined as increased HRV for intervention versus placebo group; adapted from Christensen *et al.* (2011), *Front Physiol 2*:1-9.

TABLE 2. Intervention studies showing a benefit for n-3 LCPUFA and early life HR/HRV[1,2]

Population	Dose	Duration
Healthy, term infants[3]	924 mg fish oil	9–12 months of age
Healthy term infants[4]	Unsure	Birth to 6 months of age
Healthy term infants[5]	0.32, 0.64, or 0.92% FA as DHA	Birth to 12 months of age
Fetuses[6]	600 mg DHA	Mother; 15 wks gestation–birth

[1]LCPUFA: long chain polyunsaturated fatty acid; HR: heart rate; HRV: heart rate variability; FA: fatty acid: DHA: docosahexaenoic acid
[2]"Benefit" defined as reduced HR and/or increased HRV for intervention versus placebo group
[3]Lauritzen *et al.*, 2008, *Pediatr Res 64*:610-614; [4]Pivik *et al.*, 2009, *Dev Neuropsychol 34*:139-158; [5]Colombo *et al.*, 2011, *Pediatr Res 70*:406-410; [6]Gustafson *et al.*, 2013, *Prostaglandins Leukot Essential Fatty Acids 88*:331-338

arthritis, coronary heart disease, and metabolic syndrome) are characterized by decreased parasympathetic tone and/or reduced HRV. The relationship between circulating pro-inflammatory biomarkers (such as interleukin-6, tumor necrosis factor-α, and C-reactive protein) and HRV, especially in diseased states, has been extensively reported on. In summary, with greater inflammation, pro-inflammatory biomarkers are present in higher concentrations and these are, in turn, related to lower HRV.

Taken together, we generated the hypothesis that maternal inflammatory status during pregnancy is related to infant HR and HRV, where inflammatory status includes "classic biomarkers," such as interleukin-6, tumor necrosis factor-α, and C-reactive protein. We expect inflammation to have a similar effect on early life HR and HRV as in those observational studies discussed above.

CLINICAL TRANSLATION

This work builds upon existing research documenting a role for n-3 LCPUFA in accelerating early life autonomic development while also providing insight into the effects of intrauterine exposure to inflammation on developmental programming. It is hypothesized that beneficial reduction in HR and increase in HRV with maternal and/or postnatal n-3 LCPUFA intervention can be extrapolated to early childhood values, at the very least, indicating a long-term advantage for the infant. Although bold and potentially presumptive, we suggest the alteration of maternal lifestyle factors during pregnancy has lifelong therapeutic potential in the offspring's autonomic and cardiac health.

Merritt Drewery is Nutrition, Quality, and Research supervisor at Grober Nutrition, Auburn, New York, USA. She earned a doctoral degree from the School of Nutrition and Food Sciences at Louisiana State University, Baton Rouge, Louisiana, in August 2017. Her research interests include the role of perinatal nutrition in optimizing fetal and infant development, with a focus on infants born to women with adverse metabolic conditions. Merritt has been involved in AOCS throughout her graduate career; she proposes and co-chairs technical sessions within the Health and Nutrition (H&N) division at annual meetings; and has served as co-chair of the Student Common Interest Group since 2014.

Carol Lammi-Keefe is the Alma Beth Clark Professor in the School of Nutrition and Food Sciences at Louisiana State University, Baton Rouge, Louisiana. She also holds an adjunct position at Pennington Biomedical Research Center, Baton Rouge, Louisiana, and is recognized as a Professor Emeritus at University of Connecticut, Storrs, Connecticut. Lammi-Keefe's research focuses on the mother-infant dyad and the role of maternal omega-3 fatty acid intake in developmental programming. Current research examines the potential programing effect of maternal nutrition on infant body composition in the first year of life. Lammi-Keefe has been heavily involved in AOCS for much of her professional career and continues to contribute to the H&N division as well as serving on the esteemed governing board.

Further reading

1) Barker, D.J., Fetal origins of coronary heart disease, *BMJ 311*: 171–174, 1992.

2) Christensen, J.H., Omega-3 polyunsaturated fatty acids and heart rate variability, *Front. Physiol. 2*: 1–9, 2011.

3) Porges, S.T. and S.A. Furman, The early development of the autonomic nervous system provides a neural platform for social behavior: a polyvagal perspective, *Infant Child Dev. 20*: 106–118, 2011.

4) Gustafson, K.M., S.E. Carlson, and J. Colombo, *et al.*, Effects of docosahexaenoic acid supplementation during pregnancy on fetal heart rate and variability: a randomized clinical trial, *Prostaglandins Leukot. Essential Fatty Acids 88*: 331–338, 2013.

5) Gustafson, K.M. and Popescu, E.A., "Fetal assessment using biomagnetometry: neurobehaviors, cardiac autonomic control, and research applications." In: Reissland, N. and Kisilevsky, B.S. (Eds.), *Fetal Development* (2016) Switzerland: Springer International Publishing, pp. 453–480.

2017–2018 AOCS Approved Chemists

The AOCS Approved Chemist Program recognizes the most accomplished participants in the Laboratory Proficiency Program (LPP). Certification is based on performance during the previous LPP year. Approved Chemists must work in an independent or industrial laboratory, hold AOCS membership in good standing, and establish analytical competency through the LPP. For more information about either program, contact Dawn Shepard at AOCS Technical Services (phone: +1 217-693-4810; fax +1 217-693-4855; email: dawns@aocs.org).

AAK
Louisville, KY 40208 USA
+1 502-548-7238
www.aak.com
James Houghton, Deborah McRoberts, Matt Williams: Edible Fat

Adams Vegetable Oils, Inc.
Arbuckle, CA 95912 USA
+1 530-668-2072
adamsgrp.com
Abdul Bath: Gas Chromatography

Admiral Testing Services, Inc.
Luling, LA 70070 USA
+1 985-785-8302
www.admiraltesting.com
Renato M. Ramos: Aflatoxin in Corn Meal Test Kit, NIOP Fats and Oils, Oilseed Meal, Soybean, Soybean Oil, Unground Soybean Meal

Advance Mycotoxin Laboratory
Wasco, CA 93280
+1 661-758-7790
Steven Dominguez, Elizabeth Sanchez: Aflatoxin in Almond and Pistchio, Aflatoxin in Peanut Paste

Amspec, LLC
Pasadena, TX 77503 USA
+1 713-330-1000
www.amspecllc.com
Mumtaz Haider, Kester Emefina: Aflatoxin in Corn Meal Test Kit, DDGS from Corn Meal, Gas Chromatography, NIOP Fats and Oils, Oilseed Meal, Soybean, Tallow and Grease

Areej Vegetable Oils and Derivatives SAOC
Postal Code 124
Rusayl, Muscat Sultanate of Oman
+00968-99203513
Avod.om
Analytical Team: Gas Chromatography, NIOP Fats and Oils

ATC Scientific
North Little Rock, AR 72114 USA
+1 501-771-4255
atcscientific.com
Michael White, Brian Eskridge: Aflatoxin in Corn Meal Test Kit, Oilseed Meal, Phosphorus in Oil, Soybean Oil, Unground Soybean Meal

Bachoco S.A. de C.V.
Celaya GTO 38024 Mexico
+52-01-461-6143504
Fernando Alberto Leon Cruz: Unground Soybean Meal

Bakels Edible Oils, Ltd.
Mount Maunganui 3116 New Zealand
+64 7 928 0895
www.beobakels.co.nz
Joy Thompson: Gas Chromatography

Barrow-Agee Laboratories, Inc.
Memphis, TN 38116 USA
+1 901-332-1590
www.balabs.com
Michael Hawkins: Oilseed Meal, Unground Soybean Meal, Soybean Oil

Barry Callebaut
Pennsauken, NJ 08110 USA
+1 856-486-9978
Joseph Maher: Edible Fat, Gas Chromagraphy, trans Fatty Acid Content, Solid Fat Content by NMR

Batavia Bio Processing, Ltd.
Batavia, IL 60510 USA
+1 630-326-8596
Christopher Stefan: GOED Nutraceutical Oils

Bayer CropScience
Saskatoon, SK S7K 3J9 Canada
+1 306-477-9443
Rudy Fulawka: Gas Chromatography

Betagro (Public) Company Limited
Samutprakarn 10130 Thailand
+66 2 816 5011
www.betagro.com
Benjaporn Chatchoochaikul: Fishmeal

Blue Diamond Growers
Sacramento, CA 95811 USA
+1 916-329-3311
Jerry Scheeler: Aflatoxin in Almond

CAISA
Asuncion 1827, Departamento Central Paraguay
+515 216888000
Sara Esquivel Candia: Phosphorus in Oil

Callaghan Innovation
Lower Hutt 5010 New Zealand
+64 4 931 3310
www.callaghaninnovation.govt.nz
Andrew MacKenzie, Kirill Lagutin: Marine Oil Fatty Acid Profile

Caloy Company LP
Denair, CA 95316 USA
+1 209-874-3381
www.caloyoil.com
Nicole Silva: Gas Chromatography, Phosphorus in Oil

Campi Alimentos S.A. de C.V.
Merida, Yucatan 97288 Mexico
+52-1- 999-400023
Ana L. Pomar Casares: Unground Soybean Meal

CFIA (Canadian Food Inspection Agency)
Ottawa, ON K1A 0C6 Canada
+1 613-759-1269
Angela Sheridan: Gas Chromatography

Carolina Analytical Services, LLC
Bear Creek, NC 27207 USA
+1 919-837-2021
caslabsllc.com
Brad N. Beavers, Jennie B. Stewart: Oilseed Meal, Unground Soybean Meal

Carribex S.A.
Port -Au-Prince Ouest HT 6210 Haiti
+509-4781-6494
Carl-Henri Cenafils: Palm Oil

Certispec Services, Inc.
Burnaby, BC V3N 4A3 Canada
+1 604-469-9180
www.certispec.com
Cipriano Cruz: NIOP Fats and Oils

