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AOCS

International News on Fats, Oils,
and Related Materials

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from**

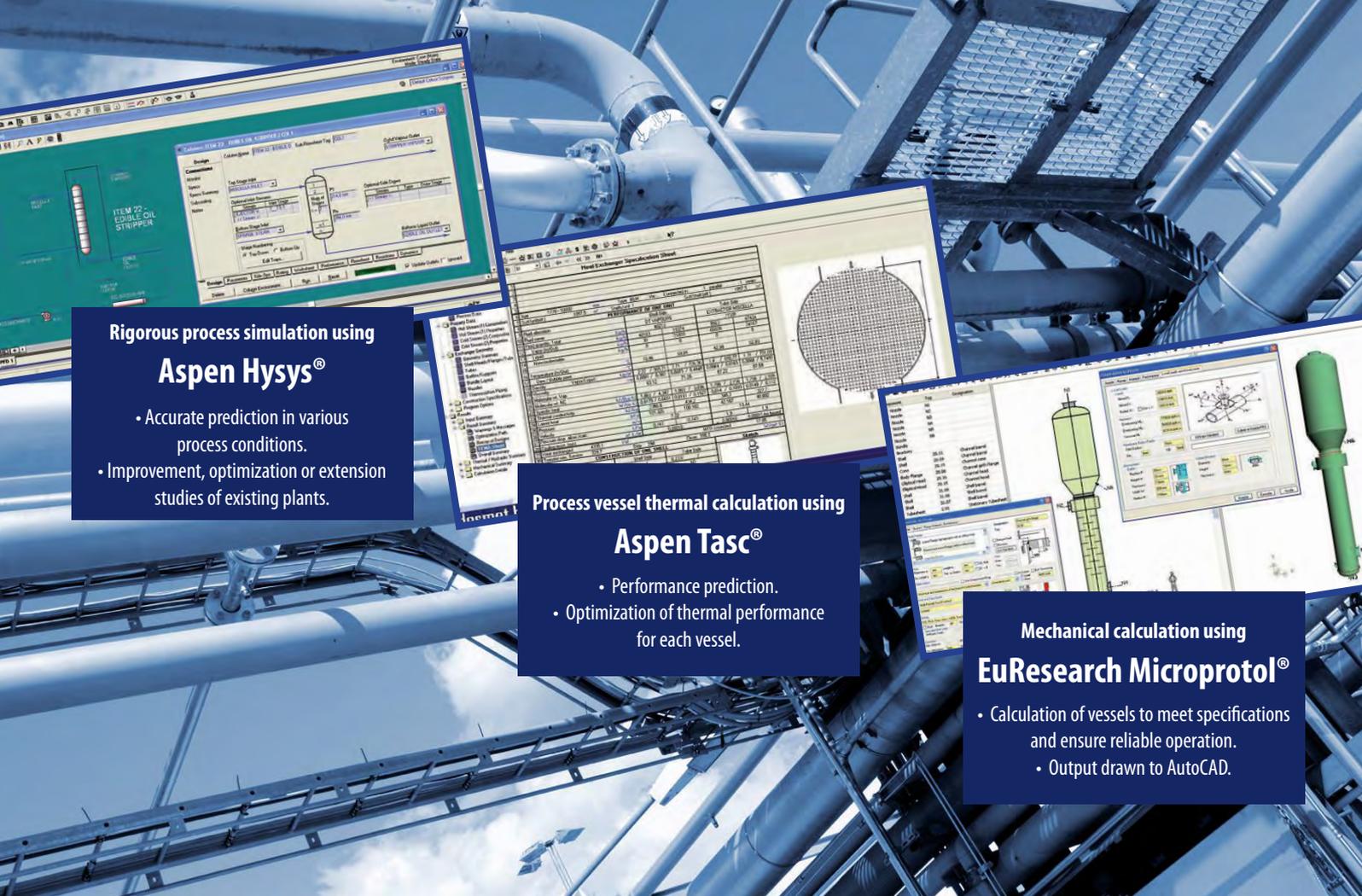
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ALSO INSIDE

"Super phos" esters

Animal-free toxicity testing

Benchtop magnetic resonance



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632 A novel green catalytic process for biodiesel production from jatropha

Learn how biodiesel could be produced from crude jatropha oil on an industrial scale by using an environmentally green direct single-step heterogeneous-catalyzed process.

637 Safety without suffering: advances in animal-free toxicity testing

Many countries, government agencies, and international bodies have committed to finding alternatives to animal testing, but eliminating such testing without compromising product safety remains a challenge for the personal care industry.

643 "Super phos" esters: the key to higher-performance products

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646 "Tiger in the tank"

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651 Making the most of algal biomass with pyrolysis

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655 The America Invents Act: Groundbreaking US patent law changes are here

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661 Regulation of dietary supplements

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665 Science in the media: explaining science to nonscientists

A novel writing exercise teaches graduate students how to explain science to nonscientists.

668 The growth of biobased metalworking fluids

To keep up with the growing demand for metalworking fluids (MWF) made from renewable resources, formulators are developing biobased products that, in many cases, perform as well as or better than conventional MWF at a comparable price.

674 2013 AOCS Annual Meeting & Expo: springtime in Montréal

The 104th AOCS Annual Meeting & Expo will be held in Montréal, Québec, Canada. Get ready to enjoy the city's centuries-old charm, freewheeling spirit, culture of cool, and more than 6,000 gastronomic venues.

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

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Calendar

December

December 4–5, 2012. European Advanced Biofuels Congress, Brussels, Belgium. Information: www.greenpowerconferences.com

December 4–5, 2012. International Algae Conference, Berlin, Germany. Information: <http://algaecongress.com>

December 4–5, 2012. 12th Practical Short Course: Trends and Markets in Aquaculture Feed Ingredients, Nutrition, Formulation and Optimized Feed Production and Quality Management, Ghent, Belgium. Information: www.smartshortcourses.com

December 4–5, 2012. Labeling Requirements and Implications for Foods Marketed in the United States, Arlington, Virginia, USA. Information: www.ift.org/meetings-and-events/short-courses.aspx

December 5, 2012. Cosmetic Raw Materials, New York, New York, USA. Information: www.sconline.org

December 5–7, 2012. Algae Technology Europe, Ghent, Belgium. Information: www.smartshortcourses.com

December 6–9, 2012. World Congress on Clinical Lipidology, Budapest, Hungary. Information: www.clinical-lipidology.com

January 2013

January 6–11, 2013. Carotenoids, Gordon Research Conference, Ventura, California, USA. Information: www.grc.org

January 12–13, 2013. Global Castorworld 2013, Jaipur, India. Information: <http://jatrophaworld.org>

January 27–February 1, 2013. Plant Lipids: Structure, Metabolism & Function, Galveston, Texas, USA. Information: www.grc.org

January 28–February 2, 2013. American Cleaning Institute Annual Meeting & Industry Convention, Orlando, Florida, USA. Information: www.cleaninginstitute.org

February 2013

February 3–8, 2013. Feeds and Pet Food Extrusion, College Station, Texas, USA. Information: <http://foodprotein.tamu.edu>

February 13–15, 2013. 4th Algae Technology Platform and Technical Short Course—Americas, Kailua-Kona, Hawaii, USA. Information: www.smartshortcourses.com

February 20–22, 2013. 7th International Symposium on Deep Frying, San Francisco, California, USA. Information: <http://www.eurofedlipid.org/meetings/sanfrancisco2013/index.php#expo>

February 21–22, 2013. US Department of Agriculture Outlook Forum, Arlington, Virginia, USA. Information: usda.gov/oce/forum

February 28–March 1, 2013. HPCI (Home and Personal Care Ingredients) Exhibition & Conference, Bombay, India. Information: www.hpci-congress.com

February 28–March 2, 2013. Commodity Classic, Kissimmee, Florida, USA. Information: <http://www.commodityclassic.com>

March 2013

March 5–8, 2013. 2013 DEUEL Conference on Lipids, Napa Valley, California, USA. Information: www.deuelconference.org

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AOCS Meeting Watch

April 3–5, 2013. AOCS Oils and Fats World Market Update 2013, Ukrainian House, Kiev, Ukraine. Information: email: meetings@aocs.org; phone: +1 217-693-4821; fax: +1 217-693-4865; <http://worldmarket.aocs.org>

April 28–May 1, 2013. 104th AOCS Annual Meeting & Expo, Palais des congrès de Montréal, Montréal, Québec, Canada. Information: email: meetings@aocs.org; phone: +1 217-693-4821; fax: +1 217-693-4865; <http://AnnualMeeting.aocs.org>

August 20–23, 2013. 15th Latin American Congress and Exposition on Fats and Oils, Sheraton Santiago Hotel and Convention Center, Santiago, Chile. Information: email: meetings@aocs.org; phone: +1 217-693-4821; fax: +1 217-693-4865; <http://aocs.org/meetings>

For in-depth details on these and other upcoming meetings, visit <http://aocs.org/meetings>. New AOCS meetings in this box and below are indicated in boldface type.

March 6–7, 2013. Mass Spec 2013: New Horizons in MS Hyphenated Techniques and Analyses, Biopolis, Singapore. Information: www.sepscience.com

March 10–15, 2013. Snack Food Processing: Extruded Snacks and Tortilla Chips, College Station, Texas, USA. Information: <http://foodprotein.tamu.edu>

March 10–12, 2013. Annual National Institute of Oilseed Products (NIOP) Convention, Scottsdale, Arizona, USA. Information: www.niop.org

March 12–14, 2013. World Biofuels Markets Congress & Exhibition, Rotterdam, Netherlands. Information: www.worldbiofuels-markets.com

March 14–15, 2013. Canola Council of Canada Annual Convention, Vancouver, British Columbia, Canada. Information: email: fyi@canolainfo.org

March 17–19, 2013. 6th Workshop on Fats and Oils as Renewable Feedstocks for the Chemical Industry, Karlsruhe, Germany.

May 4–7, 2014. 105th AOCS Annual Meeting & Expo, The Henry B. Gonzalez Convention Center, San Antonio, Texas, USA. Information: phone: +1 217-693-4821; fax: +1 217-693-4865; email: meetings@aocs.org; <http://aocs.org/meetings>

October 6–9, 2014. Montreux 2014: World Conference on Fabric and Home Care, Montreux Music & Convention Centre, Montreux, Switzerland. Information: email: meetings@aocs.org; phone: +1 217-693-4821; fax: +1 217-693-4865; <http://Montreux.aocs.org>

May 3–6, 2015. 106th AOCS Annual Meeting & Expo, Rosen Shingle Creek, Orlando, Florida, USA. Information: phone: +1 217-693-4821; fax: +1 217-693-4865; email: meetings@aocs.org; <http://aocs.org/meetings>

Information: <http://abiosus.org/kit-workshop-2013.html>

March 17–22, 2013. Pittcon 2013, New Orleans, Louisiana, USA. Information: <http://pittcon.org>

March 20–21, 2013. Leipzig Symposium: Rapeseed—Tremendous Potential for added Value Generation?, Leipzig, Germany. Information: www.eurofedlipid.org

April 2013

April 3–5, 2013. AOCS Oils and Fats World Market Update 2013, Ukrainian House, Kiev, Ukraine. Information: email: meetings@aocs.org; phone: +1 217-693-4821; fax: +1 217-693-4865; <http://worldmarket.aocs.org>

April 7–9, 2013. 8th China International High-End Healthy Edible Oil & Olive Oil Exposition, Beijing, China. Information: www.oilexpo.com.cn

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April 7–11, 2013. 245th American Chemical Society National Meeting & Exposition, New Orleans, Louisiana, USA. Information: www.acs.org

April 9–10, 2013. OFI Middle East 2013, Cairo, Egypt. Information: www.ofievents.com/middle-east

April 9–10, 2013. European Biomass to Power, London, UK. Information: www.wplgroup.com/aci/conferences/eu-ebp3.asp

April 10–12, 2013. IESD 2013—The 14th China International Exhibition on Surfactant & Detergent, Shanghai, China. Information: <http://tinyurl.com/IESD-2013>

April 14–18, 2013. Membrane and Separations Technologies, College Station, Texas, USA. Information: <http://food-protein.tamu.edu>

April 16–18, 2013. New York International Olive Oil Competition, New York City, USA. Information: <http://nyoliveoil.com>

April 28–30, 2013. Food Hydrocolloid Conference, Charleston, South Carolina, USA. Information: www.hydrocolloid.com

April 28–May 1, 2013. 104th AOCS Annual Meeting & Expo, Palais des congrès de Montréal, Montréal, Québec, Canada. Information: www.phone: +1 217-693-4821; fax: +1 217-693-4865; email: meetings@aoacs.org; http://AnnualMeeting.aoacs.org

May 2013

May 5–9, 2013. Trends in Margarine and Shortening Manufacture, Non-*trans* Products, Texas A&M University, College Station, Texas, USA. Information: <http://foodprotein.tamu.edu>

May 21–24, 2013. 2nd International Symposium on Green Chemistry: Renewable Carbon and Eco-Efficient Processes, La Rochelle, France. Information: <http://isgc2.conference.univ-poitiers.fr>

May 29–31, 2013. 11th Yeast Lipid Conference, Halifax, Canada. Information: www.yeastlipidconference.tugraz.at/Future.htm

June 2013

June 10–12, 2013. 9th World Surfactant Congress and Business Convention, Barcelona, Spain. Information: www.cesio-congress.eu

July 13–17, 2013. Institute of Food Technologists' Annual Meeting and Expo, McCormick Place, Chicago, Illinois, USA. Information: www.ift.org

June 16–18, 2013. 119th International Oil Mill Supervisors Association Summer Convention, Denver, Colorado, USA. Information: Linda Paukert (phone: +1 817-297-4668; email: paukert.linda@sbcglobal.net)

June 17–19, 2013. 27th Nordic Lipidforum Symposium, Helsinki, Finland. Information: www.lipidforum.info

August 2013

August 5–9, 2013. 36th Annual Short Course: Advances in Emulsion Polymerization, Davos, Switzerland. Information: www.davoscourse.com

August 20–23, 2013. 15th Latin American Congress and Exposition on Fats and Oils, Sheraton Santiago Hotel and Convention Center, Santiago, Chile. Information: email: meetings@aoacs.org ; phone: +1 217-693-4821; fax: +1 217-693-4865; http://aoacs.org/meetings

September 2013

September 8–12, 2013. 246th American Chemical Society National Meeting & Exposition, Indianapolis, Indiana, USA. Information: www.acs.org

September 17–21, 2013. 54th International Conference on the Bioscience of Lipids, Bari, Italy. Information: www.icbl.unibe.ch/index.php?id=81

September 17–18, 2013. 10th Oilseed & Oil Processing Short Course, Munich, Germany. Information: www.smartshortcourses.com

September 18–20, 2013. oils+fats 2013, Munich, Germany. Information: www.oils-and-fats.com

October 2013

October 9–10, 2013. American Fats & Oils Association Annual Meeting, New York, New York, USA. Information: www.americanfat-sandoilsassociation.com

October 27–30, 2013. 11th Euro Fed Lipid Congress and 30th ISF Lectureship Series, Antalya, Turkey. Information: www.euro-fedlipid.org

October 30–November 1, 2013. International Federation of Societies of Cosmetic Chemists International Conference 2013: Boosting Cosmetic Science in Rio, Rio de Janeiro, Brazil. Information: www.ifsc2013.com

2014

March 16–20, 2014. American Chemical Society National Meeting & Exposition, Dallas, Texas, USA. Information: acs.org

May 4–7, 2014. 105th AOCS Annual Meeting & Expo, The Henry B. Gonzalez Convention Center, San Antonio, Texas, USA. Information: phone: +1 217-693-4821; fax: +1 217-693-4865; email: meetings@aoacs.org; http://aoacs.org/meetings

May 11–15, 2014. International Symposium on High Performance Liquid Phase Separations and Related Techniques, New Orleans, Louisiana, USA. Information: www.HPLC2014.org

June 21–24, 2014. Institute of Food Technologists' Annual Meeting & Expo, New Orleans, USA. Information: ift.org.