Chemiservice SRL
Monopoli, BA 70043 Italy
+39 080 742 777
www.chemiservice.it
Valentina Cardone: Olive Oil Part A, B & C , Olive Oil Sensory Panel Testing

Commodity Inspection Services
Sinnamon Park QLD 4073 Australia
+61 7 3260 1500
www.commodityinspection.com.au
Katrina Saunderson: Tallow and Grease

Cumberland Valley Analytical Services
Waynesboro, PA 17268 USA
+1 301-790-1980
www.foragelab.com
Sharon Weaver: Oilseed Meal

Dallas Group of America
Jeffersonville, IN 47130 USA
+1 812-283-6675
www.dallasgrp.com
Melanie Greer, George Hicks: Vegetable Oil for Color Only, NIOP Fats and Oils

Darling Ingredients, Inc.
Darling Analytical Laboratories
Ankeny, IA 50021 USA
+1 515-289-3718
Shirley Elliott: Tallow and Grease, Gas Chromatography

Diversified Laboratories, Inc.
Chantilly, VA 20151 USA
+1 703-222-8700
www.diversifiedlaboratories.com
Thomas Scott: Tallow and Grease, Gas Chromatography

Eurofins Biodiagnostics
River Falls, WI 54022 USA
+1 715-426-0246
www.eurofinsus.com/biodiagnostics
Joseph Zalusky: Gas Chromatography

Eurofins Central Analytical Laboratory, Inc.
New Orleans, LA 70122 USA
+1 504-297-3420
www.eurofins.com
John Reuther, Derek Yeadon: Aflatoxin in Almond and Pistachio, Aflatoxin in Corn Meal, Aflatoxin in Corn Meal Test Kit, DDGS from Corn Meal, Fish Meal, GOED Nutraceutical Oils, Marine Oil, Marine Oil Fatty Acid Profile, NIOP Fats and Oils, Oilseed Meal, Olive Oil Part A, B & C, Palm Oil, Soybean, Soybean Oil, Trace Metals in Oil, Unground Soybean Meal

Eurofins Scientific
Des Moines, IA 50321 USA
+1 515-265-1461
Ardin Backous: Aflatoxin in Corn Meal Test Kit, Fish Meal, Oilseed Meal, Soybean, Soybean Oil, Unground Soybean Meal
Kent Karsjens: Aflatoxin in Corn Meal Test Kit, Fish Meal, Nutritional Labeling, Oilseed Meal, Soybean, Soybean Oil, Unground Soybean Meal
Keith Persons: Cholesterol, Edible Fat, GOED Nutraceutical Oils, Marine Oil Fatty Acid Profile, Nutritional Labeling, Tallow and Grease, trans Fatty Acid Content
Brian Gilchrist:: Cholesterol, Edible Fat, GOED Nutraceutical Oils, Marine Oil Fatty Acid Profile, Nutritional Labeling, Specialty Oils, Tallow and Grease, trans Fatty Acid Content
Anders Thomsen: Aflatoxin in Corn Meal Test Kit, Cholesterol, DDGS from Corn Meal, Edible Fat, Fish Meal, GOED Nutraceutical Oils, Marine Oil Fatty Acid Profile, Nutritional Labeling, Oilseed Meal, Soybean, Soybean Oil, Specialty Oils, Tallow and Grease, Unground Soybean Meal, Vegetable Oil for Color Only

Fieldale Farms Corp.
Baldwin, GA 30511 USA
+1 706-778-5100
Janet Smith: Oilseed Meal

Fuji Vegetable Oil, Inc.
Savannah, GA 31408 USA
+1 912-433-5331
Gregg Newman: trans Fatty Acid Content, Edible Fat

Hahn Laboratories, Inc.
Columbia, SC 29201 USA
+1 803-799-1614
Frank Hahn: Oilseed Meal, Unground Soybean Meal, Soybean Oil, Aflatoxin in Corn Meal Test Kit

Illinois Crop Improvement Association
Champaign, IL 61822 USA
+1 217-359-4053
Ilcrop.com
Sandra K. Harrison: Oilseed Meal

Imperial Western Products
Coachella, CA 92236 USA
+1 760-275-7122
Joe Boyd: DDGS from Corn Meal

Indelab SDN BHD
Port Klang, Selangor 42000 Malaysia
+603-31676929
Cheah Ping Cheong: Palm Oil

INOLASA
Puntarenas 6651-1000 Costa Rica
+506 2636-0300
www.inolasa.com
Lidieth Solera Carranza: Edible Fat, Unground Soybean Meal, Soybean
Josue Nunez Moya: Unground Soybean Meal, Soybean, Soybean Oil, trans Fatty Acid Content

Inspectorate America
Memphis, TN 38113 USA
+1 901-948-1075
www.inspectorate.com
Sandra Holloway: Aflatoxin in Corn Meal Test Kit

Intertek Agri Services
New Orleans, LA 70122 USA
+1 504-662-1420
Intertek.com
Tuyen Mai: Aflatoxin in Corn Meal Test Kit, DDGS from Corn Meal, NIOP Fats and Oils, Oilseed Meal, Soybean

Inventure Renewables
Tuscaloosa, AL 35401 USA
+1 205-764-1963
www.inventurechem.com
Amy Grano, Heather Watson: Gas Chromatography

Isotek, LLC
Oklahoma City, OK 73127 USA
+1 405-948-8889
www.isoteklabs.com
R. Bruce Kerr, George Ducsay: Tallow and Grease, Oilseed Meal, DDGS from Corn Meal, Gas Chromatography

K-Testing Laboratory
Memphis, TN 38116 USA
+1 901-332-1590
Edgar Tenent: Oilseed Meal

Land O'Lakes
Arden Hills, MN 55112 USA
+1 651-375-2282
www.rtechlabs.com
Julie Honsa: Edible Fat, Gas Chromatography, trans Fatty Acid Content

Lysi hf
Reykjavik 101 Iceland
+354 5258100
www.lysi.com
Arnar Halldorsson: GOED Nutraceutical Oils, Marine Oil, Marine Oil Fatty Acid Profile

Malaysian Palm Oil Board, AOTD
Selangor 43000 Malaysia
+60 3-87694288
www.mpob.gov.my
Ms. Hajar Musa: Palm Oil, Gas Chromatography, trans Fatty Acid Content

Modern Olives Laboratory Services
Lara, VIC 3212 Australia
+61-352729500
www.modernolives.com.au
Claudia Guillaume: Olive Oil Parts A, B & C, Olive Oil Sensory Panel Testing

Modern Olives Laboratory Service
Woodland, CA 95776 USA
+1 530-669-6924
Natalie Ruiz: Olive Oil Part A, Olive Oil Sensory Panel Testing

National Beef
Dodge City, KS 67801 USA
+1 620-338-4250
Mike Clayton: Tallow and Grease

National Beef Packing Company
Liberal, KS 67901 USA
+1 620-626-0646
Sherry Robertson: Tallow and Grease

New Jersey Feed Lab, Inc.
Trenton, NJ 08638 USA
+1 609-882-6800
www.njfl.com
Pete Cartwright: Oilseed Meal, Gas Chromatography, GOED Nutraceutical Oils, Marine Oil, Marine Oil Fatty Acid Profile

NSW Department of Primary Industries
Wagga Wagga, NSW 2650 Australia
+61 02-69381-823
Jamie Ayton: Gas Chromatography, Olive Oil Part A, B &C, Olive Oil Sensory Panel Testing, trans Fatty Acid Content

NSF INASSA S.A.C
Lima 32 Peru
+511-6165200
www.inassagroup.com.pe
Pedro Aguirre Larrain: Fish Meal

Nutreco
St. Hyacinthe, QC J2R 1S5 Canada
+1 450-501-9557
Mathieu D'Amours: Cholesterol, Marine Oil, Nutritional Labeling, Oilseed Meal, Unground Soybean Meal

Omega Protein, Inc.
Houston, TX 77040 USA
+1 713-574-7190
Omegaprotein.com
Holly Edwards: Marine Oil Fatty Acid Profile, GOED Nutraceutical Oils
Juan Li: Marine Oil

Omega Protein, Inc
Reedville, VA 22539 USA
+1 804-453-3830
www.omegaprotein.com
Otelia Robertson, Melissa V. Thrift: Marine Oil

Orkla Health, Division Denomega
Aalesund 6005 Norway
+47 405 44 818
www.denomega.com
Anne Ma Skorpen: GOED Nutraceutical Oils

Owensboro Grain Edible Oils
Owensboro, KY 42301 USA
+1 270-686-6628
***OGEO Lab*:** Soybean Oil, trans Fatty Acid Content

Pilgrims Corp.
Gainesville, GA 30501 USA
+1 770-533-4812
Lisa Marlow: Unground Soybean Meal

Pompeian Inc.
Baltimore, MD 21224 USA
+1 443-573-8006
www.pompeian.com
***Maria Garzon*:** Olive Oil Parts A, B & C, Olive Oil Sensory Panel Testing
Alex Vargo, *Brenda Vest:* Olive Oil Part A, B & C

POS Bio Sciences
Saskatoon, SK S7N 2R4 Canada
+1 306-978-2866
www.pos.ca
***Angie Johnson*:** Cholesterol, GOED Nutraceutical Oils, Marine Oil Fatty Acid Profile, Oilseed Meal, Phosphorus in Oil, trans Fatty Acid Content

PT Musim Mas
Medan, North Sumatra 20371 Indonesia
+62 616871123
www.musimmas.com
***Goh Tiam Huat*:** Gas Chromatography, NIOP Fats and Oils, Phosphorus in Oil, Solid Fat Content by NMR, Trace Metals in Oil, tans Fatty Acid Content

Proteinas Naturales S.A. de C.V.
Guadalupe, Nuevo Leon 67130 Mexico
+81 81960107
www.ragasa.com.mx
Agustin Rodriguez Argüello: Soybean, Soybean Oil, Unground Soybean Meal