October 6–9, 2014. Montreux 2014: World Conference on Fabric and Home Care, Montreux Music & Convention Centre, Montreux, Switzerland. Information: email: meetings@aoacs.org ; phone: +1 217-693-4821; fax: +1 217-693-4865; http://Montreux.aoacs.org ■

Letter from the president

One of the key strengths of AOCs is that there are virtually no limits on the things members can do and the ways in which they can become involved. The broad range of opportunities that are open to each and every one of us was recently highlighted in the AOCs You Can 2012 video (http://media.aocs.org/index.cfm?t=1&v=GhvheX_rax4&#tab1), and if you attended the annual business meeting in Long Beach, California, you may still have those “You Can,” magnets on your refrigerator reminding you that, as a member of AOCs, you can present your research, publish your work, connect with colleagues, serve on a committee, author a book, develop a new method, participate in an expert panel, and the like.

I have personally benefited from such opportunities for more than 20 years, but in my role as AOCs president, I now spend less time thinking about what I can do as an individual member and more time thinking about what “we can” accomplish together. For, as we continue to face major challenges such as the global economy, the environment and sustainability, diet and health, product safety and quality, and the identification of new chemical feedstocks, our opportunities as individual members increasingly depend on what we can do together—on whether we can continue to attract the best and brightest scientists, engineers, and technologists; provide them with opportunities to share their knowledge and to learn from others; and nurture the young professionals who will be our leaders tomorrow. Consequently, the issue most critical to AOCs’ future is: Can we do what it takes?

In 2012, we not only discovered that we can, but we did. Working together, we created a content-focused platform for our Annual Meeting and Expo (AM&E) that will continue to attract the best and brightest minds. We made deep changes in our organizational structure that will streamline decision making and improve our ability to identify and pursue emerging opportunities, as we continued to reach into new markets and expand our services to meet the needs of our members around the world.

A better Annual Meeting & Expo (AM&E)

The Annual Meeting & Expo in Long Beach, California, USA, was reformulated to provide more content and to appeal to a broader range of professionals. We upgraded the scope of our technical presentations, increased our focus on emerging science by replacing the former hot topic sessions with five forum sessions that focused on emerging topics likely to impact all of us, added an executive fast track that made it easier for busy business executives who are not scientists to attend key sessions and networking opportunities, revised the schedule to enhance the flow of information throughout the meeting, moved the annual business meeting from breakfast to lunch, and created a separate Awards Plenary Session that made it possible for more attendees to hear our scientific award winners present their outstanding original research.



We were warned that members who were familiar with the old meeting format would decide not to attend the meeting, but that did not happen. All told, 1,624 people from 49 countries attended the AM&E, which featured 440 oral presentations, 242 poster presentations, 25 forum presentations on emerging topics that prompted vigorous discussions about how these critical issues impact the business of fats and oils, and more than 85 companies exhibiting the latest equipment, products, and services in a wide range of areas.

A nimbler organizational structure

While we were in Long Beach, we presented a new organizational structure to the current committees that will allow AOCs to act more strategically, decisively, and quickly as key issues and new opportunities arise.

The new structure will allow more members to share their knowledge and expertise and become meaningfully involved in developing AOCs strategy by serving on one of three Value Center committees: Content, Networking, and Technical Services. These volunteer committees, each of which will involve six to eight members,

CONTINUED ON NEXT PAGE

will provide the high-level thinking and strategic vision needed to help AOCS identify emerging trends and opportunities. In some cases, they may form *ad hoc* committees of experts to more thoroughly investigate areas related to their expertise. During the past several months, we have been actively recruiting members to serve on these committees. The majority of our invitations have been accepted, and the committees are already starting to move forward with their visioning process.

While the Value Centers will provide the vision, it will be up to AOCS staff to determine the best way to implement the visions that come out of each center. Their guidance on tactical matters will then provide a streamlined Governing Board with the information it needs to make timely and optimal decisions.

A growing global presence

Our influence continues to grow globally. Today, over a third of our members live outside the continent of North America, which is why it is more important than ever

that we continue to reach out to new markets, strengthen our relationships, and expand the services we offer to our members, prospective members, and partners across the globe.

In September, AOCS CEO Patrick Donnelly represented AOCS at the 10th Household Auto Care International Seminar & Exhibition in São Paulo, Brazil, where he spoke on the sustainable use of resources and technologies. His visit grew out of a partnership in which AOCS promoted the seminar outside Latin America, and the seminar's sponsor, *H&C (Household & Cosméticos)* magazine, promoted Singapore 2012 within Latin America. The partnership and Pat's speech were unique opportunities to reach into the growing surfactant and detergent markets within Latin America, where we were previously relatively unknown.

At the end of September, I traveled to Nagasaki, Japan, to celebrate the 60th anniversary of our sister organization, JOCS. I was there for the opening of JOCS' World Congress on Oleo Science, September 30–October 4, 2012, and I had the pleasure to present JOCS with an anniversary gift from

AOCS: a vase with a fine line black starburst design and distinctive border of feathers that was made by a Native American artist from the pueblo of Acoma in New Mexico, USA.

In an effort that grew out of our last Ismir Conference and World Market Update, we made plans to host our first meeting in Kiev, Ukraine. Ukraine is a rapidly growing oilseed market, and this meeting represents our first venture into a CIS country. Meanwhile, we have established a formal partnership with an agribusiness consulting agency in the CIS countries that will help us strengthen our outreach there on an ongoing basis, and we are working with our counterparts in India to host a joint seminar with OTAI next August.

AOCS will continue to build on these and other initiatives during 2013. I thank you for your membership in 2012 and hope you will continue to be a part of AOCS in 2013 and during the exciting years that lie ahead of us.

Deland Myers
AOCS President 2012–2013

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February 20–22, 2013, San Francisco, CA/USA



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www.eurofedlipid.org/meetings/sanfrancisco2013

A novel green catalytic process for biodiesel production from JATROPHA

Aijaz Baig and Flora T.T. Ng

The high cost of edible feedstocks, an increasing demand for food worldwide, and concerns about using virgin forest and arable land for large-scale biodiesel production have thrown considerable attention on nonedible oils, such as jatropha, as attractive alternative feedstocks (Fig. 1). Unfortunately, many nonedible oils have a high content of free fatty acids (FFA), which significantly reduce biodiesel yields during conventional homogeneous base-catalyzed transesterification reactions.

Crude jatropha oil can have an FFA content of up to 15%, which is beyond the acceptable limit for processing using a conventional base-catalyzed process. This limitation can be overcome by using a two-step process involving an acid-catalyzed esterification followed by a base-catalyzed transesterification (Baig, 2003). However, this two-step (or multi-step) process increases the system complexity and raises the cost of producing biodiesel from a particular feedstock (Olutoye and Hameed, 2011)

Using a conventional homogeneous-catalyzed process (Fig. 2, page 634) to produce biodiesel from crude jatropha oil is technically, economically, and environmentally more challenging than using the same process to make biodiesel from edible oils. It requires multi-step processing, oil pretreatment, neutralization of the waste homogeneous catalyst, water washing of the crude biodiesel and glycerol, and treatment of the waste generated—all of which make the purification of the biodiesel to meet biodiesel quality standards more difficult (Baig and Ng, 2011; Baig *et al.*, 2012).

Most of the processes reported in the literature on biodiesel production from jatropha focus on using conventional homogeneous base-catalyzed processes, two-step or multi-step homogeneous-catalyzed processes, heterogeneous base-catalyzed processes, or heterogeneous acid and base catalyzed processes (Juan *et al.*, 2011; Endalew *et al.*, 2011). However, all these processes are complex and too inefficient to be considered for industrial-scale production of biodiesel. Furthermore,

1st GENERATION FEEDSTOCK

Soybean Oil
Rapeseed Oil
Sunflower Oil
Palm Oil
Canola Oil



DISADVANTAGES

- Edible Oils
- Food Vs Fuel
- Expensive
- Limited Availability
- Required Fertilize Land

2nd GENERATION FEEDSTOCK

Yellow Grease/Waste Oil
Jatropha Oil
Animal Fat
Trap / Brown Grease
Plant waste Biomass / Aquatic Biomass



ADVANTAGES

- Non – Edible Oils
- Inexpensive
- Abundant Availability
- Marginal Lands
- Virtually Grow Anywhere

FIG. 1. Edible (first-generation) vs. nonedible (second-generation) feedstocks.

all the reported processes to date require the use of pre-treated jatropha oil, not crude jatropha oil. The price of crude jatropha oil is much lower than refined and deodorized jatropha oil, which has an FFA content of more than 1% (Juan *et al.*, 2011). To produce biodiesel on an industrial scale, one should use crude jatropha oil.

The problems associated with using a homogeneous-catalyzed process to make biodiesel from feedstock with high FFA content have been addressed by using a heterogeneous-catalyzed process for the production of biodiesel from oil containing FFA (Baig and Ng, 2010). Recently, we developed a novel technology using a simple and environmentally green direct single-step heterogeneous-catalyzed process to produce high-quality biodiesel from crude jatropha oil as shown schematically in Figure 3 (page 635).

In contrast to a conventional homogeneous-catalyzed process, this catalytic technology does not require complex downstream washing and separation processes, and the heterogeneous catalyst can be recycled and is environmentally benign. This technology provides a direct route for the synthesis of biodiesel from crude jatropha oil. Furthermore, the heterogeneous catalysts are also potentially inexpensive. Heterogeneous catalysts can be customized so that the presence of FFA or water does not adversely affect the catalytic

Biodiesel is defined by the American Society for Testing and Materials (ASTM) as the mono alkyl ester of long-chain fatty acids derived from a renewable lipid feedstock (Baig and Ng, 2010).

Globally, the availability of feedstocks for biodiesel production varies considerably according to location and climate. The important factors to be considered in the selection of biodiesel feedstocks are (i) the chemical composition of the fat or oil, (ii) its cost and availability, and (iii) transport and pretreatment. The chemical composition is important to determine the amount of free fatty acids in the oil, which is an important factor to be considered in the selection of biodiesel production technologies for industrial-scale applications (Olutoye *et al.*, 2011).

Currently, edible oils are widely used for biodiesel production. In Europe rapeseed oil is mainly used, while in Malaysia and Indonesia palm oil predominates. In the United States, soybean oil and animal fats are primary feedstocks. Among the nonedible oils, jatropha is considered one of the most advantageous feedstocks for biodiesel production in terms of economical, sociological, and environmental implications (Juan *et al.*, 2011).

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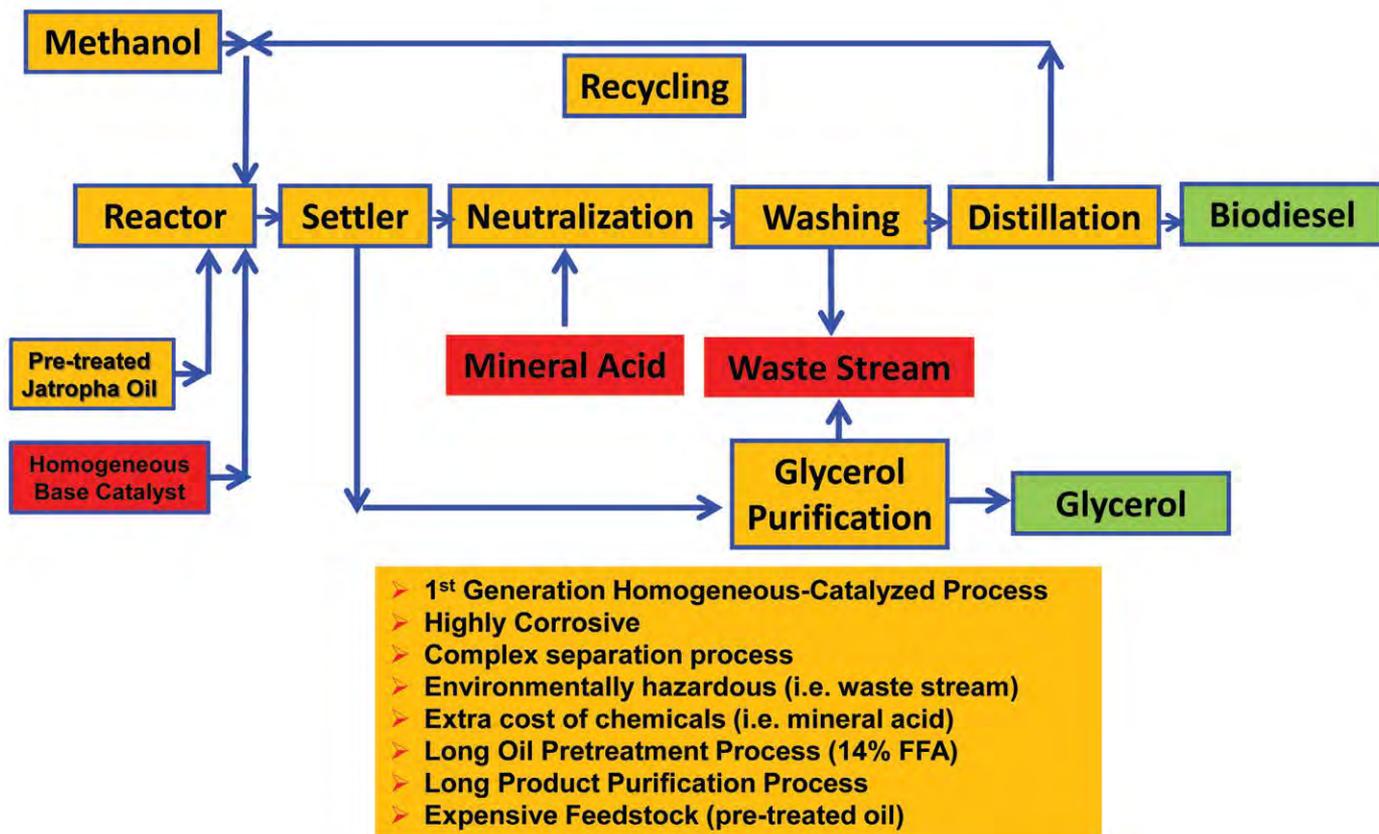


FIG. 2. Conventional first-generation homogeneous base-catalyzed process. FFA, free fatty acids.

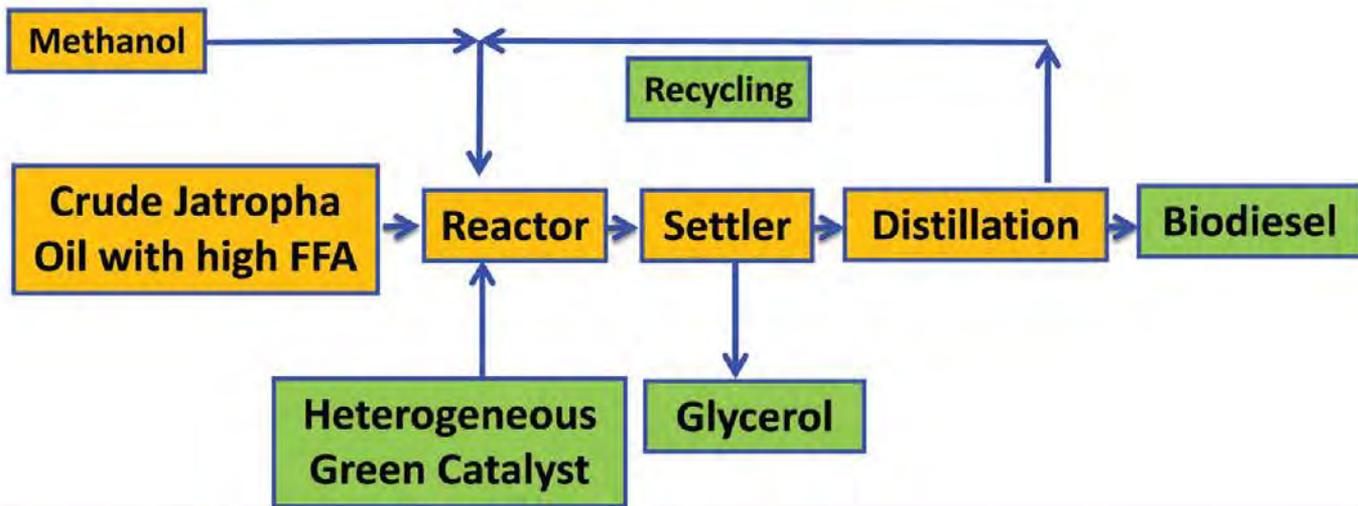
activity during and after the biodiesel production process. Consequently, this novel catalytic technology could be used to produce biodiesel from crude jatropha oil on an industrial scale.

Aijaz Baig received his M.A.Sc. in chemical engineering (specialization in biodiesel) and a collaborative graduate program in environmental engineering from the University of Toronto, Canada. His doctoral work in the department of chemical engineering at the University of Waterloo, Canada, focused on the development of novel green second-generation biodiesel technologies for industrial applications. Baig is a leading inventor of innovative green technologies for industrial-scale production of biodiesel. He has been awarded the professional designation of Chartered Chemist (C. Chem.) from the Association of the Chemical Profession of Ontario, Canada and the distinguished Waterloo Institute of Nanotechnology Fellowship. He is the author of over 30 scientific publications and presentations at national and international conferences, and is an active member of the AOCS, the American Chemical Society, the Chemical Institute of Canada, and the Canadian Society for Chemical Engineering. His research interests are focused on catalysis, innovative biofuels and bioprocess technologies, green chemistry and engineering, sustainable energy, biofuels quality, and nanotechnology. He can be contacted at a6baig@uwaterloo.ca.

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FIG. 3. Novel green catalytic technology for the production of biodiesel from crude jatropha oil.

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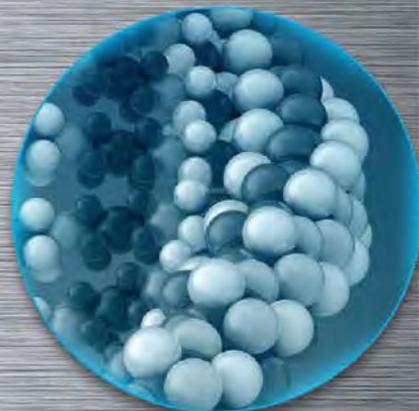
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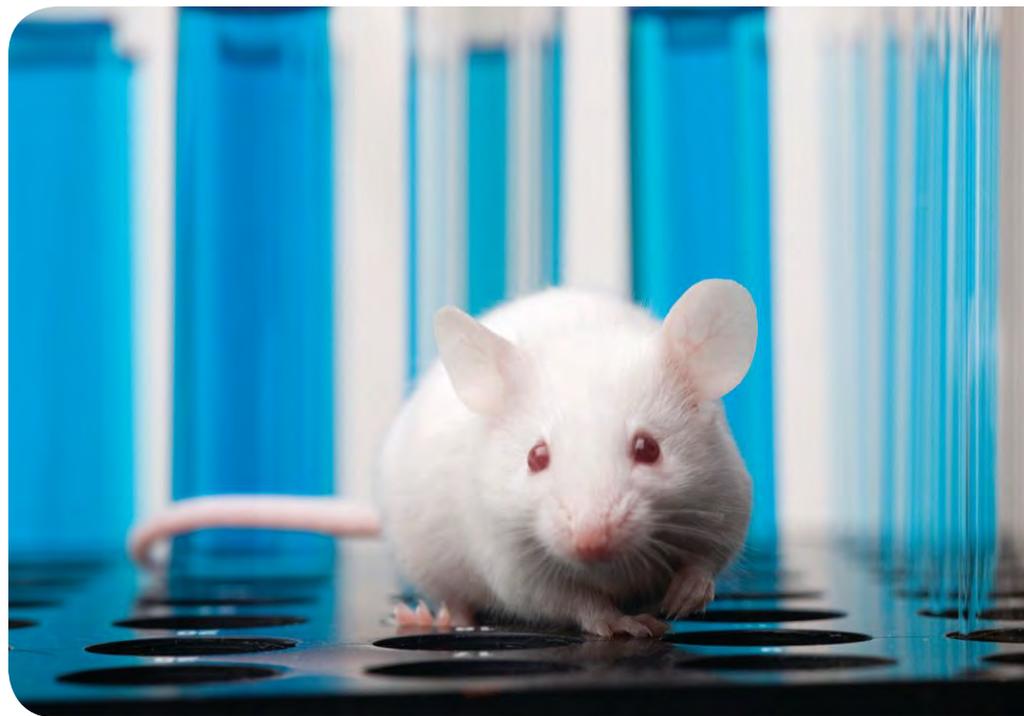
Safety without suffering: advances in animal-free toxicity testing

Laura Cassiday

The thought of mice, rabbits, or dogs languishing in experimental labs tugs at the heartstrings of animal lovers. Yet most people recognize that animal testing is essential to establish the safety of products such as drugs, shampoos, cosmetics, and household cleaners. Increasingly, however, government agencies and manufacturers worldwide are embracing the 3 Rs: *replacing* animal tests with alternatives, *reducing* the number of animals used, and *refining* methods so that animals experience less pain and distress. In the process, they are discovering that alternative tests not only allay ethical concerns but in the long run may prove superior to whole-animal tests for predicting adverse effects in humans.

People have been using animals for scientific experimentation since at least the time of the ancient Greeks. In the fourth century BCE, Aristotle and Erasistratus were among the first to document experiments on living animals. Later, in the second century CE, the Roman physician Galen dissected living and dead animals to study their anatomy, earning him the title “father of vivisection.” But it wasn’t until the late 1800s, with the advent of the synthetic chemical industry, that animal testing began in earnest. Suddenly, researchers needed to understand how tens of thousands of new substances could affect human health.

Scientists reasoned that the anatomical similarities between lab animals and humans made them good models for predicting the effects of chemicals on people. Moreover, rats, mice, and other small mammals are easy to breed and have short life cycles, accelerating the analysis of chronic and reproductive



effects. In 1927, British pharmacologist J.W. Trevan proposed the median lethal dose, or LD_{50} test to determine the dose of a chemical that kills half of the animals exposed to it. The LD_{50} remains a popular metric for comparing chemical toxicities.

Other common ways to quantify a chemical’s toxicity are the no observable effect level (NOEL) and the lowest observed effect level (LOEL). The NOEL refers to the highest dose of a substance that has no significant adverse effects, whereas the LOEL is the lowest dose that causes any adverse effects. Scientists at the US Environmental Protection Agency (EPA) have recognized the potential pitfalls of extrapolating animal data to humans: To calculate the maximum acceptable dose for humans of a toxic substance, EPA scientists divide the NOEL by a 10-fold “uncertainty factor” to account for the fact that humans may be more sensitive to the chemical than other animals.

Interspecies variability has led some people to question the reliability of animal tests. Although many biochemical pathways

are conserved among mammals, species-specific differences in genetics, metabolism, and environmental conditions could cause drastically different responses to chemicals. In addition, animals are often treated with massive doses of test substances that far exceed realistic human or environmental exposure levels.

For example, in 1981 the US National Toxicology Program (NTP) listed the artificial sweetener saccharin as a suspected human carcinogen. This decision was based primarily on studies in which rats developed bladder cancer after consuming a daily dose of saccharin equivalent to that of a person drinking about 1,000 cans of diet soda per day (American Council on Science and Health, *Of Mice and Mandates: Animal Experiments, Human Cancer Risk, and Regulatory Policies*, 1997). After subsequent animal and human population-based studies failed to confirm a link between saccharin and cancer, the NTP removed the sweetener from its *Report on Carcinogens*, 9th Edition, in 2000.

CONTINUED ON PAGE 639

TABLE 1. International toxicity testing^a

Country or federation	Key regulating agencies	Animal testing requirements	Key animal welfare legislation
United States	<p>Food and Drug Administration (FDA)—regulates food additives, pharmaceuticals.</p> <p>Environmental Protection Agency (EPA)—regulates commercial chemicals, home care products, pesticides.</p> <p>Interagency Coordinating Committee on the Validation of Alternative Methods (ICCVAM)—recommends valid alternative toxicological tests for US regulatory and research agencies.</p>	US law requires specific animal tests to establish the safety of food additives, drugs, chemicals, home care products, and pesticides. Personal care and cosmetic products have no specific testing requirements, but manufacturers are legally responsible for ensuring product safety.	Animal Welfare Act (1966) —establishes minimum standards for animal care and use; requires researchers to consider alternatives to animal testing for distressful or painful procedures.
European Union (EU)	<p>Directorate General for Enterprise—regulates pharmaceuticals, chemicals, cosmetics.</p> <p>Directorate General for the Environment—regulates pesticides, protects laboratory animals.</p> <p>European Center for the Validation of Alternative Methods (ECVAM)—validates alternative test methods at the EU level.</p>	Various EU Directorates require animal testing data to establish product safety. Since 2009, animal testing on cosmetic products has been prohibited, with some exemptions until 2013.	<p>Directive 86/609/EEC (1986)—regulates use of animals for scientific purposes.</p> <p>7th Amendment to EU Cosmetics Directive (2003)—creates deadlines for banning animal testing of finished cosmetic products and their raw ingredients. Also bans the sale of animal-tested cosmetics produced outside the EU.</p>
Japan	<p>National Institute of Health Sciences (NIHS)—regulates pharmaceuticals, foods, and chemicals.</p> <p>Japanese Center for the Validation of Alternative Methods (JaCVM)—validates alternative test methods for use in Japan.</p>	Animal testing is required for drugs, foods, chemicals, and “quasi-drug” cosmetics such as skin-lightening products, suntan lotion, and hair growth products.	Law for the Humane Treatment and Management of Animals (1973) —regulates animal use and testing in Japan.
International	Organisation for Economic Cooperation and Development (OECD) —internationally harmonizes policies on a broad range of topics, including toxicological testing. The 34 member countries include European nations, the United States, Canada, Mexico, and Japan.	Results from non-animal toxicity tests validated by a member country in accordance with OECD guidelines are accepted by other OECD member countries. Individual countries and/or agencies decide whether a particular test is suitable for their needs.	N/A

^aSource: AltTox.org.

In general, animal testing methods have not kept pace with technological innovation. In a February 25, 2011, editorial in *Science* magazine, US Food and Drug Administration (FDA) Commissioner Margaret Hamburg wrote: “Most of the toxicology tools used for regulatory assessment rely on high-dose animal studies and default extrapolation procedures and have remained relatively unchanged for decades, despite the scientific revolutions of the past half-century” (*Science* 2011, DOI:10.1126/science.1204432).

New technologies such as bioinformatics, proteomics, systems biology, and metabolomics (the study of metabolic responses to drugs, environmental changes, and diseases) offer an unprecedented ability to understand, on a molecular level, how chemicals interact with biology. In addition, researchers can now use robotics to conduct *in vitro* tests to achieve rapid, automated testing of many more samples than would be possible for live animals. Tissue engineering provides increasingly relevant models of skin and other tissues. Furthermore, advances in computational toxicology have spawned new *in silico*, or computer-based, assessment methods.

In contrast, animal testing is not very efficient or economical, says Jack Fowle, recently retired deputy director for the Health Effects Division in the EPA’s Office of Pesticide Programs. “To register a single pesticide, you are probably killing thousands of rodents, spending millions of dollars, and taking four to five years,” he says. In addition to this inefficiency, “It is heartbreaking to think of subjecting animals to these chemicals,” says Fowle. “So from an ethical standpoint but also from economic, informative, and efficiency perspectives, one would like to move away from animal tests.”

In 2007, the US National Research Council issued a report, “Toxicity Testing in the Twenty-first Century: A Vision and a Strategy,” that called for a paradigm shift in toxicology that would “rely less heavily on animal studies and instead focus on *in vitro* methods that evaluate chemicals’ effects on biological processes.” Such an approach would generate data more relevant to human risk assessment, expand the number of chemicals that could be scrutinized, and be faster and more cost effective than animal testing, the report argues.