Richardson Oilseed
Lethbridge AB T1H 6P5 Canada
+1 403-329-5537
Adel Ghabour: Edible Fat, Gas Chromatography, Trace Metals in Oil, Vegetable Oil for Color Only, Phosphorus in Oil

Richardson Oilseed
Yorkton, SK S3N 2X3 Canada
+1 306-828-2255
www.richardson.ca
Eoin Moloney: Trace Metals in Oil, Phosphorus in Oil

Russell Marine Group–PNW
Portland, OR 97218 USA
+1 503 224 9325
***Robert Carr*:** Oilseed Meal, Soybean

Sanimax–ACI, Inc.
Charny, QC G6X 3R4 Canada
+1 418-832-4645
Sanimax.com
***Jean-Francois Harvey*:** Tallow and Grease

SDK Laboratories
Hutchinson, KS 67501 USA
+1 620-665-5661
www.sdklabs.com
***Dennis Hogan*:** Tallow and Grease, Aflatoxin in Corn Meal Test Kit, DDGS from Corn Meal

Select Supplements, Inc.
Carlsbad, CA 92008 USA
+1-760-431-7509
Sarah Garcia, Martin Johnson: Marine Oil Fatty Acid Profile

Ser–Agro S.A.
Chinandega Nicaragua
+505 2340-3493
***Norma Hernandez*:** Peanut, Aflatoxin in Peanut Paste

Setton Pistacho of Terra Bella
Terra Bella, CA 93270 USA
+1 559-535-6050
www.settonfarms.com
Douglas Lovejoy: Aflatoxin in Pistachio and Almond

SGS Canada, Inc.
Burnaby, BC V5A 4W4 Canada
+1 604-638-2349
www.sgs.com
Cathy Sun: Oilseed Meal

SGS North America
St. Rose, LA 70087 USA
+1 504-471-6489
www.sgs.com
***William Spence*:** Oilseed Meal, Olive Oil Part A, B & C, NIOP Fats and Oils, DDGS from Corn Meal, Aflatoxin In Corn Meal Test Kit

Snyder's–Lance
Columbus, GA 31901 USA
+1 706-649-6350
Adam Powell: Aflatoxin in Peanut Paste Test Kit

Southern Edible Oil Industries (M) Sdn Berhad
Klang, Selangor 42200 Malaysia
+603 32508877
www.southern.com.my
Low Thing: Palm Oil

Stratas Foods–RDI Center
Bartlett, TN 38133 USA
+1 901-387-2237
www.stratasfoods.com
***Eddie L. Baldwin, Helen Cianciolo, Derek Gum*:** Gas Chromatography, trans Fatty Acid Content, Solid Fat Content by NMR, Edible Fat

Team Foods Columbia
Bogatá 1100 Colombia
+77 09000
www.team.co
Elkin Dario Rueda Parra: Gas Chromatography, Specialty Oils

Testing Services (Sabah) Sdn. Bhd.
Sandakan Sabah 90712 Malaysia
+60 89-210431
***Kong Khim Chong*:** Palm Oil

Thai Vegetable Oil
Bangkok, 10600 Thailand
+662 4779020
***Piyanut Boriboonwiggai*:** Unground Soybean Meal, trans Fatty Acid Content

Thionville Laboratories, Inc.
Harahan, LA 70123 USA
+1 504-733-9603
www.thionvillenola.com
Paul C. Thionville, Andre Thionville, Kristopher Williams: Aflatoxin In Corn Meal, Aflatoxin in Corn Meal Test Kit, DDGS from Corn Meal, Fish Meal, Gas Chromatography, Marine Oil, Marine Oil Fatty Acid Profile, NIOP Fats and Oils, Oilseed Meal, Palm Oil, Soybean, Soybean Oil, Tallow and Grease, Trace Metals in Oil, trans Fatty Acid Content, Unground Soybean Meal

University of Georgia
Athens, GA 30605 USA
+1 706-542-9023
www.aesl.ces.vga.edu
Jordan Burbage: Olive Oil Part A & B

University of Missouri–Columbia
Columbia, MO 65211 USA
+1 573-882-2608
***Thomas P. Mawhinney*:** Cholesterol, Gas Chromatography, Nutritional Labeling

University of Stirling
Institute of Aquaculture
Nutrition Analytical Service
Stirling, FK9 4LA United Kingdom
+44 1786 467997
http://www.aqua.stir.ac.uk.nas
Douglas Tocher: Marine Oil Fatty Acid Profile

USDA–ARS
North Carolina State University
Raleigh, NC 27606 USA
+1 919-515-9110
www.ars.usda.gov/southeast-area/raleigh-nc/marketquality
Lisa Dean: Gas Chromatography

Viterra Canola Processing
St. Agathe MB R0G 1Y0 Canada
+1 877-882-2565
www.viterra.com
***Jitendra Patel*:** Oilseed Meal, Gas Chromatography, Trace Metals in Oil, trans Fatty Acid Content, Phosphorus in Oil

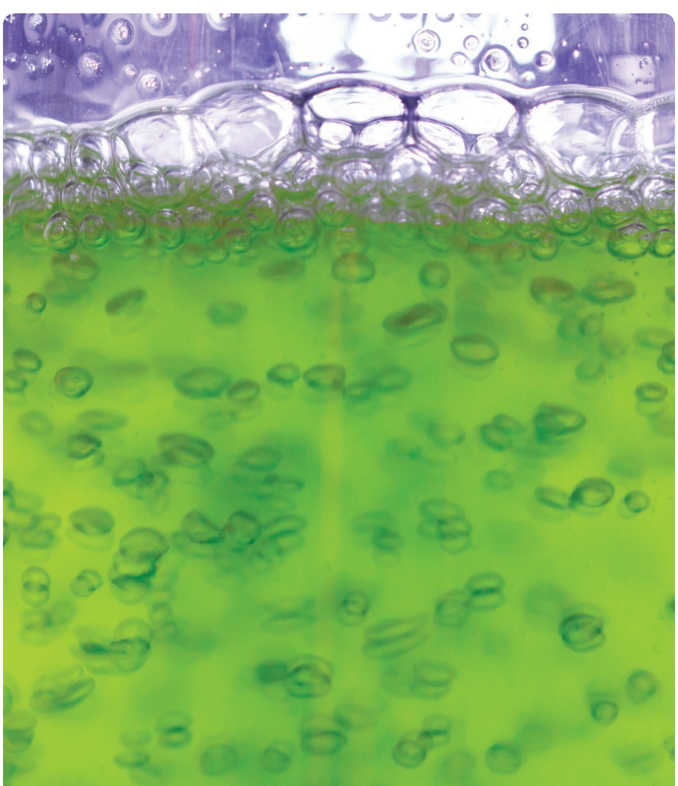
Whitbeck Laboratories, Inc.
Springdale, AR 72764 USA
+1 479-756-9696
www.whitbecklabs.com
***Gordon Whitbeck*:** Tallow and Grease , Unground Soybean Meal, Aflatoxin in Corn Meal Test Kit, Oilseed Meal

Genetically modified algae doubles biofuel yield

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

Laura Cassiday

A few years ago, research into algae-based biofuels was a promising avenue for overcoming a looming shortage of fossil fuels. Then, petroleum prices plummeted, due mainly to improvements in hydraulic fracturing technology, and interest in algal biofuels lagged. Now a *Nature* paper reports that genetic modification of a strain of algae that already produces abundant lipids doubles the potential biofuel yield (Ajjawi, I., *et al.*, http://dx.doi.org/ 10.1038/nbt.3865, 2017). Although the productivity of algal biofuel still has a long way to go to be economically competitive with fossil fuels, this research represents a major step in that direction.



The research arose from a collaboration between oil company ExxonMobil and Synthetic Genomics, the La Jolla, California-based biotech company co-founded by genomics pioneer J. Criag Ventner. In 2009, the two companies teamed up to develop algae-based biofuels. They began with a strain of algae called *Nannochloropsis gaditana*, which can naturally produce and accumulate lipids at up to 60% of its dry weight. Researchers had noticed previously that *N. gaditana* accumulates the highest levels of lipids uder conditions of nitrogen starvation. However, starvation slows the growth of the algae, which in turn decreases productivity.

To find a way to genetically simulate nitrogen starvation without the accompanying decrease in growth rate, the Syntethic Genomics team, led by Imad Ajjawi, used a technique called RNA-Seq to compare the gene expression profile of *N. gaditana* with and without nitrogen starvation. The researchers found that nitrogen starvation induced the differential expression of 1,064 genes. Twenty of these were transciption factors that were downregulated under conditions of nitrogen starvation. The team reasoned that at least some of these downregulated transcription factors may be negative regulators of lipid accumulation in *N. gaditana*. If they could halt or dial down the expression of one or more of these transcription factors, they could perhaps boost biofuel yield in the absence of nitrogen starvation.

So Ajjawi and colleagues used the CRISPR-Cas9 system to disrupt the gene of each transcription factor, creating non-functional knockout (KO) mutants. One of these mutants, in a zinc finger transcription factor called ZnCys, accumulated lipids at three times the level of the wild-type *N. gaditana*, mimicking the lipid accumulation of nitrogen-starved *N. gaditana.* However, like nitrogen-starved *N. gaditana*, growth of the algae was severely inhibited by the ZnCys mutation.

As a result, the researchers decided to genetically reduce ZnCys expression instead of completely knocking out the transcription factor. The team tried different approaches to dialing down the expression of ZnCys in algae. The most successful approach used RNA interference (RNAi) to reduce ZnCys mRNA to about 10% of the wild-type level. The ZnCys-RNAi strain produced double the lipids of wild-type *N. gaditana*, with a similar growth rate.

Next, Ajjawi and colleagues investigated the mechanism behind the increased lipid production of the ZnCys-KO and ZnCys-RNAi strains. These studies revealed that ZnCys appears to be involved in the incorporation of nitrogen into proteins. When ZnCys expression is attenuated, fewer proteins are produced, and more carbon is available for lipid production. "Our findings represent a step toward understanding and controlling lipid production in algae," the authors conclude. "This ability to control algal lipid production might eventually enable the commercialization of microalgal-derived biofuels."

Although the biofuel boost is an important first step, researchers have a long way to go before algal biofuel is economically viable. The ZnCys-RNAi strain produces about 5 g/m^2/d of lipid—an impressive amount for a pilot study. However, experts estimate that a yield of at least 30 g/m^2/d of lipid is required to make the commercialization of algal biofuel feasible (Posewitz, M.C., http://dx.doi.org/10.1038/nbt.3920, 2017). With the recent availabilty of genome-modifiying tools such as CRISPR-Cas9, this yield is now within the realm of possibility.

Olio is produced by Inform*'s associate editor, Laura Cassiday. She can be contacted at laura.cassiday@aocs.org.*

Information

Ajjawi, I., *et al*. (2017) "Lipid production in *Nannochloropsis gaditana* is doubled by decreasing expression of a single transcriptional regulator." *Nature 35*, 647–652. http://dx.doi.org/10.1038/nbt.3865

Posewitz, M. C. (2017) "Algal oil productivity gets a fat bonus." *Nature 35*, 636–638. http://dx.doi.org/10.1038/nbt.3920

SC Johnson reveals 368 potential skin allergens in its products

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

Tammy Lovell

Consumer products conglomerate SC Johnson has disclosed the 368 potential skin allergens that may be used in its products, including household brands such as Glade, Pledge, Scrubbing Bubbles, and Shout.

The international company has added the list of fragrance and non-fragrance ingredients to its transparency website, WhatsInsideSCJohnson.com. It says that by 2018, it will also indicate which skin allergens are contained within each product.

To determine the list of ingredients to disclose, SC Johnson scientists analyzed more than 3,000 data sets from public and industry sources for potential skin allergens identified on country regulatory lists, fragrance industry lists, and individual supplier safety data sheets. This included both natural and synthetic skin allergens.

The company then validated its findings with a panel of experts.

Potential skin allergens down to 0.01% will be disclosed. In a press release, SC Johnson says this is the standard in the European Union and that "the general consensus among the scientific community is that a dose of less than 0.01% is unlikely to cause a reaction for most skin allergens in rinse-off products."

But Kelly Semrau, SC Johnson's senior vice president of global corporate affairs, communication and sustainability, told *Chemical Watch* the company is not planning to reformulate its products to avoid their use because they are "not a safety concern at all."

"We use such a low level of these allergens that they're designed to not induce an allergy, or even elicit an allergy if you've already been exposed."

"This is to inform and educate consumers so they can make a choice," she said.

Semrau emphasized that the skin allergens were also used in similar products by other companies.

"We understand that other companies might choose not to do the work to communicate low levels of potential skin allergens in their products," said Fisk Johnson, chairman and CEO. "With our decades-long commitment to being more and more transparent, we are continuing on a path to provide more and more information to the people who buy our products so they can make choices that are best for them and their families," he added.

RAISING THE BAR

Ken Cook, president and co-founder of Environmental Working Group (EWG), called the move "groundbreaking" and said that SC Johnson was "raising the bar for other companies."

"This level of transparency is sweeping across other industries and is rapidly becoming the new normal for companies, like SC Johnson, who place a premium on giving consumers more, rather than less, ingredient information," he added.

Dev Gowda, toxics advocate for the US Public Interest Research Group (PIRG), called the initiative "a great move for chemical transparency in consumer products."

But he added that while this is a good first step, the company should "take the next step and completely remove skin allergens from its products."

Semrau said this is the latest in a series of SC Johnson transparency initiatives. Later this year there are plans to release a product which is 100% natural and fully disclosed.

And she says the company will reveal the science behind its green list program.

Previously, it published its fragrance palette with a list of its 1,300 approved fragrance ingredients as well as a list of its ingredient restrictions, and then began rolling out product-specific fragrance disclosure. Last year, it launched a product collection with 100% fragrance ingredient transparency, and started its European ingredient transparency program.

Such initiatives are on the rise among consumer goods companies. Earlier this year, Unilever announced plans to provide consumers with information about specific fragrance ingredients used in its personal care products. P&G also announced the launch of a website that allows consumers to see which preservatives are in its products.

There has also been increasing pressure for ingredient transparency in cleaning products in the United States. Bills have been introduced in the US Congress and in California that would require products to bear a label listing ingredients, and New York has floated a proposal for manufacturers to publicly disclose ingredients and identify chemicals of concern.

Tammy Lovell is a business reporter for Chemical Watch.

Nominations are now being accepted for

AOCS Awards recognize individuals and companies who have made outstanding contributions to science, industry, and our Society. With your help, the AOCS awards program can continue to recognize those individuals who are deserving of this honor.

Society Awards

A.R. Baldwin Distinguished Service

This is the Society's highest service award. It recognizes long-term, distinguished service to AOCS in positions of significant responsibility.

Nature of the Award: $2,000 honorarium, $1,500 travel allowance, and a plaque provided by Cargill.

AOCS Award of Merit

This award recognizes an AOCS Member with a history of outstanding service to AOCS. The recipient will display leadership in administrative activities, meritorious service on AOCS committees, or perform an outstanding activity or service that has advanced the Society's prestige, standing, or interests.

Nature of the Award: A plaque.

AOCS Fellow

The status of Fellow is awarded to members of AOCS whose achievements in science entitle them to exceptionally important recognition or to those who have rendered unusually important service to the Society or to the profession.

Nature of the Award: Fellow membership status and a plaque.

Scientific Awards

Supelco AOCS Research

This award recognizes outstanding original research of fats, oils, lipid chemistry, or biochemistry. The recipient must have published the research results in high-quality technical papers regarding fats, oils, lipid chemistry, or biochemistry.

Nature of the Award: $10,000 honorarium, $1,500 travel allowance, and a plaque. The award is sponsored by MilliporeSigma.

Stephen S. Chang

This award recognizes a scientist, technologist, or engineer who has made decisive accomplishments in research for the improvement or development of products related to lipids.

Nature of the Award: $1,500 honorarium and a jade horse, provided by the Stephen and Lucy Chang endowed fund.

AOCS Young Scientist Research

This 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.

Nature of the Award: $1,000 honorarium, $1,500 travel allowance provided by the International Food Science Centre A/S, and a plaque.

Alton E. Bailey

This award recognizes outstanding research and/or exceptional service in the field of lipids and associated products.

Nature of the Award: $750 honorarium and a plaque.

Division Awards

Herbert J. Dutton

The Analytical Division initiated this award to recognize an individual who has made significant contributions to the analysis of fats, oils, and related products or whose work has resulted in major advances in the understanding of processes utilized in the fats and oils industry.

Nature of the Award: $1,000 honorarium, $1,000 travel allowance, and a plaque.

Ching Hou Biotechnology

The Biotechnology Division initiated this award to recognize a scientist, technologist, or leader who has made contributions to the advancement of edible oils, or to the Biotechnology Division's field of interest.

Nature of the Award: $1,000 honorarium and a plaque.

Timothy L. Mounts

The Edible Applications Technology Division initiated this award to recognize research relating to the science and technology of edible oils or derivatives in food products, which may be basic or applied in nature.

Nature of the Award: $750 honorarium and a plaque provided by Bunge North America.

Edible Applications Technology Division Outstanding Achievement

This award recognizes a scientist, technologist, or leader who has made significant contributions to the Division's field of interest, or made contributions to the advancement of edible oils.

Nature of the Award: $500 honorarium and a plaque.

Ralph Holman Lifetime Achievement

The Health and Nutrition Division established this award to recognize an individual who has made significant contributions to the Division's field of interest, or whose work has resulted in major advances in health and nutrition.

Nature of the Award: $500 honorarium, $1,000 travel allowance, and a signed orchid print.

Health and Nutrition Division New Investigator Research

This award recognizes a young scientist who is making significant and substantial research contributions in one

The award recipient must agree to attend the AOCS Annual Meeting & Expo and present an award address. The 2018 AOCS Annual Meeting will be held in Minneapolis, Minnesota, USA from May 6–9, 2018.

the 2018 AOCS Awards Program

Awards

Nomination deadline is November 1, 2017.

of the areas represented by the Health and Nutrition Division of AOCS.

Nature of the Award: $1,000 honorarium and a plaque.

ACI/NBB Glycerine Innovation

The Industrial Oil Products Division initiated this award to recognize outstanding achievement for research in new applications for glycerine with particular emphasis on commercial viability.

Nature of the Award: $5,000 honorarium and a plaque provided by the American Cleaning Institute (ACI) and the National Biodiesel Board (NBB).

Processing Division Distinguished Service

The award recognizes and honors outstanding and meritorious service to the oilseed processing industry.

Nature of the Award: Travel allowance and a certificate.

Samuel Rosen Memorial

Milton Rosen and the Surfactants and Detergents Division initiated this award to recognize a surfactant chemist for significant advancement or application of surfactant chemistry principles.

Nature of the Award: $2,000 honorarium and a plaque.

Surfactants and Detergents Division Distinguished Service

This award recognizes outstanding and commendable service to the surfactants, detergents, and soaps industry.

Nature of the Award: A plaque.

Student Awards

Honored Student

This award recognizes graduate students in any area of fats and lipids. To receive the award, a candidate must remain a registered graduate student and must not have received a graduate degree or have begun career employment prior to the Society's Annual Meeting.

Nature of the Award: $500 travel allowance, complimentary AOCS Annual Meeting registration and lodging, and a certificate.

Ralph H. Potts Memorial Fellowship

This award recognizes a graduate student working in the field of chemistry of fats and oils and their derivatives. Qualifying research will involve fatty acids and their derivatives, such as long-chain alcohols, amines, and other nitrogen compounds.