“There is a major shift going on, and a lot of interest not just by industry, but by regulators, to pursue these new methodologies,” says Francis Kruszewski, director of health

TABLE 2. Examples of nonanimal toxicity tests^a

Measured end point	Method	Description	Validation
Endocrine disruption	Estrogen receptor-alpha transcriptional activation assay	Substances that activate estrogen receptors in cultured human cells drive transcription of a chemiluminescent reporter gene.	OECD US EPA
Eye irritation and corrosion	Bovine corneal opacity and permeability assay	Researchers treat cow eyes obtained from slaughterhouses with test substances and observe effects on corneal opacity and dye permeability.	ICCVAM ECVAM JaCVAM
Genotoxicity	<i>In vitro</i> mammalian cell micronucleus test	Dividing human or rodent cells are treated with test substances. Micronucleus formation indicates chromosomal breakage or changes in chromosomal number.	ECVAM
Reproductive and developmental toxicity	Embryonic stem cell test	Mouse embryonic stem cells and fibroblast cells are treated with test substances. Researchers monitor cell differentiation, viability, and proliferation.	ECVAM
Skin irritation and corrosion	EpiDerm™ skin irritation test	Human keratinocytes are cultured on membranes to form an epithelial barrier that resembles human skin. Researchers add test substances and monitor cell death and inflammatory responses.	ECVAM

^aSource: AltTox.org. For abbreviations see Table 1.

and human safety at the American Cleaning Institute, a trade association that serves the US cleaning products industry. Researchers have also begun to change their focus from trying to find *in vitro* methods that can predict animal tests, to finding *in vitro* methods that predict human responses. “If researchers can

identify the mechanism of toxicity at the biochemical level, then they can more universally apply the mechanism across various species and human biology,” says Kruszewski.

By elucidating the precise chemical and biological interactions of a particular toxic substance with living organisms, researchers

can begin to map adverse outcome pathways, or the chain of events that leads to negative health effects. Once researchers have compiled an extensive catalog of adverse outcome pathways, they may be able to use a combination of chemical structural analysis, computational methods, and *in vitro* tests to map new substances onto the pathways. In this way, they could predict adverse outcomes for previously untested substances and then, if necessary, perform focused animal tests.

do not use live animals, although some are variations of the same test. International organizations such as the European Center for the Validation of Alternative Methods and the Organisation for Economic Co-operation and Development likewise perform validation tests and make recommendations to member countries. However, the ultimate decision to accept a nonanimal test depends on each country's laws and on the testing needs of specific federal agencies.

For this reason, some experts in the EU and elsewhere believe that a complete ban on animal testing of cosmetics is premature. "What we see clearly spreading throughout the world is the wish to reduce, refine, and replace animal testing wherever appropriate alternative methods are available," says Vincent. However, in the absence of alternative methods, he deems the spreading of the ban to countries outside the EU unlikely. "The EU ban is a politically and ethically based

"MOST OF THE TOXICOLOGY TOOLS USED FOR REGULATORY ASSESSMENT RELY ON HIGH-DOSE ANIMAL STUDIES AND DEFAULT EXTRAPOLATION PROCEDURES AND HAVE REMAINED RELATIVELY UNCHANGED FOR DECADES, DESPITE THE SCIENTIFIC REVOLUTIONS OF THE PAST HALF-CENTURY."

In recent years, many countries, government agencies, and international bodies have committed to finding alternatives to animal testing, or at the very least, to promoting the 3Rs (Table 1, page 638). The regulatory process varies among countries and organizations. For example, in the United States, different government agencies oversee various classes of chemicals: The FDA regulates food additives and pharmaceuticals, whereas the EPA regulates commercial chemicals, home care products, and pesticides. For new drugs and pesticides, each agency requires a specific battery of toxicity tests, most of them using animals, to obtain premarket approval of a product. Increasingly, however, nonanimal alternatives are being added to the list of acceptable toxicity tests.

Before a US government agency can accept a nonanimal test, it must be scientifically validated to be reproducible and to accurately predict existing safety data. Many countries have an interagency organization that analyzes and recommends nonanimal tests, for example, the Interagency Coordinating Committee on the Validation of Alternative Methods (ICCVAM) in the United States and the Japanese Center for the Validation of Alternative Methods in Japan. According to the ICCVAM website, ICCVAM has contributed to the national and international regulatory acceptance of 33 safety tests that

The European Union (EU) has taken a particularly active role in mandating the replacement of animal tests with alternative methods. In 2003, the 7th Amendment to the EU Cosmetics Directive created deadlines for banning animal testing of cosmetic products and their raw ingredients. As of 2009, companies are prohibited from testing beauty and hygiene products on animals within the EU, although some tests for toxicity and fertility effects can continue outside the EU until 2013.

According to Frédéric Vincent, health and consumer policy spokesperson for the European Commission, the executive body of the EU, great progress has been made in identifying *in vitro* tests for relatively simple adverse effects or "end points," such as eye or skin irritation and corrosion (Table 2, page 639). However, for more complex end points such as carcinogenicity and reproductive effects, multiple *in vitro* tests will likely be required to replace a single animal test. Currently, researchers have not identified suites of appropriate alternative tests to predict complex chemical responses. As a result, "After the expiration of the 2013 deadline, we expect that there will be insufficient data to defend existing ingredients in the case of new safety concerns, and in particular, it will be difficult to bring new cosmetic ingredients to the market," says Vincent.

approach that may not be shared by all," he acknowledges.

In contrast to other products such as commercial chemicals, pharmaceuticals, and pesticides, the US government does not require cosmetic manufacturers to establish product safety as a condition of market approval. Nevertheless, companies are legally responsible for the safety of their products, says Sebastian Cianci, policy analyst at the FDA. The battery of tests used to ensure product safety is at the complete discretion of the manufacturer. Increasingly, manufacturers are labeling cosmetics as "cruelty-free" or "not tested on animals." However, these terms have no legal definition or standing, says Cianci. "Such claims may be applied to finished products, ingredients, or both," he says. "Or they may mean that the company relied on historical data derived from animal testing, even though it doesn't conduct any new animal testing."

Many cosmetic and personal care companies have voluntarily adopted alternatives to animal testing, motivated by ethical and economic concerns as well as the consumer appeal of "cruelty-free" labeling. "Cosmetic and personal care product companies largely stopped animal testing on finished products in the 1980s, and the use of animal testing on individual ingredients has also declined dramatically," says Halyna Breslawec, chief

scientist at the Personal Care Products Council, a Washington, DC, USA-based trade association that represents the global cosmetic and personal care products industry. She notes, however, that there remain significant gaps in the scientific knowledge needed to replace all types of animal testing. “The industry has invested hundreds of millions of dollars in developing alternative methods for all safety end points,” Breslawec says.

As an example, in March 2012 the cosmetic company L’Oréal committed \$1.2 million to the EPA’s Office of Research and Development to help advance the EPA’s high-throughput chemical screening system, known as ToxCast. To rapidly screen chemicals for toxicity, ToxCast integrates data from hundreds of automated cell-based and cell-free assays, such as transcription factor, protein level, and enzyme inhibition assays. In addition to being faster than animal toxicity tests, the method is much less expensive. “ToxCast screened over 300 chemicals in over 500 tests in about one year, at a cost of \$6 million,” says David Dix, deputy director of the EPA’s National Center for Computational Toxicology. “In comparison, it took 30 years and \$2 billion to run the same number of chemicals in traditional animal toxicity tests.”

Consumer goods giant Procter & Gamble (P&G; Cincinnati, Ohio, USA) has invested over \$300 million in the past 25 years to find replacements for animal tests. “More than 99% of our safety assessments are completed using alternative methods,” says Mark Lafranconi, section head of Central Product Safety at P&G. These include reapplication of existing information, *in vitro* methods, predictive statistical modeling, and clinical evaluations. A remaining challenge is finding alternative tests for effects that can occur after repeated exposures to a chemical. According to Lafranconi, P&G scientists are exploring methods to fill this gap.

Cosmetics manufacturers who sell their products in Europe are subject to the EU animal testing ban. P&G has fully committed to the restrictions imposed by the 2009 deadline. With regard to the 2013 deadline for animal testing of all safety end points, “Leading experts from global academia, industry, and regulatory authorities concur that the science is not yet available to fully ban safety testing using animals,” says Harald Schlatter, technical external relations specialist at P&G. He notes that Cosmetics Europe, the European personal care products trade association, concluded that although the ban

complex biological responses such as carcinogenicity and skin allergy are lacking, as are tests for toxicity due to chronic low-level exposures. Also, critics argue that *in vitro* tests don’t assess bioavailability—how the body as a whole absorbs, metabolizes, and eliminates toxic substances.

“I would like to think that at some point in the future we will not have to use any animal tests, but I don’t think it will be in our lifetimes,” says Fowle. It is more likely, he says, that safety regulators will adopt an integrated approach to testing, in which they combine multiple *in vitro* and *in silico* methodologies to screen chemicals for specific toxic effects. Those chemicals that raise red flags could then be tested only for the predicted effects in studies using limited numbers of animals. As new methods are added to the “toolbox,” safety assessments will provide more information with less reliance on animals.

“We have a system in place for analyzing chemicals that is based on whole animals, and we are not going to be able to abandon it overnight,” says Fowle. “Decisions to change the status quo are based not only on scientific facts but also on feelings and factors like economics, politics, technological availability, and legal constraints.” But the poten-

“WHAT WE SEE CLEARLY SPREADING THROUGHOUT THE WORLD IS THE WISH TO REDUCE, REFINE, AND REPLACE ANIMAL TESTING WHEREVER APPROPRIATE ALTERNATIVE METHODS ARE AVAILABLE.”

Dix and his colleagues validated the ToxCast system using hundreds of pesticides and other chemicals with well-known toxicity profiles. As part of their collaboration, L’Oréal is providing the EPA with 20 chemicals found in their cosmetic products, along with toxicity data on the substances obtained from prior animal testing. EPA researchers will screen the chemicals with ToxCast and then compare the results to the animal testing data. “We’ll provide the data to L’Oréal, and they’ll make the determination about whether ToxCast is a suitable alternative to animal testing of their products,” says Dix.

will negatively impact innovation and knowledge that benefits the consumer, it will have little or no impact on global animal welfare. According to Cosmetic Europe’s report, animal testing of cosmetic ingredients comprised only 0.0125% of total animal testing in the EU in 2008. “We believe that such a ban in the United States would be premature and would only be a symbolic gesture, without providing a meaningful reduction in animal testing,” says Lafranconi.

In the drive to eliminate animal testing without compromising product safety, significant challenges remain. Suitable tests for

tial benefits of replacing animal testing with alternative methods—reduced cost, faster analysis of more chemicals, and more relevant information for humans—promise a brighter future not only for the animals but also for industry and the consumer.

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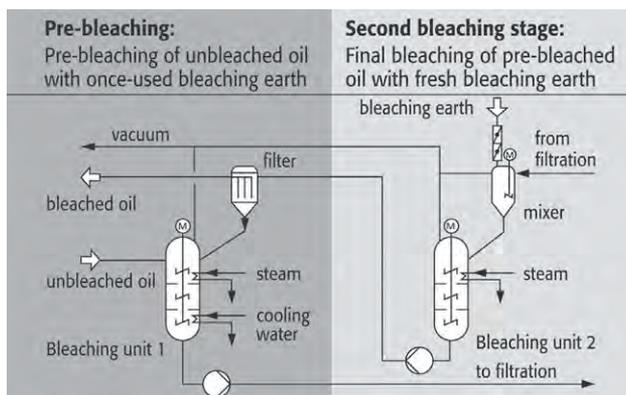


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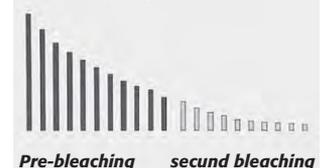
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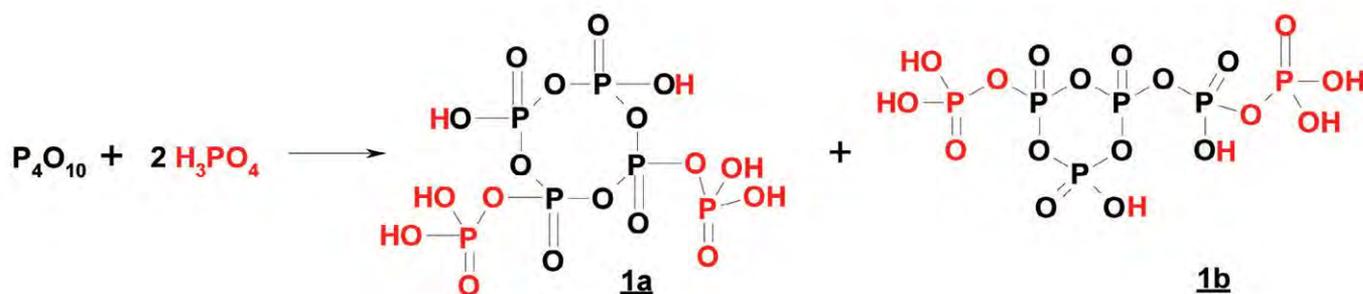


FIG. 1. Theoretical metaphosphate structures.

“Super phos” esters: the key to higher-performance products

“Super phos” hybrid technology has introduced a wide range of new phosphation compositions for product development and improvement that were previously not available through traditional polyphosphoric acid or P_4O_{10} processes. Here, Robert L. Reiersen, winner of the 2012 Samuel Rosen Memorial Award recognizing accomplishments in surfactant chemistry, describes how phosphation process control makes it possible to systematically design and adjust product compositions to deliver maximum performance, achieve competitive advantage, and even gain patent protection. His article is based on the presentation he made at the 103rd AOCS Annual Meeting & Expo in Long Beach, California, USA. The complete PowerPoint presentation is available via *inform's* digital edition. Log in at aocs.org/login and click on the supplement tab.

Robert L. Reiersen

Phosphate esters are distinguished from other surfactants by the wide range of structures and compositions that can be created to adapt them to specific applications and also by important functional properties, such as adhesion enhancement, that offer unique advantages beyond those typically offered by anionic surfactants. Phosphate esters are ubiquitous in nature and essential to life, where their chemistry reaches a state of near-perfection. Although industrial chemistry is not nearly as sophisticated, industrial chemists have managed to adapt the phosphation process and starting material structures to produce a wide range of commercially valuable products.

Phosphate structural and process considerations

Both natural and industrial phosphate esters have the phosphate in common as the essential functional group and, if the raw materials used to make them are naturally sourced, both are ultimately biodegradable and therefore complete the natural cycles.

Their uniqueness is derived from their structure. Since phosphoric acid (PA) is tribasic, it can form two anionic esters: a dibasic, monoalkyl ester (MAP) and a monobasic dialkyl ester (DAP). Consequently, phosphate esters are stable and active surfactants over a wide pH range and, in water at physiological pH, the MAP and PA can serve as buffers. Industrial products contain all three components, and their net properties are strongly influenced by their MAP/DAP ratio.