Nature of the Award: $2,000 honorarium, $500 travel allowance, and a plaque. The award is supported by AkzoNobel, Inc.

Hans Kaunitz

This award recognizes a student doing research in the sciences relating to fats, oils, and detergent technology.

Nature of the Award: $1,000 honorarium, $500 travel allowance, and a certificate.

AOCS Division Awards for Students

These awards recognize students at any institution of higher learning, who are studying and doing research towards an advanced degree in fats, oils, proteins, lipids, surfactants, detergents, and related materials. These student awards are currently being offered by AOCS Divisions.

Nature of the Student Award: Awards can consist of $100 to $1,000 and a certificate.

Nature of the Poster Award: Awards can consist of $100 to $350 and a certificate.

Division	*Due Nov 1, 2017* Student Award	*Due Feb 1, 2018* Poster Award*
Analytical	✓	✓
Biotechnology	✓	
Edible Applications Technology	✓	✓
Health and Nutrition	✓	✓
Industrial Oil Products	✓	✓
Lipid Oxidation and Quality		✓
Processing	✓	
Protein and Co-Products		✓
Surfactants and Detergents	✓	✓

** Poster awards are subject to changes at the discretion of the Divisions.*

Each award has its own specific and unique nomination requirements. Please refer to the website for full details. Nominations must be submitted through our online process and must include all required letters, forms, and references for consideration. Self-nominations are welcomed and encouraged.

AOCS Awards contact ➤ awards@aocs.org • **aocs.org/awards**

Industrial applications of enzymes in Latin America

Leslie Kleiner

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

Enzymes are now widely used in industrial processes, and their applications can be found in many industries related to fats and oils. To learn more about enzymatic technology and applications in Latin America, I interviewed Per Munk Nielsen, Senior Science Manager Oils & Fats, at Novozymes North America Inc.

Q: In Latin America, which are the main industries that use enzymes during processing?

The use of industrial enzymes can be traced back to the late 1800s, and continues to evolve today. Depending on the application, enzymes are used as a processing aid in a variety of Latin American industries, including food and beverage, biofuels, agriculture, and as an active ingredient in certain household cleaners.

Looking specifically at vegetable oil production in Latin America, two primary enzyme classes are currently used. "Phospholipase type C" is used to reduce the P-content to < 200ppm in water degumming, and "phospholipase type A" is used to reduce the P-content to < 5ppm in fully refined oil. These processing aids contribute to operating efficiency by improving process robustness and increasing oil yields. Both types of enzymes have proven successful in full-scale operating conditions, and have material penetration in both water- degummed and deep-degummed oil. This allows enzyme suppliers to continue to introduce higher performing enzymatic solutions.

Other enzymatic applications related to oil processing in Latin America are biodiesel production and enzymatic inter-esterification (EIE). An enzymatic process enables a biodiesel producer to use lower quality and less expensive feedstocks. EIE enzymes provide an alternative solution to harsh chemicals, reducing the generation of unhealthy byproducts.

Q: How are the enzymes used?

Enzymes can be applied in several different forms. Our largest segment is the more common liquid variety, but the industry also offers granulated and immobilized types as well. The enzymes used in oil processing and biodiesel production are liquid-formulated and are normally applied in continuous stirred tank reactors. However, our EIE solution is immobilized.

Q: Which types of lipases are most commonly used in oil processing applications in Argentina and Brazil? In comparison to chemical processes, how much of the market for vegetable oil refining is driven by enzymatic processes?

Phospholipases are primarily used in these types of applications. The portion of the market currently using an enzymatic solution is significant and growing.

Q: In the United States, soybean and canola oil are some of the most common oils. In Argentina and Brazil, corn and sunflower oil are also common. For processing, are the same lipases used to refine soybean oil used to refine sunflower or corn oil?

For some markets, unique enzymatic solutions are developed for a targeted crop. However, the enzyme technology is flexible enough to be used with different types of oils. We have solutions for most major grains produced globally.

Q: What are the differences between lipases used for biodiesel production and those used to produce vegetable oils?

During the production of Biodiesel, our solution (Eversa 2.0) drives an esterification/transesterification reaction on free fatty acids (FFA) and glycerides to produce the fatty acid methyl ester. The Quara product family (phospholipases) serves a different function for vegetable oil degumming. This solution is used to improve the separation of neutral oil from the gums so that more oil can be captured during the process, thereby improving oil yield. In addition, an extra diglyceride or FFA is produced from the reaction. The FFA can then be processed further into biodiesel using our enzymatic solution. Also, for our EIE lipases produce healthier, more stable margarines and shortenings by eliminating the high-temperature step that is otherwise necessary during the interesterification of oil blends.

Q: What are your thoughts on new developments in enzymatic technology?

While the use of enzymes is well over 100 years old, there is still much to be gained through advancing enzyme technology. New applications are being discovered, and existing applications are becoming more productive, robust, and economical. As companies aim to improve sustainability, enzymes are playing a key role in getting more from less and replacing less desirable production practices.

From an operational standpoint, costs for participants in all industries are constantly fluctuating, along with market values of raw materials and end-products. This type of environment creates pressure on manufacturers to operate more efficiently and with more certainty. Many are turning to enzymes to help improve the robustness, stability and profitability of their operations.

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

PATENTS

Metabolic imprinting effects of specifically designed lipid component

Van Der Beek, *et al.*, N.V. Nutricia, US9532966, January 3, 2017

The invention relates to the use of specifically designed lipid component with optimal fatty acid profile; an enhanced portion of the palmitic acid residues in the sn-2 position and present as lipid globules with a phospholipid coating for an early in life diet for improving the development of a healthy body composition, in particular prevention of obesity, later in life.

Low-saturated-fat sunflower and associated methods

Gerdes, *et al.*, Dow AgroSciences LLC, US9538715, January 10, 2017

Provided are sunflowers, parts thereof, cultures of, and seeds that are capable of producing sunflower oil that is low in saturated fat and, optionally, high in linoleic acid as well as associated methods.

Lipid compositions for the treatment of gastrointestinal disorders and the promotion of intestinal development and maturation

Bar Yosef, *et al.*, Enzymotec Ltd., US9561207, February 7, 2017

The present invention provides a use of a lipid composition for the preparation of a nutritional, pharmaceutical, or nutraceutical composition, or a functional food, for the prevention and treatment of gastrointestinal diseases and disorders, and for promoting intestinal development, maturation, adaptation, and differentiation.

Method for the fractionation of phospholipids from phospholipid-containing material

Bruecher, *et al.*, Cargill, Inc., US9567356, February 14, 2017

The present invention relates to a counter-current extraction process involving a plurality of mixing and separation stages for fractionating a phospholipid-containing feed material into two or more fractions enriched in one or more phospholipids, comprising (a) contacting the phospholipid-containing starting material under agitation with an extractant comprising an aliphatic alcohol selected from C1 to C3 alcohols; (b) separating the obtained emulsion into a phospholipid-enriched extract from a residual raffinate.

Cosmetic composition and method for making the same

Constantine , *et al.*, Cosmetic Warriors Ltd., US9561173, February 7, 2017

A solid cosmetic composition includes (i) an outer layer having (a) a hard vegetable butter in an amount of 10 to 45 wt.% based on the outer layer, and (b) a soft vegetable butter in an amount of 55 to 80 wt.% based on the outer layer. The composition also includes an (ii) inner core which is (a) a soft vegetable butter composition; (b) a fondant; or (c) a liquid cosmetic.

Polymer-liposome nanocomposite composition for percutaneous absorption, and method for preparing same

Park, *et al.*, Amorepacific Corp., US9572769, February 21, 2017

Provided are a polymer/liposome nanocomposite composition comprising lipids and poly(amino acids), and a method for preparing same. The polymer/liposome nanocomposite composition has excellent formation stability with respect to surfactants and salts, and can be used in various ways as a drug delivery system in the fields of medicine and cosmetics.

Tropicalizing agent

Whitehouse, Nestec S.A., US9572358, February 21, 2017

Use of particles of insoluble water-absorbing food ingredient, such as dietary fiber, as carrier for a humectant, such as water of glycerol, for the introduction of the humectant into a chocolate product. Furthermore, a tropicalizing agent comprising discrete particles of insoluble food ingredient material, such as citrus fiber particles, loaded with water or humectant. That tropicalizing agent is dispersed in liquid fat, such as cocoa butter. The tropicalizing agent is used to make chocolate products heat-resistant.

Compositions and processes for mesoporous silicas having large pores with narrow diameter distributions

Akolekar, *et al.*, Dow Global Technologies LLC, US9573818, February 21, 2017

A process for preparing a mesoporous silica comprising contacting as starting components (1) a structure-directing template selected from the group consisting of hydrogenated and non-hydrogenated natural vegetable oils, silicone oils, and combinations thereof; (2) water; (3) silica; and, optionally, (4) a structure-directing co-template selected from tetramethylammonium hydroxide, tetraethylammonium hydroxide, tetrapropylammonium hydroxide, cetyltrimethylammonium bromide (CTAB), hexadecyltrimethylammonium chloride; hexadecyltrimethylammonium hydroxide hydrate, hexadecyltrimethylammonium p-toluenesulfonate, hexadecyltrimethylammonium bis-sulfonate, poloxamers having a weight average molecular weight ranging from 5,000 to 20,000 Daltons (Da), and combinations thereof; in the substantial absence of an alcohol solvent; under conditions such that a mesoporous silica having an average pore diameter ranging from 50 to 175 angstroms and a pore diameter distribution that, within one standard deviation of its mean, is substantially unimodal, is formed. The product composition may offer preferred use in, for example, gas separations, sensing, water purification, heavy metal removal, and electronics, where its narrower pore diameter distribution increases its selectivity.