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Control over this important parameter has been achieved through a precise correlation of the composition to the reaction parameters in this generally complex process. Among these, the most important is the nature of the phosphation reagent. A polyphosphoric acid (PPA)-based process produces a characteristic mixture on one end of the spectrum, and phosphoric anhydride produces a very different one on the other. Because of its higher adhesion, a composition with higher MAP content is often more desirable.

The highest MAP/DAP molar ratios, 93:7 to 98:2, are produced with 115% to 105% polyphosphoric acid (PPA), respectively. Commercially available 115% PPA consists of essentially linear, oligomeric chains of linked phosphate groups of from one to over a dozen phosphate units. This composition, which involves many shorter, less-reactive chains, results in a final product mixture with a very high

residual phosphoric acid content, typically accounting for 30 to 60 mole percent of the starting PPA charge.

Phosphoric anhydride, P_4O_{10} , is a tetrahedral structure in which the phosphorus atoms are at the vertices, each with one apical double-bonded oxygen, and each phosphorus atom is connected to each of the other three phosphorus atoms through oxygen (P-O-P) links. This multicyclic structure theoretically would produce two MAP and two DAP esters (a “sesquiphosphate” mixture) by reaction with six moles of alcohol. That requires anhydrous conditions, however, so the orthophosphate molar ratio (PA/MAP/DAP) is more commonly 9:61:30.

An ideal reagent for a high MAP composition would seem to be a metaphosphate structure, empirical formula $H_nP_nO_{3n}$, equivalent to 122.5% PPA. While this can be approximated by very high molecular weight polyphosphoric acids, such acids are very difficult to use as phosphation reagents; in the 118–124% range, they are rubbery to glassy solids with very low solubilities in everything but water.

Improved process provides new reagent and product composition

It was discovered that phosphoric acid would react readily but controllably with P_4O_{10} . The first mole theoretically would open the highly strained P_4O_{10} tetrahedron and the second mole would open the still strained, resulting bicyclic structure (see Reference 3 for proposed, copyrighted structure) to produce the phosphate-substituted, monocyclic metaphosphate structures 1a and 1b shown in Figure 1, page 643.

This composition was considerably easier to work with than the metaphosphate glass, and its novelty was affirmed by a patent

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(Reierson, 2000). The utility was further expanded when it was found that this hybrid reagent could be selectively generated in the liquid alcohol (or ethoxylate) being phosphated. This allowed a significantly broader range of reagent strengths to be created, and, as expected, as the strength approached either extreme, represented by 115% PPA or P_4O_{10} , the phosphate ester composition produced by it was more like that produced by the respective commercial reagent. Most importantly, this expanded the range of usable phosphation reagent strengths and made the new phosphate ester compositions from them systematically available for optimization of the final ester performance. Often the mixture produced by the hybrid reagent performed better than either traditional mixture or blends of the two mixtures.

The principal benefits provided by the high MAP phosphate ester formulations are attributable to the MAP structure being a more efficient detergent over a wider pH range and providing greater affinity to natural, synthetic, and mineral (or metal) surfaces. Pre-determined MAP/DAP ratios of 88:12 to 91:9 (which were comparable in performance to the PPA 93:7 or higher mixtures) can be produced, without the large, residual excess of phosphoric acid and its attendant problems (viscosity, high salt content). The high DAP compositions, useful in emulsification, are readily prepared in the P_4O_{10} process so there would seem to be less advantage to be gained. However, here too, the hybrid offers options for improving the processes for substrates that are difficult to phosphate by P_4O_{10} alone. Phosphate ester compositions have been prepared from hydrophobes of 4 to 30 carbons (aliphatic, olefinic, and aromatic), with degrees of ethoxylation from 0 to 50 in which the residual nonionic (alcohol/ethoxylate) and phosphoric acid are each typically less than 6 weight percent (moderately higher for the highly viscous or very hydrophobic starting materials but still much better than from traditional processes).

Phosphate esters are naturally compatible

In addition to adhesion enhancement and excellent surfactant properties, one additional property deserves mention: mildness for compatibility with living tissue. This makes phosphate esters a natural choice for personal care applications. A 14-day cumulative skin irritation study showed that a potassium lauryl MAP achieved the lowest score, 4.9 out of 42.0, essentially non-irritating. The sodium cocoamphoacetate, mild enough for baby shampoos, received the second-lowest score, 11.9, near the bottom of the low irritation range. For comparison, the sodium lauryl sulfate scored 40.3. A baby wet wipe

formulation with potassium lauryl MAP as the primary skin-cleansing surfactant was found to have a zero skin irritation potential, same as the deionized water control. The potassium lauryl MAP also had low eye irritation and imparted a fresh, long-lasting after-wash skin sensation of cleanness, smoothness, and softness.

Such properties would be valued in other applications where effective cleansing of sensitive tissues is important, such as feminine hygiene, make-up removal, and oral care products. Other compositions would be useful in leave-on products for skin and hair protection, as well as for enhanced deposition and delayed release of actives to reduce the amount needed and extend the period of efficacy in their products. These would include sunscreen agents, moisturizers, fragrances, quaternary ammonium biocides, healing agents (wound debriders), pain relievers, and hair dyes.

Expanding application options

A series of PAM (phosphate adhesion monomers) has been developed to provide the benefits of phosphate esters in polymers for use in applications where the simpler structures are not adequate to meet performance requirements. Incorporation of the phosphate as a pendant group on the copolymer chain has significantly improved the adhesion, toughness, and durability of industrial and architectural coatings. In personal care, these copolymers similarly provide more durable, protective films for skin, hair, and teeth. In cases where the phosphate group might come in contact with phosphatase enzymes that would cleave the phosphate anchor to the surface, thus releasing the "dephosphated" film, the option exists for developing ablatable films for delayed actives release.

A brief list of references is provided for more detailed discussion of the above topics (page 644).

Robert L. Reierson has more than 40 years of experience in industrial research and development. He is currently a principal scientist and manager of new product development for the Novicare division of Rhodia, a Member of the Solvay Group, in Cranbury, New Jersey, USA. During the past 20 years, much of his work has focused on new product and process development for phosphate esters, with an emphasis on defining the optimal compositions for specific applications and the processes to consistently produce them. Reierson has more than 90 US and foreign patents and publications in areas of specialty monomers, functional fluids and additives, and surfactants. He can be contacted at Robert.Reierson@us.rhodia.com.

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Animal fat methylester



5050 km

Soy methylester



1.200 km

Mineral diesel



850 km

FIG. 1. Driving distance on the basis of 100 kg fossil CO₂ output of different fuels. Source: European Fat Processors and Renderers Association (EFPRA).

'Tiger in the tank'

Biodiesel produced from animal fats has a better impact on greenhouse gas emissions than vegetable oil. The following article examines the challenges posed by processing this feedstock, as well as different national interpretations of European Union (EU) regulations.

Martin Alm

Phrases such as "Tiger in the tank," "Horse power," or "How many cats do you need per 100 km?" have accompanied the niche market of biofuel production from animal fats since its earliest days. For those outside the business of biofuel production, the question arising has been: "How to make biofuels out of animals?"

Animal co-products, or by-products, are melted and rendered into edible or non-edible animal fats, respectively. The highest volumes produced are by-products of slaughter, which are not directly used in human consumption. Currently, only 1–15% of the raw material processed originates from dead animals that have died on the farm. It is important to note, however, that no animal dies

specifically to create biofuels. All produced fats are by-products of the meat chain. A member of the German parliament once stated that herds of animals are raised in Eastern Europe for biofuel production in Germany. As a result of this, his party stopped promoting biofuels originating from animal tissues, making it the only country in the EU (and perhaps worldwide) to do so. It was a body blow for farmers, the slaughter, meat, and rendering industries, and finally, for the environment.

This article aims to elucidate the challenges and possible risks of the use of animal products for the production of biofuels.

Processing of animal tissue

It is important to distinguish between non-edible and edible fats. The latter are melted for human consumption. Products produced from edible fats are lard, tallow, or poultry fat, such as duck or goose fat. These fats are normally not used in Europe for the production of

biofuels. Their destinations are usually food, feed and petfood, or the oleochemical industry.

By-products considered not fit for human consumption are rendered into different products. Rendering includes a sterilization and drying step, followed by the separation of dried material into fat and solids (protein), although the sequence of these processes can change. Compared to the process of vegetable oils, the rendering process always includes a thermal processing step.

The fatty acid profile is dependent on the species of the processed animal; and quality issues such as color and free fatty acids (FFA) are linked to the process, temperature, and freshness of raw material.

The European animal by-product regulation 1069/2009 (ABPR), divides animal by-products (ABP) into three risk categories. Category 3 considers ABP fit for human consumption, while Category 2 defines materials posing a known and manageable risk, such as microbiological risks, which can be reliably reduced by sterilization. Category 1 includes material bearing a new, nonclassical risk such as transmissible spongiform encephalopathy (TSE) in specified risk materials from ruminants. Other Category 1 materials are ABP with known medicament contamination such as zoo, circus, or pet animals, or ABP with proven contamination of forbidden substances such as heavy metals or drugs. The European Food Safety Authority (EFSA) approved the biodiesel process, even for these high risk fats, as safe.

When these three categories were introduced by a predecessor regulation in 2002, nearly all Category 1 meals and fats were burnt or incinerated. While fats were then used in thermal boilers for energy production, and later in the biodiesel process, Category 1 meat and bone meal (MBM) is still incinerated in cement kilns or power stations. Category 2 MBM and Category 3 processed animal protein (PAP) are used as fertilizer or in petfood (Category 3 only).

The use of Category 1 and 2 fats in the oleochemical industry is allowed under certain restrictions but, according to statistics of the European Fat Processors and Renderers Association (EFPRA), only a very small amount is used. It is not known whether this is due to an issue of quality, lack of profitability, or consumer concern.

The use of these fats in biodiesel production is currently more profitable than combustion in thermal boilers. The same applies to used cooking oils (UCO), which fall under animal by-product regulations—owing to the risk of spreading diseases—even if they are mainly based on vegetable oils. They are not allowed to be used in feed.

The biodiesel process

The production of biodiesel (fatty acid methyl ester, or FAME) from animal fats is more challenging than producing biodiesel from vegetable oils. Depending on the source of the ABP (Category 1, 2 or 3), and the rendering process (especially the temperature and the cleaning procedure), animal fats can contain constituents and impurities that can hamper the process and/or the biodiesel quality. The biodiesel process is negatively influenced by high phosphorus and FFA contents. Additionally, both parameters, as well as polymerized triglycerides, sulfur (from hairs, hides, feathers), ash (from bones), and plastics, harm the biodiesel quality itself.

TABLE 1. Typical default values for biofuels as defined in Annex 5 of EU Regulation 2009/28/EC. Source: EFPRA.

Biofuel production pathway	Typical greenhouse gas emission saving	Default greenhouse gas emission saving
sugar beet ethanol	61 %	52 %
wheat ethanol (process fuel not specified)	32 %	16 %
wheat ethanol (natural gas as process fuel in CHP plant)	53 %	47 %
corn (maize) ethanol, Community produced (natural gas as process fuel in CHP plant)	56 %	49 %
sugar cane ethanol	71 %	71 %
rape seed biodiesel	45 %	38 %
soybean biodiesel	40 %	31 %
palm oil biodiesel (process not specified)	36 %	19 %
waste vegetable or animal (*) oil biodiesel	88 %	83 %
pure vegetable oil from rape seed	58 %	57 %
biogas from wet manure as compressed natural gas	84 %	81 %
biogas from dry manure as compressed natural gas	86 %	82 %

Most biodiesel plants therefore have a distillation at the end of the process to reduce unwanted amounts of impurities. To handle higher FFA contents, an esterification is installed before transesterification in order to avoid saponification, which hinders the separation of the oil/water phase. If the fat is refined before, esterification is unnecessary. The process can be discontinuous or continuous; the former is more common for animal fats.

Newer than the biodiesel process, but not really new anymore, is the Neste oil process, in which vegetable oils and animal fats are hydrotreated and refined in different fractions to be used in both cars and airplanes. Initial tests in aircraft have yielded good results.

For a few years, the production of biofuels from nonfood cellulosic material and lignocellulosic material has been heavily promoted. The process contains a gasification step, followed by a Fischer-Tropsch synthesis. The process is able to synthesize a fuel with a requested specification. Unfortunately, it is currently predicted that this technology will not be available before 2020 or later. Nevertheless, all carbon-rich, rendered products such as MBM or fats can be used in this process as well.

RED—Renewable Energy Directive

Compared to vegetable oil, the processing of animal fats into biodiesel is much more sophisticated and challenging—so why do it? In the beginning, it was simply the price difference to vegetable oils, which allowed a more complex and expensive process. Meanwhile, the greenhouse gas (GHG) performance of animal fat-based biodiesel was proven and confirmed to be much better than biodiesel from any vegetable oil. On 29 April 2009, the European Parliament and the Council published the Renewable Energy Directive (RED) “on the promotion of the use of energy of renewable sources.” Article one, entitled “Subject matter and scope,” stated that “sustainable criteria for biofuels and bioliquids should be established.” By definition, “energy from renewable sources” means energy from

renewable, nonfossil sources such as biomass. The definition of “biomass” explicitly includes animal substances in the fraction of products, wastes, and residues of biological origin from agriculture and related industries. The RED provides typical GHG emission savings and sets default GHG emission savings.

Table 1 (page 647) shows data of some typical biofuels.

Biodiesel from vegetable and animal waste oil has the highest GHG emission saving. The better performance of animal fats is due to the fact that the whole production chain of meat is not taken into account, because ABP must be considered as a residue of the meat industry, and ABP are not produced for that purpose. This default value will not change in the future, even if indirect land use change (ILUC) is included in the calculation, because this burden cannot be allocated to the residues of a production process.

Figure 1 (page 646) illustrates what these default numbers mean for the three different fuels: mineral diesel, soya biodiesel, and animal fat biodiesel.

Assuming that all three cars are allowed to exhaust only 100 kg of fossil CO₂, the mineral diesel-fueled car drives 850 km. The calculation of fossil fuel into fossil CO₂ is 1:1, because every CO₂ molecule comes from fossil fuel. In using soya biodiesel, the GHG emission saving is only 31%. In other words, 69% of the exhausted CO₂ can be considered as if it is of fossil origin. The same car drives 44% further. Much more impressive is biodiesel from animal fat: The distance is 6.5 times longer than the distance of a mineral fuel-driven car. This comparison clearly shows the difference in distance you can travel with the same impact on the climate.



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The RED also states, in Article 22, that “the contribution made by biofuels produced from waste, residue . . . shall be considered to be twice that made by other biofuels.” With this so-called “double counting,” the animal fat biodiesel car would be considered to drive even 13 times further than a car running on mineral fuel.

Wastes and residues

While the wording seemed unambiguously clear, a discussion in different EU member states suddenly erupted on how it was to be interpreted. Germany’s first step was simply to exclude Categories 1 and 2 biodiesel from the biofuel promotion scheme. Some years later, the Ministry of Environment published a study that showed animal fats are environmentally friendlier when used in the steel industry and cement kilns than in the production of biofuels, because they are used to replace coal. Incinerating fat in these industries is rather unusual, and only small amounts went into them after the bovine spongiform encephalopathy crisis in Germany, when new legislation and rising stocks of fats pushed down the prices. Additionally, the same calculation was not used for the use of vegetable oils in boilers. Therefore, there was no way to compare the effect of the difference between the two on the climate.

Moreover, since the start of this year, Category 3 fat was excluded as well. The reason for this is that there is already an existing market for these fats. The same German ministry argued that the waste framework directive (WFD) exempts from its scope animal by-products that are covered by the ABPR. Thus, animal by-products cannot be considered as waste or residues. Thus, used cooking oils are outside the scope of the promotion scheme of double counting as well, which was obviously not wanted. In a communication to the German biofuel association, the definition was given that UCO from vegetable oils only, in which meat was fried, are out of the scope of the ABPR and are thus categorized as waste. But who will guarantee that UCO is free of tallow? Who can control it? And how? UCO collectors, traders, and users consequently work in constant uncertainty.

Denmark always avoided the “Food vs. Fuel” debate by not promoting the transformation of food or feed into fuel. Thus, the biodiesel plant of Daka, in Løsning, which runs only on Category 1 and 2 fats, was very welcome. A Danish study confirmed that the use of animal fats in biofuels is more sustainable than all other uses, making it suitable for double counting.

In the Netherlands, all fats are currently acknowledged as “waste and residues,” but from January 1, 2013, Category 3 fats will be excluded from the double counting, as they are seen more as a product than as a waste or residue.

In the UK, a recent discussion in the Department for Transport (DFT) about the Renewable Transport Fuel Obligation (RTFO) led to the conclusion that there is a market for fats from Categories 2 and 3. Thus, they are excluded from double counting. It is interesting to note that, for years, Category 2 fat was neither produced in the UK nor used in Europe (the only possible user is the oleochemical industry).

Despite these facts, the DFT announced they are ready to re-evaluate the RTFO with regard to Category 1, if this can be used for other purposes (which is, theoretically, already the case). The UK excludes animal fats for an only virtual market, which has not existed in the EU for years.

It is a similar story in Spain: Category 1 fats and UCO count double, Category 3 is out, and Category 2 is under discussion.

Animal fats and greases continue to slide

Global exports of tallow and greases are expected to continue to decline in 2012/13 to the “lowest level in more than 20 years,” according to Oil World ISTA Mielke GmbH. Shrinking livestock production owing to “skyrocketing feed costs” and rising usage for biodiesel have led to lower production, the Hamburg-based consultancy noted in a recent market report.

Italy is close to finishing the implementation of the RED into national law. Currently, a cap of 10–20% is foreseen for double-counting biofuels. In other words, only a certain amount of biofuels can count double. The legal background for that decision is more than questionable, but the RED does not foresee this cap.

For EFPRA, the varied interpretations in a joint European market are difficult to understand. The WFD exempts all animal by-products from its scope, owing to the fact that they are already better, more precisely and stringently ruled in the ABPR, and not because they cannot be seen as waste or residues.

The general understanding must be that animal by-products would have been ruled under the WFD if the ABPR had not been in place.

In July 2012, the Concerted Action on the Renewable Energy Sources Directive (CA-RES) started. For three years the CA-RES has supported the transposition and implementation of the RED and the achievement of national targets. It is organized as a confidential dialog between national authorities responsible for the implementation of the RED or their nominated representatives. In the CA-RES, member states share experiences and best practices, and develop common approaches.

Regarding biofuels from animal fats and UCO, the CA-RES subgroup WG-8 recognized that the implementation in some member states is the same, but overall there is a disharmonized implementation. WG-8 recommends to count Categories 1 and 2 as double, while Category 3 should not be counted double. UCO is defined almost all over Europe as a waste, but the huge demand for UCO is causing prices to rise. Occasionally, the price is even higher than the price for unused cooking oil. This situation is not ideal. WG-8 participants concluded that one problem stems from the fact that there is no clear definition for what constitutes “used,” and further research is required. WG-8 considers the market would benefit from a clear and general policy.

Finally, rendered products—especially fats transformed into biodiesel—have an excellent impact on GHG emission savings. Nevertheless, as these volumes are naturally limited, they can only be seen in the big mosaic of measures to stop climate change. Due to their high performance, however, they can clearly be seen as a shining piece.

Martin Alm is technical director of the European Fat Processors and Renderers Association. He can be contacted at mail@dr-alm.eu.

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Making the most of algal biomass with pyrolysis

Kaige Wang and Robert C. Brown

Scientists and the general public alike are intrigued by the potential for using algae as a feedstock for next-generation biofuels. Algae-based biofuels are appealing on several levels: They can be cultivated to contain high levels of lipids suitable for fuel production, they can be higher yielding than terrestrial crops, they can be grown in wastewater, they are natural recyclers of carbon dioxide, and their use avoids the difficult biofuel issue of competition over arable lands.

In spite of these appealing advantages, algae-based fuel has several barriers to commercial production. Extraction of lipids from algae is energy-intensive. Most microalgae strains have thick cell walls, which make wall disruption and lipid extraction costly in terms of energy and expense. Researchers also struggle with a trade-off between lipid concentration and overall biomass yield. Finally, because of algae's high intrinsic protein content, algae cultivation requires significant amounts of nitrogen.

Iowa State University (ISU; Ames, USA) is exploring fast pyrolysis as a means to mitigate these problems. Rather than separately processing the algal lipid fraction, ISU researchers explored ways to process whole algal biomass into chemical products. First attempts to pyrolyze algae, by rapidly heating them in the absence of oxygen, were not completely successful. Although lipids converted into fatty acids, the protein converted into various nitrogenous compounds including pyrroles, nitriles, indoles, pyridines, and poly-heteroaromatics. The resulting bio-oil from algae contains 5% to 10% nitrogen, which is high enough to poison catalysts during bio-oil upgrading and to produce unacceptable nitrogen oxide emissions during combustion.

ISU researchers, recognizing that zeolite catalysts can deoxygenate organic compounds during pyrolysis, speculated that these catalysts might also denitrify protein in biomass feedstocks. Pyrolysis experiments were conducted in a micro-furnace pyrolyzer. A commercially available zeolite catalyst was used. Algae samples—some with and some without the catalyst—were loaded into deactivated stainless steel cups, which were in turn automatically dropped into the preheated furnace. Helium carrier gas was used to sweep pyrolysis vapors into a gas chromatograph.

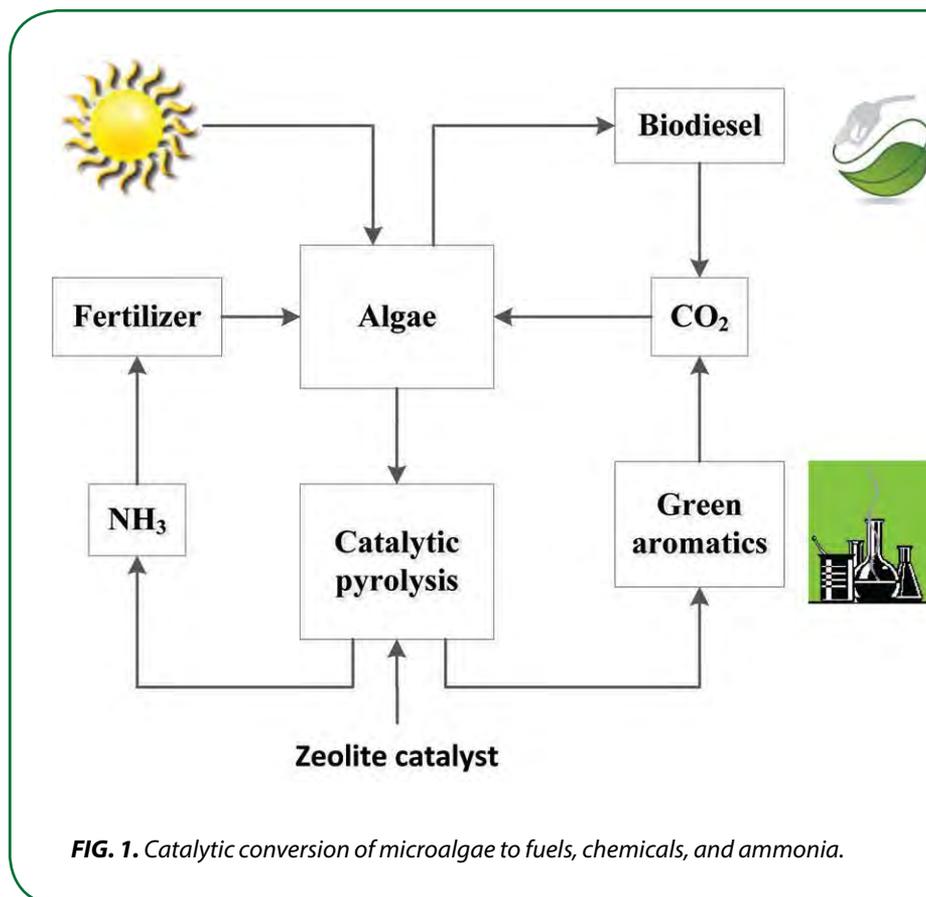


FIG. 1. Catalytic conversion of microalgae to fuels, chemicals, and ammonia.

The chromatographic results indicated a significant effect of catalysis on the pyrolysis products of microalgal biomass. All of the nitrogen- and oxygen-containing compounds were eliminated, and aromatic hydrocarbons were generated. The result indicates that protein-derived nitrogen compounds can be converted to aromatic hydrocarbons just as oxygenates from lignocellulosic biomass are converted.

Analysis of the pyrolysis vapors showed benzene, toluene, and xylene (BTX) to be the most abundant aromatic products. Total aromatics yield increased with increasing temperatures, with the increase becoming insignificant at temperatures above 600°C.

BTX are important petrochemicals. Benzene is used as an intermediate to make other chemicals. Toluene is a common solvent and a raw material for TNT (trinitrotoluene). It can also be used as an octane enhancer in gasoline fuels. Xylene is the precursor to terephthalic acid and related derivatives, which are used in production of PET (polyethylene terephthalate). Like toluene, xylene can also be blended into gasoline to increase octane numbers.

information

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Separate experiments were performed to analyze the production of ammonia (NH₃) during catalyzed pyrolysis. Ammonia generated from this process increased with increasing temperatures. For elevated reaction temperatures (above 700°C), more than half of the nitrogen in the microalgae was released as NH₃, which suggests the potential to recycle NH₃ as a nutrient for microalgae cultivation.

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Nutrient supplies, especially nitrogen supplies, represent a significant impact on cost and sustainability of microalgae-based biofuel production. Nitrogen fertilizer for microalgae growth is typically supplied in ammonia or nitrate forms, which are historically produced by the energy-intensive Haber-Bosch process. Considering the impact of nitrogen on energy use and greenhouse gas emissions, the viability of microalgae-based biodiesel production is questionable. With catalytic pyrolysis, nitrogen recycling reduces the amounts of fertilizer required, which in turn reduces both production costs and greenhouse gas emissions.

Biodiesel refinery processes focus on lipids in the microalgae while discarding the protein. Since protein is the largest component in fast-growing microalgae, in practice, these methods waste as much as 60% of the plant. In contrast, in catalytic pyrolysis conversion, it is possible to use every component of algal biomass to produce fuels and chemicals. This efficiency offers promise for catalytic pyrolysis as a feasible approach for the commercial utilization of algal biomass.

This technology would also be feasible to use alongside biodiesel lipid extraction to process the algae remnants of that process. These remnants represent about 30–80 wt% of the dry microalgae, depending on the particular microalgae strains and growth conditions. While the market for biofuels and nutraceuticals is growing fast, the same cannot be said for the abundant by-products. Historically, these remnants are used for animal feed. However, due to the relatively small size of the animal feed market compared to transportation fuel markets, use as animal feed has limited potential to address the problem of finding uses of microalgal remnants. In fact, if microalgae were to replace petroleum as the feedstock for the US gasoline supply, 750 million metric tons of algal remnants would be produced annually—50 times the amount of feed supplement required by the 100 million cattle in the United States. Fast pyrolysis of the protein-rich remnants into nitrogen-free aromatic compounds would greatly improve the economic prospects of microalgae biorefineries.

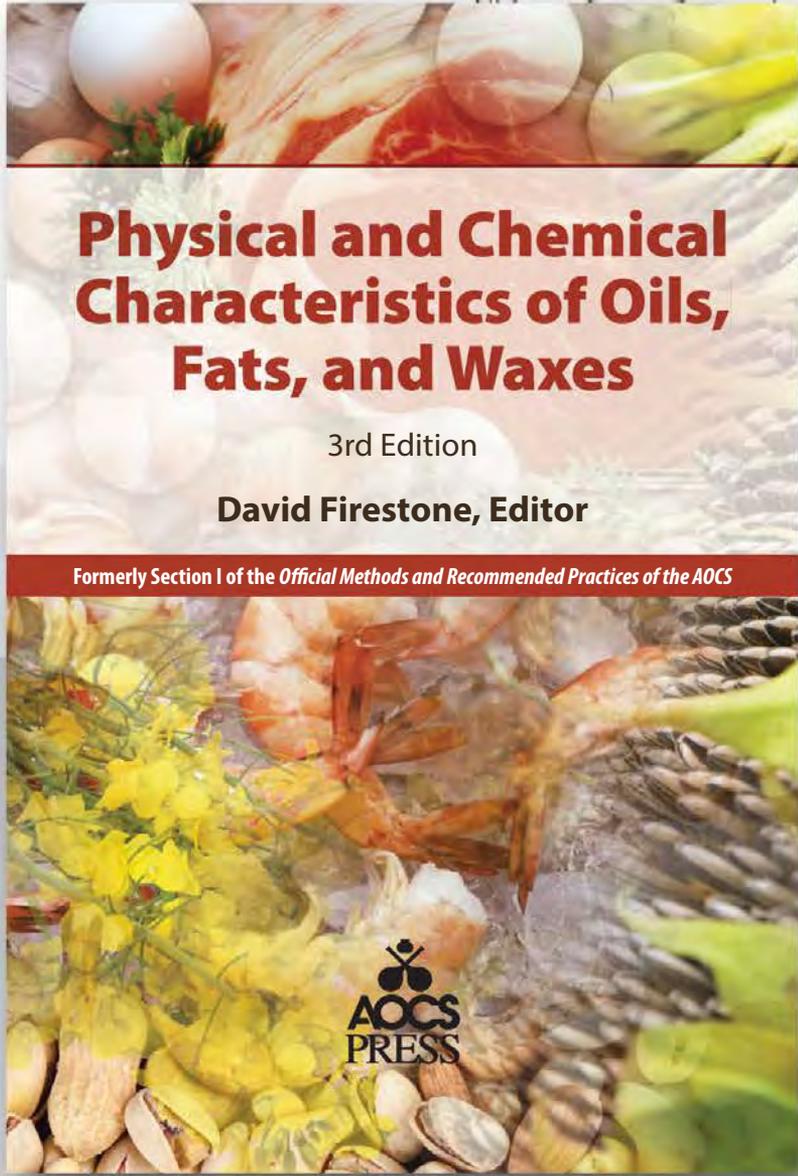
Based on the experiments and discussion above, we propose a pathway for catalytic pyrolysis processing of microalgal biomass (Fig. 1, page 651).