Process for making saturated hydrocarbons from renewable feeds

Wang, *et al*., ExxonMobil Research and Engineering Co., US9574138, February 21, 2017

Provided are processes for making saturated hydrocarbons from renewable feed sources. In an embodiment, a process for producing a lube basestock and/or a diesel fuel from a feedstock of biological origin includes: contacting the feedstock in a single reactor in the presence of hydrogen with catalyst components including a first catalyst and a second catalyst, wherein the first catalyst comprises an acidic material, a basic material, or a combination of both, and wherein the second catalyst is a hydrogenation catalyst including a hydrothermally stable binder.

Biobased biocide compositions and methods of preserving therewith

Frenkel, Galata Chemicals LLC, US9580574, February 28, 2017

Biobased biocide compositions containing epoxidized 2-ethylhexyl soyate and a solvent such as epoxidized soybean oil are disclosed. When incorporated into polymers such as PVC, preserved polymer compositions result. The invention also provides methods for preserving polymers, including incorporating these compositions into PVC in accordance with these methods.

Alkyl glycoside-based micellar thickeners for surfactant systems

Galleguillos, *et al*., Lubrizol Advanced Materials, Inc., US9579272, February 28, 2017

A rheology modifier which includes a mixture of short and long chain fatty acid esters is suitable for use in liquid surfactant-based compositions. A surfactant-based composition includes a surfactant, the rheology modifier, and water. The rheology modifier includes a mixture of alkyl glycoside fatty acid esters including a long-chain fatty acid ester of an alkyl glycoside and a short-chain fatty acid ester of an alkyl glycoside. The long-chain fatty acid ester includes at least one fatty acid residue: R.sup.1(O)O--, wherein R.sup.1 is a C.sub.12 or higher hydrocarbon. The short-chain fatty acid ester includes least one fatty acid residue: R.sup.2(O)O--, wherein R.sup.2 is a C.sub.6-C.sub.10 hydrocarbon.

Lipid and nitrous oxide combination as adjuvant for the enhancement of the efficacy of vaccines

Grobler, *et al*., North-West University, US9585955, March 7, 2017

The invention provides for a method of enhancing immunological responses to an antigen in a vaccine formulation, and for a vaccine formulation that provides for an enhanced immunological response to an antigen. In the method and formulation the antigen is administered with an adjuvant which adjuvant comprises a solution of nitrous oxide gas in a pharmaceutically acceptable carrier solvent for the gas and which adjuvant includes at least one fatty acid or ester or other suitable derivative thereof selected from the group consisting of oleic acid, linoleic acid, alpha-linolenic acid, gamma-linolenic acid, arachidonic acid, eicosapentaenoic acid [C20: 5.omega.3], decosahexaenoic acid [C22: 6.omega.3], ricinoleic acid, and derivatives thereof selected from the group consisting of the C1 to C6 alkyl esters thereof, the glycerol-polyethylene glycol esters thereof, and the reaction product of hydrogenated natural oils composed largely of ricinoleic acid based oils, such as castor oil with ethylene oxide.

Silicone surfactant for use in polyurethane foams prepared using vegetable-oil-based polyols

Heisler, *et al*., Momentive Performance Materials Inc., US9587068, March 7, 2017

Silicone surfactants for use in polyurethane foams prepared using natural-oils-based polyols comprise silicone copolymers possessing alkyl and polyalkylene oxide polyether pendants. The silicone surfactants yield foams having improved physical properties as compared to other surfactant compositions when used in vegetable-oil-based urethane foams.

Thermally stable oil-in-water emulsions containing an oil that contains polyunsaturated fatty acids

Mai, *et al*., DSM IP Assets B.V., US9585837, March 7, 2017

The present invention relates to thermally, stable oil-in-water emulsions comprising an oil comprising a polyunsaturated fatty acid, water, an emulsifier, and a water-soluble stabilizer, and processes for preparing the thermally stable oil-in-water emulsions. The thermally stable oil-in-water emulsions remain flowable at a temperature of -40°C, and are free from a variation in particle size after 9 months in storage at a temperature of -40°C to -15°C.

Patent information is compiled by Scott Bloomer, a registered US patent agent.

Review articles published in AOCS journals during 2017

Journal of the American Oil Chemists' Society

Sustainable synthetic approaches for the preparation of plant oil-based thermosets

Llevot, A., *J. Am. Oil Chem. Soc. 94:* 169–186, 2017, http://dx.doi.org/10.1007/s11746-016-2932-4.

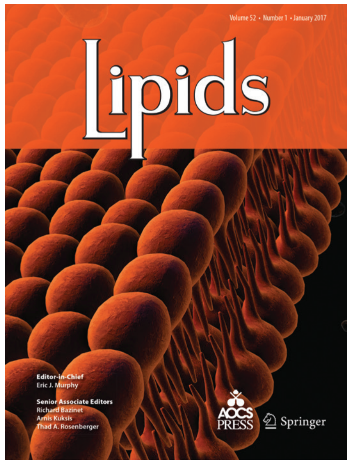
To be in line with the principles of green chemistry, the use of renewables should achieve an overall sustainable process that involves catalytic procedures, comparably less or non-toxic chemicals, and reduction of waste and energy consumption. Plant oil-based thermoset materials offer an advantage with respect to overall sustainability, because they do not require a separation or purification step prior to polymerization. This article reviews the direct homopolymerization and copolymerization of plant oils, and the sustainability of the synthesis of a broad range of thermosets, including epoxy resins, polyurethane networks, polybenzoxazines and unsaturated polyesters.

Biological implications of lipid oxidation products

Vieira, S.A., G. Zhang, and E.A. Decker, *J. Am. Oil Chem. Soc. 94*: 339–351, http://dx.doi.org/10.1007/s11746-017-2958-2.

Lipid oxidation generates potentially toxic products that can enter the body through the diet and develop *in vivo* during the digestion of lipids. Oxidation products can be absorbed into the blood and in some cases transported to tissues. This article reviews how potentially toxic lipid oxidation products are formed and evaluates their potential to impact health. The focus is on the oxidation products that are most reactive and studied: namely acrolein, 4-hydroxy-*trans*-nonenal, 4-hydroxy-*trans*-hexanal, crotonaldehyde, malondialdehyde, and cholesterol.

Lipids



Barth Syndrome: connecting cardiolipin to cardiomyopathy

Ikon, N. and R.O. Ryan, *Lipids 52*: 99–108, 2017, http://dx.doi.org/10.1007/s11745-016-4229-7.

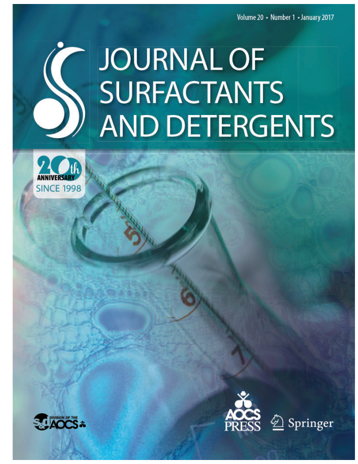
Barth syndrome (BTHS) an inherited metabolic disorder with characteristic phenotypic features that include altered mitochondrial membrane phospholipids, lactic acidosis, organic acid-uria, skeletal muscle weakness, and cardiomyopathy. The underlying has been traced to mutations in the tafazzin (*TAZ*) gene locus on chromosome X. *TAZ* encodes a phospholipid transacylase that promotes cardiolipin acyl chain remodeling. Absence of tafazzin activity results in cardiolipin molecular species heterogeneity and increased levels of monolysocardiolipin and lower cardiolipin abundance. In skeletal muscle and cardiac tissue mitochondria, these alterations in cardiolipin perturb the inner membrane, compromising electron transport chain function and aerobic respiration. The prolonged deficiency in ATP production capacity underlying the cell and tissue pathology is described in detail.

Lipid droplets: formation to breakdown

Meyers, A., T.M. Weiskittel, and P. Dalhaimer, *Lipids 52*: 465–475, 2017, http://dx.doi.org/10.1007/s11745-017-4263-0.

One of the most exciting areas of cell biology during the last decade has been the study of lipid droplets. Lipid droplets allow cells to store non-polar molecules such as neutral lipids in specific compartments where they are sequestered from the aqueous environment of the cell yet can be accessed through regulated mechanisms. These structures are highly conserved, appearing in organisms throughout the phylogenetic tree. Until somewhat recently, lipid droplets were widely regarded as inert. However, progress in the field has continued to demonstrate their vast roles in a number of cellular processes in both mitotic and post-mitotic cells. No doubt the increase in the attention given to lipid droplet research is due to their central role in current pressing human diseases such as obesity, type-2 diabetes, and atherosclerosis. This review provides a mechanistic timeline from neutral lipid synthesis through lipid droplet formation and size augmentation to droplet breakdown.

Journal of Surfactants and Detergents



How to attain ultralow interfacial tension and three-phase behavior with a surfactant formulation for enhanced oil recovery: a review—Part 3. Practical procedures to optimize the laboratory research according to the current state of the art in surfactant mixing

Salager, J.L., A.M. Forgiarini, and M.J. Rondón, *J. Surfact. Deterg. 20*: 3–19, 2017, http://dx.doi.org/10.1007/s11743-016-1883-y.

The minimum interfacial tension to be reached in enhanced oil recovery by surfactant flooding implies the attainment of a so-called optimum formulation. Part 1 of this review showed that this formulation may be described as a numerical correlation between the involved variables defining the oil, the water, the surfactant and the temperature. Part 2 showed that there are many formulation variables and thus too many possibilities to easily choose experimental conditions. Part 3 shows that by cleverly using a three-surfactant mixture, the experimental work to attain a very low interfacial tension for a given reservoir case can be considerably reduced. It is a matter of using the available information along a proper sequential step by step path toward the optimum.

EXTRACTS & DISTILLATES

Comprehensive evaluation of the bactericidal activities of free bile acids in the large intestine of humans and rodents

Watanabe, M., *et al.*, *J. Lipid Res. 58*: 1143–1152, 2017, http://dx.doi.org/10.1194/jlr.M075143.

In addition to functioning as detergents that aid digestion of dietary lipids in the intestine, some bile acids have been shown to exhibit antimicrobial activity. However, detailed information on the bactericidal activities of the diverse molecular species of bile acid in humans and rodents is largely unknown. Here, we investigated the toxicity of 14 typical human and rodent free bile acids (FBAs) by monitoring intracellular pH, membrane integrity, and viability of a human intestinal bacterium, *Bifidobacterium breve* Japan Collection of Microorganisms (JCM) 1192^T, upon exposure to these FBAs. Of all FBAs evaluated, deoxycholic acid (DCA) and chenodeoxycholic acid displayed the highest toxicities. Nine FBAs common to humans and rodents demonstrated that α-hydroxy-type bile acids are more toxic than their oxo-derivatives and β-hydroxy-type epimers. In five rodent-specific FBAs, β-muricholic acid and hyodeoxycholic acid showed comparable toxicities at a level close to DCA. Similar trends were observed for the membrane-damaging effects and bactericidal activities to *Blautia coccoides* JCM 1395^T and *Bacteroides thetaiotaomicron* DSM 2079^T, commonly represented in the human and rodent gut microbiota. These findings will help us to determine the fundamental properties of FBAs and better understand the role of FBAs in the regulation of gut microbiota composition.

Deep or air frying? A comparative study with different vegetable oils

Santos, C.S.P., *et al.*, *Eur. J. Lipid Sci. Technol. 119*: 1600375–1600375, 2017, http://dx.doi.org/ 10.1002/ejlt.201600375.

A comparative study of deep-frying and air-frying was performed, using two commercial air-frying equipment's and four common frying oils–sunflower, soybean, canola, and olive oils. Fried potatoes were compared in terms of color, moisture, lipid composition and degradation indicators, tocopherols, total ascorbic acid, β-carotene, antioxidant activity, acrylamide, and sensory analysis. The results show that fried potatoes obtained by air-frying processes presented an average of 70% less fat, which leads to a reduction of 45 kcal per 100 g. Most chemical parameters were similar on both frying processes, including acrylamide content, or showed slightly better results with air-frying process, namely ascorbic acid amounts. However, incorporated fat was more damaged in deep-fried potatoes, with a significant increase on both *p*-anisidine and polar compounds. Despite the less significant differences observed in the two air-frying equipment's tested, lipid oxidation allowed their distinction, particularly *p*-anisidine that was higher in Airfryer than Actifry, independently of the oil type. All tested oils behaved similarly, and were mainly responsible for potatoes enrichment in tocopherols, phenolics, and β-carotene, but lower lipid oxidation was observed with olive oil. From the assessor's perspective, taste and odor qualities were more determinant for acceptability of fried potatoes than color.

Stability of tocopherol homologs in soybean, corn, canola, and olive oils under different moisture contents at 25°C

Jung, J., *et al.*, *Eur. J. Lipid Sci. Technol. 119*: 1600157–1600157, 2017, http://dx.doi.org/10.1002/ejlt.201600157.

The stability of tocopherol homologs and oxidative stability were determined in edible oils including soybean, corn, canola, and olive oils under different relative humidities (RH), ranging from 0 to 93%, at 25°C for 8 months. The degree of oxidation was determined by conjugated dienoic acid (CDA) and *p*-anisidine values (*p*-AV), and any remaining tocopherols were also analyzed. The stability of tocopherols was significantly influenced by the presence of both moisture and other tocopherol homologs. Soybean and corn oils under low moisture conditions had significant remaining tocopherol levels, whereas canola and olive oils had different stabilities of tocopherols. The presence of δ-tocopherol, which is found in soybean and corn oils, seems to play an important role in the stability of α- and γ-tocopherols in vegetable oils stored at 25°C. The moisture content of the tested oils was different even when oils were stored at the same RH.

Omega-3 fatty acids and risk of preeclampsia

Rani, A., *et al.*, *Lipid Technol. 29*: 47–51, 2017, http://dx.doi.org/10.1002/lite.201700014.

Omega-3 fatty acids are crucial for fetal brain and retina development and are transferred to the fetus from the mother. This transfer of omega-3 fatty acids depends on the development and function of the placenta, which is a connecting organ between the mother and the fetus. However, lower levels of maternal omega-3 fatty acids can alter early developmental processes of the placenta leading to pregnancy disorders such as preeclampsia (PE). Future studies are required to understand the effect of omega-3 fatty acid supplementation on placental oxidative stress and inflammation in women with PE.


Call for Papers

Protective effect of camellia oil (*Camellia oleifera* Abel.) against ethanol-induced acute oxidative injury of the gastric mucosa in mice

Tu, P.-S., *et al.*, *J. Agric. Food Chem.* 65: 4932–4941, 2017, http://dx.doi.org/10.1021/acs.jafc.7b01135.

Camellia oil, a common edible oil in Taiwan and China, has health effects for the gastrointestinal tract in folk medicine, and it contains abundant unsaturated fatty acids and phytochemicals. However, the preventive effect of camellia oil on ethanol-induced gastric ulcers remains unclear. This study was aimed to evaluate the preventive effect of camellia oil on ethanol-induced gastric injury *in vitro* and *in vivo* as well as its mechanisms of action. In an *in vitro* study, our results showed that pretreatment of RGM-1 cells with camellia oil enhanced the migration ability as well as increased heat shock protein expression and reduced apoptotic protein expression. In animal experiments, mice pretreated with camellia oil effectively showed improved ethanol-induced acute injury of the gastric muscosa and oxidative damage through the enhancement of antioxidant enzyme activities and heat shock protein and PGE_2 production, as well as the suppression of lipid peroxidation, apoptosis-related proteins, pro-inflammatory cytokines, and NO production. Histological injury score and hemorrhage score in ethanol-induced gastric mucosal damage dramatically elevated from the control group (0.00 ± 0.0) to 3.40 ± 0.7 and 2.60 ± 0.5, respectively. However, treatments with camellia oil or olive oil (2 mL/kg bw) and lansoprazole (30 mg/kg bw) showed significant decreases in elevation of injury score and hemorrhage score ($p < 0.05$). Therefore, camellia oil has the potential to ameliorate ethanol-induced acute gastric mucosal injury through the inhibition of inflammation and oxidative stress.

Simultaneous silencing of *GhFAD2-1* and *GhFATB* enhances the quality of cottonseed oil with high-oleic acid

Liu, F., *et al.*, *J. Plant Physiol.* 215: 132–139, 2017, https://doi.org/10.1016/j.jplph.2017.06.001.

Cottonseed oil has become an important source of edible oil due to its significant cost advantage. However, there is a growing concern over its fatty acid composition and nutritional value. In *Gossypium hirsutum*, *GhFAD2-1* and *GhFATB* encoding the microsomal oleate desaturase and palmitoyl-acyl carrier protein thioesterase, respectively, play critical roles in regulating the proportions of saturated and polyunsaturated fatty acids in cottonseed lipids. In this study, RNAi technology was used to simultaneously inhibit the expression levels of *GhFAD2-1* and *GhFATB* to improve the quality of cottonseed oil by increasing oleic acid content. Transgenic cotton plants with reduced levels of both target genes were successfully generated. In mature seed kernels of transgenic plants, the content of oleic acid was 38.25%, accordingly increasing by 156.96%, while the content of palmitic acid and linoleic acid was 19.15% and 36.68%, decreasing by 21.28% and 33.92%, respectively, compared with that of the control. The total oil content in transgenic and control kernels was 22.48% and 29.83%, respectively. The reduced oil level in transgenic seeds was accompanied by a reduction in seed index, thereby causing disadvantageous effects on seed germination potentiality and seed vigor, particularly under cool stress conditions. Our results demonstrated the feasibility of simultaneous manipulation of multiple genes using RNAi technology and showed the important role of oil content in seed development and vigor. Our findings provide insight into the physiological significance of the fatty acid composition in cottonseeds.

δ-Tocotrienol suppresses tumorigenesis by inducing apoptosis and blocking the COX-2/PGE2 pathway that stimulates tumor–stromal interactions in colon cancer

Wada, S., *et al.*, *J. Functional Foods* 35: 428–435, 2017, https://doi.org/10.1016/j.jff.2017.06.002.

Anticancer effects of δ-tocotrienol have been reported for several types of cancer, but have not been fully elucidated in colorectal cancer. We investigated the anti-proliferative effect of tocotrienols *in vitro*, in colon epithelial cells and stromal cells, and *in vivo*, in an induced colorectal cancer mouse model. Of the four isoforms tested, δ-tocotrienol exerted the most potent anti-proliferative effect on colon adenocarcinoma cells. δ-Tocotrienol reduced the nitrite and prostaglandin E_2 (PGE_2) concentrations in mouse embryonic fibroblasts (MEFs) pretreated with δ-tocotrienol and stimulated with lipopolysaccharide (LPS) and interferon γ. Furthermore, supernatants of LPS-stimulated MEFs promoted adenocarcinoma cell proliferation, while δ-tocotrienol treatment suppressed this effect. Additionally, a δ-tocotrienol-enriched diet significantly suppressed tumor formation in azoxymethane and dextran sulfate sodium-treated mice. Taken together, these data suggest that a δ-tocotrienol-enriched diet prevents colorectal cancer. At the molecular level, tocotrienols exert a direct anti-proliferative effect on colon adenocarcinoma cells, and an indirect, stromal cell-mediated, anti-proliferative effect.

Emerging roles for conjugated sterols in plants

Albert Ferrer, *et al.*, *Prog. Lipid Res.* 67: 27–37, 2017, https://doi.org/10.1016/j.plipres.2017.06.002.