Algae—either whole algae or algal remnants after lipid extraction—may be used as feedstock. The green aromatics generated from catalytic pyrolysis may be used as petrochemicals or fuels. The use of aromatics or microalgae-based biodiesel creates CO₂, which is, again, consumed by algae production. Ammonia released from algal pyrolysis may be used as fertilizer for algae cultivation, thereby offsetting fertilizer expenses for microalgae biorefineries. By offering environmentally sustainable closed systems for nitrogen and CO₂ recycling and the efficient use of all microalgal components, a catalytic pyrolysis pathway such as this offers great promise for the economic and environmental feasibilities of microalgae biorefineries.

Kaige Wang joined Prof. Robert C. Brown's research group at ISU as a graduate research assistant after receiving his master's degree in energy engineering at Zhejiang University (China) in 2010. His current research interests focus on pyrolysis of microalgae and lignocellulosic biomass for production of fuels and value-added chemicals. He can be contacted at kaigew@gmail.com.

Dr. Brown is Anson Marston Distinguished Professor of Engineering and Gary and Donna Hoover Chair in Mechanical Engineering at Iowa State University (ISU). He is the director of ISU's Bioeconomy Institute and the Center for Sustainable Environmental Technologies. He can be contacted at rcbrown3@iastate.edu.

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The America Invents Act:

Groundbreaking US patent law changes are here

Paul S. Tully and Jeremy E. Noe

The recent enactment of the Leahy-Smith America Invents Act (AIA) has set the stage for what are probably the most sweeping US patent law reforms in the past 40 years. The AIA is a culmination of numerous (and mostly fizzled) efforts over a five-year period to bring US laws into harmony with the practices and procedures followed by most other countries around the world. As discussed below, implementation of the AIA's provisions has drastically changed—but not necessarily leveled—the patent playing field.

The AIA affects nearly every facet of patent law as we know it. Here are several key AIA changes that all industry leaders should be keenly aware of going forward:

- The AIA establishes a first-to-file system vs. the current first-to-invent scheme.
- There are many new routes and procedures available for review of US patents and patent applications.
- The AIA expands prior user rights, particularly in the area of process technologies, which are typically held by companies as trade secrets.
- The provisions of the AIA will almost certainly lead to a greater prominence (and scrutiny) of provisional patent application filings.

First to invent vs. first to file

Of the major changes found in the AIA, perhaps the most significant is the change from a first-to-invent system to a first-to-file system. Under the old rules, a person who demonstrated that he or she was first to invent by showing both the conception of a novel idea and the reduction to practice, i.e., a showing of a working or fully functional

embodiment of the idea, would be rewarded with a patent over other inventors who had subsequently invented the same subject matter. As long as an inventor was diligent in pursuing a patent for the new invention, who filed an application first was somewhat without moment in determining who ultimately was rewarded with a patent in the case of two competing inventors. The old “first to invent” rule has been in operation since the modern inception of the US patent system.

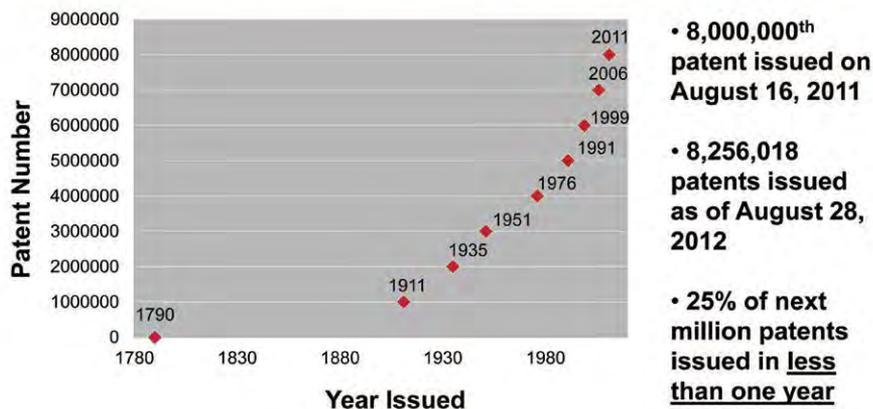
The new rules of the AIA implement a system whereby the first-to-file inventor (who can still show conception and reduction to practice) wins the patent battle against a competing inventor. The new rule, which goes into effect on March 16, 2013, is essentially in harmony with the rest of the world, which

has operated under a first-to-file regime for many years.

There some important caveats that all inventors and companies should keep in mind concerning the new first-to-file rules:

- There is a limited one-year grace period to the new rule for the publication of new inventions by inventors. (This exception does *not* include sale activities or public use of an invention, only printed disclosure.)
- This exception provides a powerful sword and shield for inventors, whereby an earlier inventor publication will not prevent that inventor from obtaining first-filer status, but it

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FIG. 1. As this graphic shows, the pace of innovation is accelerating rapidly. It took 75 years for the United States Patent and Trademark Office to issue the one millionth patent. Each successive million patents has issued at an accelerating rate. It required only seven years between Patent No. 6,000,000 and Patent No. 7,000,000, and only five more years to reach Patent No. 8,000,000. As of August 28, 2012, Patent No. 8,256,018 had issued; i.e., more than 25% of the next million patents issued in less than one year.

CONTINUED ON NEXT PAGE

will act as damaging prior art to another inventor—even if that inventor is first to file. Put another way, if an inventor publishes a new invention, a competing inventor files a patent application first, and the original publishing inventor files a patent application second, but within one year of his or her publication date, the first inventor may still be able to obtain patent rights over the first filer, despite the fact that the first inventor's patent application was second in line.

There are many practical implications to the shift from first-inventor to first-file rules. First, the inventor who is first to invent is essentially irrelevant. The person who is first to file (or publish) controls priority and key prior art dates related to the technology at issue. This is likely to result in a rush to file (or publish) first, ask questions later. Unfortunately, it may also result in an increase of the filing of incomplete inventions. Overall, it is highly likely in a competitive field that there will be an increase in preemptive disclosures, and/or provisional patent application filing practice will likely increase.

To prosper in today's competitive environment, it is important not only to obtain and protect one's own patent rights but also not to encroach on the patent rights of others. It can be difficult, however, to determine with any reasonable certainty the metes and bounds of another's exclusionary patent rights, short of expensive and lengthy litigation in a federal district court. To address this problem, the AIA implemented several new routes for lower-cost review of pending and issued claims by the United States Patent and Trademark Office (USPTO).

New routes for patent and trademark review

Preissuance submission (benefit to third parties). Preissuance submissions are submissions of prior art by third parties in any pending patent applications. They are intended to reduce the risk that unpatentable claims will mistakenly be allowed by examiners who are unfamiliar with the prior art.

A third party may file a preissuance submission for any pending patent application that is filed on or after September 16, 2012. The

preissuance submission may be filed anonymously but must be based on a prior art publication and must contain a concise statement of the asserted relevance of each submitted publication to the pending claims. Preissuance submissions must be filed before (i) a notice of allowance is issued or (ii) the later of six months after the application is published, or the date of the first rejection of any claim.

This route offers significant advantages over other processes for challenging patent claims:

- The submitter can remain anonymous.
- The submitter may comment on the prior art that is submitted (formerly, third-party submissions of prior art were allowed, but the submitter was not allowed to comment on the art, which risked having the examiner not apply the art in the way the third party desired).
- According to proposed rules implementing this provision, the concise description of relevance can take the form of a claim chart; i.e., the submitter can propose specific rejections of pending claims based on prior art.
- Unlike other routes, the third party need not submit the best prior art known to it, and can reserve better prior art for a post-issuance challenge.
- This route will have a lower cost compared to other routes; proposed rules suggest that no fee will be required for submission of three or fewer publications.

Supplemental examination (benefit to patentees). As of September 16, 2012, a patentee may file a request for supplemental examination of any issued patent still in force. Unlike other routes for correcting issued patents, the request for supplemental examination may be based on any grounds believed to be relevant and is not limited to printed publications. The USPTO must decide within three months whether a substantial new question of patentability is created by the submitted information, in which event the USPTO will order a reexamination of the issued claims.

Several key advantages of this route include:

- It provides a post-issuance route for a patentee to submit prior art for USPTO consideration without having to formally seek reissue or reexamination.

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- It provides an avenue to address potential grounds for invalidity or unenforceability before an infringer can raise them.
- It can cure unintentional failures to disclose prior art, because a patent will not be held unenforceable based on information that was not considered during prior examination, so long as it is considered by the PTO in a supplemental examination. (However, it cannot be used to cure intentional failures to disclose prior art, which constitutes material fraud).

Post-grant review (benefit to third parties). Under the AIA, a third party may file a request for post-grant review, based not merely on printed publications, but rather any ground for potential lack of patentability, including prior sale and prior public use, both of which—until now—could only be raised during litigation.

Post-grant review applies to patents issued on applications filed after March 16, 2013, and may not be filed anonymously. A petition must be filed within nine months of patent grant or issuance of a reissue patent but may not be filed if the submitter has previously filed a civil action alleging invalidity of the patent at issue. Like a request for supplemental examination, the grounds for a petition for post-grant review are not limited merely to printed publications and rather can include prior sale or prior public use.

The USPTO must act within three months of the submission, and will order a post-grant review of the issued claims if (i) under a more likely than not standard, at least one challenged claim is unpatentable or (ii) the submission presents a novel or unsettled legal issue.

Advantages to this route will be:

- The patentee's response is limited to showing why the petition should not be granted.

- The post-grant review will be conducted by technically proficient Patent Trial and Appeal Board reviewers.
- The patentee has only one opportunity to amend or propose substitute claims.
- The review can be terminated by settlement between the parties.

Prior user rights

A second major change to US patent laws under the new AIA is found in the expansion of prior user rights. These rights are a desirable but seldom available patent infringement defense, particularly in the chemical industry, where many processes, both on the small research and development (R&D) scale and the large industrial scale, are often held by companies as closely guarded trade secrets. Under the new rules of the AIA, which went into effect on September 16, 2011, protections for process users, who are practicing technologies out of the public eye, are expanded to cover “a[ny] process, or consisting of a machine, manufacture, or composition of matter used in a manufacturing or other commercial process.” This is an important provision for the chemical sector that cuts both ways:

- The defense is a personal defense requiring “good faith” commercial use in the United States.
- The defense must be asserted “in connection with an internal commercial use.”

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What is prior art?

Prior art refers to the sum total of information—documented and undocumented—that has been disclosed publicly in any form (patents, published articles, presentations, things known or used publicly, and the like) from anywhere in the world about an invention before a given date. It establishes novelty and is the main criterion used to determine whether an invention is patentable.

- The commercial use in question must occur at least one year before the earlier of “(a) the effective filing date of the claimed invention or (b) the date on which the claimed invention was disclosed to the public.”
- The technology at issue cannot be licensed/assigned except as good faith transfer of an entire business or line of business.
- Defense is not available to products themselves.

The bottom line: prior user rights under the AIA provide a defense to claims of patent infringement claims, where a company has been secretly using a patented process, for perhaps many years. From a practical perspective, the new prior user rights rules may dissuade some inventors from filing for process patents, which in turn may lead to a decrease in publicly available information, and an increase in trade secrets.

Greater prominence (and scrutiny) of provisional patent applications

Provisional patent application was introduced to US patent law in 1994 to “match” the longstanding 12-month priority that was accorded to foreign-filed applications. A provisional patent application is nothing more than a first step toward obtaining a patent; by itself, it will never mature into a patent. But a provisional patent application nevertheless can be beneficial because it (i) is cheaper and less complex to prepare; (ii) may establish a potentially earlier effective

filing date; (iii) permits use of a “patent pending” notice; and (iv) is never published; it is abandoned.

Under the AIA, the statutory benefits of provisional patent applications remain unchanged. However, the strategic benefits of provisional filings gain more prominence because such filings are likely to become ubiquitous to secure the earliest possible priority date. For a solo inventor or a small entity, a provisional filing can be a hedge against losing a USPTO filing race to a larger and better-funded entity. By the same token, to a large entity a strategy of serial provisional filings can be a hedge against losing a technology development race to a competitor.

Particularly in view of the Draconian first-to-file system ushered in by the AIA, provisional patent applications are also likely to be subject to much more scrutiny. A disgruntled “also-ran” will be likely to dissect any provisional application that confers priority of invention to another. A provisional filing later deemed to be insufficient will not preserve a priority date and may render invalid any patent based on that provisional filing.

A perhaps unintended outcome of the AIA is that provisional patent applications will likely become more complex and complete in content, in order to best support the earliest possible priority date for the broadest possible subject matter, and hence may end up mimicking a regular patent filing in most respects, including costs to prepare.

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Jeremy E. Noe is an owner/partner with McDonnell Boehnen Hulbert & Berghoff LLP. Noe’s practice spans all aspects of patent litigation, patent and trademark preparation and prosecution, opinion preparation, and client counseling. In his patent prosecution practice, Noe leverages a deep understanding of cosmetic chemistry and consumer product formulation, acquired during his 15-year R&D career in consumer product development prior to becoming an attorney. He also assists clients on a wide range of other patent and trademark issues, including due diligence investigations, pre-litigation counseling, portfolio management, and evaluation of licensing opportunities. His technology experience involves a broad range of life sciences technologies, including pharmaceutical compounds and formulations, drug delivery systems, medical devices, and biotechnology, as well as a broad range of process engineering and manufacturing technologies.

[FAST FACTS]

Per capita fats and oils consumption

World consumption of oils and fats increased from about 148 million metric tons (MMT) in 2006 to 176 MMT in 2011. Below is a geographical breakdown of the average per capita consumption of oils and fats in 2011.