In plants, sterols are found in free form (free sterols, FSs) and conjugated as steryl esters (SEs), steryl glycosides (SGs), and acyl steryl glycosides (ASGs). Conjugated sterols are ubiquitously found in plants, but their relative contents highly differ among species and their profile may change in response to developmental and environmental cues. SEs play a central role in membrane sterol homeostasis and also represent a storage pool of sterols in particular plant tissues. SGs and ASGs are main components of the plant plasma membrane (PM) that specifically accumulate in lipid rafts, PM microdomains known to mediate many relevant cellular processes. There are increasing evidences supporting the involvement of conjugated sterols in plant stress responses. In spite of this, very little is known about their metabolism. At present, only a limited number of genes encoding enzymes participating in conjugated sterol metabolism have been cloned and characterized in plants. The aim of this review is to update the current knowledge about the tissue and cellular distribution of conjugated sterols in plants and the enzymes involved in their biosynthesis. We also discuss novel aspects on the role of conjugated sterols in plant development and stress responses recently unveiled using forward- and reverse-genetic approaches.

Rapeseed hull oil as a source for phytosterols and their separation by organic solvent nanofiltration

Lammerskötter, A., *et al.*, *Eur. J. Lipid Sci. Technol. 119*: 1600090–1600090, online December 2016, https://doi.org/10.1002/ejlt.201600090.

Hulling of rapeseed leads to hulls as an additional valuable by-product containing remarkable concentrations of phytosterols and tocopherols compared to the concentrations in the entire seed and double-pressed kernel. It was shown that the content of these substances in hull oil is significantly higher than in the entire seed. In particular, the sterol concentration with 3% is remarkably high. Another characteristic of hull oil is the high concentration of a-tocopherol. Therefore, the hull oil can be an interesting source for sterols and tocopherols, which are used as health promoting additives in the food industry. The nanofiltration with organic solvent-resistant membranes was tested for the enrichment of these substances. The experiments were performed in a dead-end cell at pressures up to 20 bar at ambient temperature using *n*-hexane and *n*-heptane as solvents. Fluxes of pure solvent and solutions of triglycerides and sterols were determined. Rejections of sterols and triglycerides were measured in binary (solvent-sterol or solvent-oil) and ternary (solvent-sterol-oil) solute solvent systems. A five-fold increase in sterol concentration in the solvent-free permeate was possible with a single nanofiltration step. After removal of the solvent from the permeate, sterol crystallization was observed. Further investigations have to be done to optimize membrane separation and coupling with a crystallization of sterols.

Green synthesis of conjugated linoleic acids from plant oils using a novel synergistic catalytic system

Zhang, Y., *et al.*, *J. Agric. Food Chem. 65*: 5322–5329, 2017, https://doi.org/10.1021/acs.jafc.7b00846.

A novel and efficient method has been developed for converting plant oil into a specific conjugated linoleic acid (CLA) using a synergistic biocatalytic system based on immobilized *Propionibacterium acnes* isomerase (PAI) and *Rhizopus oryzae* lipase (ROL). PAI exhibited the greatest catalytic activity when immobilized on D301R anion-exchange resin under optimal conditions (PAI dosage of 12 410 U of PAI/g of D301R, glutaraldehyde concentration of 0.4%, and reaction conditions of pH 7.0, 25 °C, and 60 min). Up to 109 g/L *trans*-10,*cis*-12-CLA was obtained after incubation of 200 g/L sunflower oil with PAI (1659 U/g of oil) and ROL (625 mU/g of oil) at pH 7.0 and 35 °C for 36 h; the corresponding conversion ratio of linoleic acid (LA) to CLA was 90.5%. This method exhibited the highest proportion of *trans*-10,*cis*-12-CLA yet reported and is a promising method for large-scale production.

Analysis of lysophospholipid content in low-phytate rice mutants

Tong, C., *et al.*, *J. Agric. Food Chem. 65*: 5435–5441, 2017, https://doi.org/10.1021/acs.jafc.7b01576.

As a fundamental component of nucleic acids, phospholipids, and adenosine triphosphate, phosphorus (P) is critical to all life forms, however, the molecular mechanism of P translocation and distribution in rice grains are still not understood. Here, with the use of five different low phytic acid (*lpa*) rice mutants, the redistribution in the main P-containing compounds in rice grain, phytic acid (PA), lysophospholipid (LPL), and inorganic P (Pi), was investigated. The *lpa* mutants showed a significant decrease in PA and phytate-phosphorus (PA-P) concentration with a concomitant increase in Pi concentration. Moreover, defects in the *OsST* and *OsMIK* genes result in a great reduction of specific LPL components and LPL-phosphorus (LPL-P) contents in rice grain. In contrast, defective *OsMRP5* and *Os2-PGK* genes led to a significant increase in individual LPL components. The effect of the *Os2-PGK* gene on the LPL accumulation was validated using breeding lines derived from a cross between KBNT-*lpa* (*Os2-PGK* mutation) and Jiahe218. This study demonstrates that these rice *lpa* mutants lead to the redistribution of Pi in endosperm and modify LPL biosynthesis. Increase LPLs in the endosperm in the *lpa* mutants may have practical applications in rice breeding to produce "healthier" rice.

The discovery of citral-like thiophenes in fried chicken

Cannon, R.J., *et al.*, *J. Agric. Food Chem. 65*: 5690–5699, 2017, https://doi.org/10.1021/acs.jafc.7b01371.

The isomers of 3,7-dimethyl-2,6-octadienal, more commonly known together as citral, are two of the most notable natural compounds in the flavor and fragrance industry. However, both isomers are inherently unstable, limiting their potential use in various applications. To identify molecules in nature that can impart the fresh lemon character of citral while demonstrating stability under acidic and thermal conditions has been a major challenge and goal for the flavor and fragrance industry. In the study of fried chicken, several alkyl thiophenecarbaldehydes were identified by gas chromatography–mass spectrometry and gas chromatography–olfactometry that provided a similar citral-like aroma. The potential mechanism of formation in fried chicken is discussed. Furthermore, in order to explore the organoleptic properties of this structural backbone, a total of 35 thiophenecarbaldehyde derivatives were synthesized or purchased for evaluation by odor and taste. Certain organoleptic trends were observed as the length of the alkyl or alkenyl chain increased or when the chain was moved to different positions on the thiophene backbone. The 3-substituted alkyl thiophenecarbaldehydes, specifically 3-butyl-2-thiophenecarbaldehyde and 3-(3-methylbut-2-en-1-yl)-2-thiophenecarbaldehyde, exhibited strong citrus and citral-like notes. Several alkyl thiophenecarbaldehydes were tested in high acid stability trials (4°C vs 38°C) and outperformed citral both in terms of maintaining freshness over time and minimizing off-notes. Additional measurements were completed to calculate the odor thresholds for a select group of thiophenecarbaldehydes, which were found to be between 4.7–215.0 ng/L in air.

Characterization of the saffron derivative crocetin as an inhibitor of human lactate dehydrogenase 5 in the antiglycolytic approach against cancer

Granchi, C., *et al.*, *J. Agric. Food Chem. 65*: 5639–5649, 2017, https://doi.org/10.1021/acs.jafc.7b01668.

Inhibition of lactate dehydrogenase (LDH) represents an innovative approach to tackle cancer because this peculiar glycolytic metabolism is characteristic of most invasive tumor cells. An investigation into the biological properties of saffron extracts led to the discover of their LDH-inhibition properties. In particular, the most important saffron components, crocetin, was found to inhibit LDH (IC_{50} = 54.9 ± 4.7 μM). This carotenoid was independently produced by chemical synthesis, and its LDH-inhibition properties manifested via its antiproliferative activity against two glycolytic cancer cell lines (A549 and HeLa, IC_{50} = 114.0 ± 8.0 and 113.0 ± 11.1 μM, respectively). The results described in this article suggest that saffron may be a helpful alimentary component in the prevention of cancer that potentially contributes to the efficacy of approved cancer therapies.

Industrial Applications

Twin-screw extrusion technology for vegetable oil extraction: a review

Uitterhaegen, E. and P. Evon, *J. Food Eng. 212*: 190–200, 2017, https://doi.org/10.1016/j.jfoodeng.2017.06.006.

Vegetable oils present a valuable class of bioresources with applications in both food and non-food industries and a production that has been steadily increasing over the past 20 years. Their extraction from oilseeds is a key process, as it exerts a strong impact on the resulting oil characteristics and quality. In view of the recent pressure towards sustainability, oilseed processing industries are taking renewed interest in thermomechanical pressing as a means to obtain high-quality oils. This work focuses on twin-screw extrusion for vegetable oil extraction and reviews recent technological advancements and research challenges for the design and optimization of novel oil extraction processes. It comprises a critical analysis of the application of twin-screw extruders against their more conventional single-screw counterparts. Further, a comprehensive overview of the key parameters influencing the process performance is provided, while considerable attention is given to the development of innovative green extraction processes using twin-screw extrusion.

Hydrotreatment of vegetable oils: a review of the technologies and its developments for jet biofuel production

Vásquez, M.C., *Biomass Bioenergy 105*: 197–206, 2017, https://doi.org/10.1016/j.biombioe.2017.07.008.

Hydroprocessing of oils and fats has been a subject of extended research works and discussions over time. It has proved to be an effective pathway for processing vegetable oils into biofuels, especially in the aviation industry. This study presents an evaluated review of recent literature about development, conversion routes, and role of processing conditions to maximize the production of renewable jet fuel. Reaction temperature and acidic strength of the catalyst had greater influence on the composition of final products. Decarboxylation and decarbonylation reactions are dominant during the production of aviation biofuel, because they are preferred over technological alternatives at higher temperatures. Nickel immobilized on a moderately acidic support and palladium on activated carbon catalysts has shown better yields of kerosene, under mild conditions. Continued and systematic efforts need to be made mainly over catalyst design to establish optimum and effective hydrotreating and hydrocracking processing alternative. Wide ranges of feedstocks have been studied for the production to jet biofuel. Jatropha and Camelina are promising options because they are crops for degraded soils; having in mind that in addition to sustainability and availability, costs is a main driver, and feedstock represents from 60 to 75% of final cost. Current initiatives and companies boosting jet biofuels production are also discussed.