■ European Union	61 kg
■ United States	50 kg
■ India	15 kg
■ China	25 kg
■ Ethiopia	4 kg

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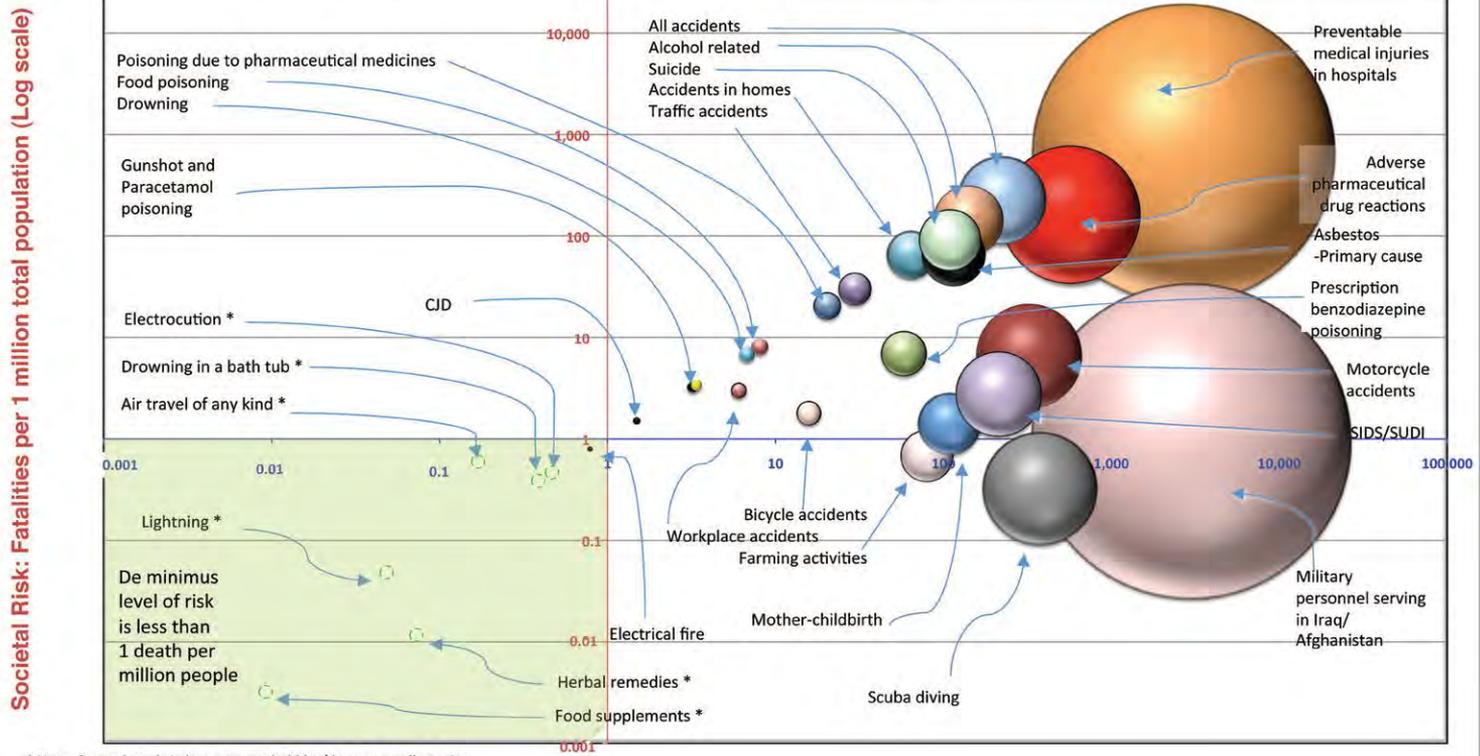
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Societal vs Individual Risk of Death in the United Kingdom

Societal risk is represented as the risk of death per million total population. Individual risk is represented as the risk of death per million exposed to that hazard. Bubble size represents the relative risk to an individual. By way of example, the bubbles representing deaths due to preventable medical injuries in hospitals and military personnel in Iraq/Afghanistan are a similar size because the risk of death to a patient in a UK hospital is similar to that for a soldier deployed to a war zone. Medical injury poses a greater risk to society simply because vastly more citizens are exposed to that risk and hence die. **Note: Log scales.**



* Note: Green dotted circles represent bubbles/dots too small to print
 Sources: Variety of UK Government and NGO databases, reports, officials and expert advisers.
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Individual Risk: Fatalities per 1 million people exposed to risk (Log scale)

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Regulation of dietary supplements

The category of products known as nutraceuticals has drawn considerable attention in recent decades, underscoring the need for regulatory attention and surveillance. Most frequently, the term nutraceutical relates to two categories of products: dietary supplements and functional foods. The following article focuses on dietary supplements as opposed to functional foods.

Alexander G. Schauss

The dietary supplement industry and the US Food and Drug Administration (FDA) have challenged each other over several decades. Until the passage of the Dietary Supplement Health and Education Act of 1994 (DSHEA), FDA had the option to declare a supplement a food or a drug. This caused considerable controversy and confusion. In an effort to resolve the issue, Congress introduced legislation in 1992 that created a new regulatory scheme for supplements. When DSHEA was passed unanimously in both houses of Congress in 1994 and signed into law by President Bill Clinton, it gave FDA widespread authority to regulate the industry. However, not until 2010 did all regulatory aspects as envisioned by Congress go into effect, much to the consternation of advocates both within and outside the industry who supported such regulatory oversight.

CONTINUED ON NEXT PAGE

Regulations in Europe

In Europe, food supplements are regulated as foods, and legislation focuses on vitamins and minerals. The main legislation is Directive 2002/46/EC, which sets labeling requirements and requires that maximum and minimum levels be set for each vitamin and mineral added to supplements.

An annex to the directive, which was amended by Regulation 1170/2009 in November 2009, contains a list of permitted vitamin or mineral substances that may be added for specific nutritional purposes in food supplements. To be considered for inclusion in the list, vitamin and mineral substances must first be evaluated for safety and bioavailability by the European Food Safety Authority (EFSA) and companies wishing to market a substance not included in the permitted list need to submit an application to the European Commission (EC).

EFSA finalized its evaluations of all general function claims, other than those related to botanicals, in June 2011, having published a total of 341 opinions covering 2,758 claims. The European Commission and the Member States agreed that a limited number of these claims would be eligible for further assessment by EFSA, based on additional data submitted by Member States.

Using EFSA's scientific advice, the Commission has already adopted a list of 222 approved general function claims for use in the European Union. EFSA will continue to assess health claim applications under the individual authorization procedure.

of enforcement actions taken against supplement companies in recent years, often beginning with warning letters. Failure to remove claims challenged by the agency provides the FDA the authority to shut down a distributor or manufacturer if compliance is rejected. Such enforcement actions have occurred with increasing frequency particularly when the claims made were so egregious that the agency had no choice but to use the force of law to seize products and shut down the offender's operation.

When it comes to claims, DSHEA allows manufacturers and distributors to make "structure/function claims." These claims describe the role of a nutrient or dietary ingredient intended to affect normal structure and/or function in humans. FDA offers numerous examples of what would be an acceptable structure/function claim [1]. The key is to objectively determine the evidence in support of the claim in terms of whether it implicitly or explicitly is a disease claim. FDA provides 10 criteria to help clarify the types of claims that may be made without prior authorization or approval by the agency. DSHEA does not allow a claim to be made for the prevention, mitigation, or treatment of a disease. When such claims are made, the agency has the authority to demand that the claim be removed, usually by sending a warning letter to the manufacturer. In most cases, the company quickly removes the claim to avoid further regulatory scrutiny or enforcement action.

The US Federal Trade Commission (FTC) also has regulatory authority in terms of advertising claims made for dietary supplements. The agency has provided guidance for the industry related to claims. It requires that any claim made be based on "competent and reliable" scientific evidence. Attempts by the FTC to require that health claims be substantiated based on randomized, double-blind, placebo-controlled studies was challenged by an administrative law judge in a landmark ruling issued on May 21, 2012, in the matter of *FTC v. POM Wonderful LLC* [2], while also ruling that the defendant had engaged in deceptive advertising. A final decision in this matter by the FTC Commission, whether to adopt, in whole, in part, or not at all the administrative judge's opinion, is expected by the end of 2012.

On June 22, 2007, the FDA issued dietary supplement final rules for current good manufacturing practices (cGMP). DSHEA established regulations to require cGMP to ensure proper controls are in place so that products are processed in a consistent manner, are able to meet quality standards, and that any representations or claims made about a product are substantiated by adequate evidence.

The cGMP apply to all domestic and foreign companies that manufacture, package, label, or hold dietary supplements, including those involved with the testing, quality control, packaging, labeling, and/or distribution in the United States.

The rules require manufacturing of products to be consistent for identity, purity, strength, and composition. The rules also apply provisions related to the design and construction of physical plants that facilitate maintenance, cleaning, manufacturing operations, quality control procedures, testing of the final product, including incoming and in-process materials, how consumer complaints are handled, and record maintenance.

To allow the supplement industry to comply with cGMP requirements, the agency staggered inspections over a three-year period to allow small businesses sufficient time to become compliant. The final rules for large companies, however, required compliance by 2008, or one year after the final rule was published.

To date, nearly all supplement companies have been inspected by the agency, resulting in hundreds of warning letters. Warning letters

The term "dietary supplements" is defined in 21 U.S.C. 321(ff) to mean:

"... a product (other than tobacco) intended to supplement the diet that bears or contains a vitamin, mineral, or herb or other botanical, an amino acid, a dietary substance for use by man to supplement the diet by increasing the total dietary intake, or a concentrate, metabolite, constituent, extract, or combination of any of the above ingredients."

The definition goes on to state that such products are intended for ingestion. They cannot be a conventional food and must be clearly labeled as a dietary supplement. A supplement cannot be an approved drug or biologic, or authorized for clinical investigation under an IND [investigational new drug application], unless the material in question was previously marketed as a dietary supplement or as a food.

With the passage of DSHEA, such basic needs such as labeling information have become much more consumer-friendly and uniform in placement and appearance.

What claims can be made for supplements remains a contentious issue as it can tread on First Amendment concerns. Numerous lawsuits have debated this issue in federal courts with victories reported by both sides. However, DSHEA is clear on one critical point: Whatever claim is made on the label or in advertising, it must be "truthful and nonmisleading" per the regulation. This provision of DSHEA is its cornerstone. Failure to abide by this principle has resulted in hundreds

are public domain documents and as such, various trade associations such as the American Herbal Products Association (APHA) and trade publications notify their members and readerships on who receives such warning letters.

On December 22, 2006, President George W. Bush signed into law the Dietary Supplement and Nonprescription Drug Consumer Protection Act (DSNDCP). It amends existing laws with respect to adverse event reporting and recordkeeping for both supplements and nonprescription drugs. Under the Act, serious adverse events must be reported to the FDA within 15 business days using MedWatch Form 3500A after a manufacturer, packer, or distributor of a supplement receives a report of a serious adverse event associated with the use of supplements in the United States. Prompt submission is important for public health reasons.

Since mandatory reporting of serious adverse reports was instituted in June 2009 under DSNDCP, there have been no reported deaths associated with the use of dietary supplements according to the US National Poison Data System [3]. This is quite remarkable in that it is estimated that over half of the American population consumes dietary supplements each day. If each person consumed a single supplement each day, that would equal 165 million individual doses a day, for a total of more than 60 billion doses annually. The experience in the United States is comparable to that of other countries, as illustrated in Figure 1 for the United Kingdom.

Economically motivated adulteration of dietary supplements with approved pharmaceutical ingredients (API) and/or their analogs is a concern. How to bring an end to this illegal activity is being debated in Congress. The supplement industry is urging FDA—with mixed results—to vigorously use existing enforcement powers to stop such clandestine activities.

Adulteration of supplements with undeclared medications has been limited largely to products intended to support or enhance weight loss efforts, sexual function, or bodybuilding, with API such as sibutramine, sildenafil or tadalafil, or steroids, respectively. Numerous undeclared ingredients have been discovered to be analogs of API (e.g., homosildenafil, thiosildenafil, hydroxyhomosildenafil, amino-tadalafil, piperadino vardenafil), by modification of the original chemical structure of the compound, in an attempt to avoid analytical detection. In some rare cases, the agency has learned from contract analytical labs that perform forensic investigations of supplements that the labs have discovered combinations of drugs such as phentolamine, an α -adrenergic blocker used as an antihypertensive agent, with PDE-5 inhibitors [4]. A recent report by the *Chicago Tribune* newspaper reveals how undeclared medications get into supplements, documenting how difficult it can be for the FDA or industry self-policing efforts to stop such activity [5].

Ending such clandestine adulteration of supplements is an ongoing challenge that, until it is accomplished, will undermine confidence by the public in the purity and quality of dietary supplements. Economically motivated adulteration of finished dietary supplement products is not limited to the United States but is found throughout the world in every country with far stricter regulations.

For the pharmaceutical industry, the problem is deliberate counterfeiting of finished products, as distinct from compromised manufacturing [6]. According to the Chinese Ministry of Public Security, authorities seized more than \$182 million worth of counterfeit medicines and detained more than 1,900 people suspected of selling fake drugs during police raids held in August 2012. The drugs had been advertised as treatments for hypertension, diabetes, cancer, and other

information

1. US FDA, *Guidance for Industry: Structure/Function Claims, Small Entity Compliance Guide*, <http://tinyurl.com/FDA-DS-Guidance>.
2. Administrative Law Judge Uphold's FTC's Complaint that POM Deceptively Advertised Its Products as Treating, Preventing, or Reducing the Risk of Heart Disease, Prostate Cancer, and Erectile Dysfunction, <http://ftc.gov/opa/2012/05/pom.shtm>.
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5. Tsouderos, T., Dangerous pharmaceuticals marketed as supplements, *Chicago Tribune*, August 10, 2012, <http://tinyurl.com/CT-Supplement> (requires free registration).
6. Organisation for Economic Co-operation and Development, *The Economic Impact of Counterfeiting and Piracy, Executive Summary, 2007*, <http://tinyurl.com/OECD-Counterfeit>.
7. Thayer, A., China cracks down on fake drugs, *C&EN* 90 (33):6, 2012.

conditions, but in fact caused liver, kidney, and cardiovascular damage. Additionally, according to China's state-run news agency, *Xinhua*, in February 2013, China's State Food & Drug Administration (SFDA) will introduce tougher standards for the non-active ingredients used to formulate drugs. The move to toughen standards was made after it was revealed that Chinese companies manufactured drug capsules using industrial-grade gelatin made from leather scraps containing high levels of chromium [7].

Since the passage of DSHEA in 1994, the dietary supplement industry has gone through a paradigm shift in how it is regulated. The flow of warning letters issued by the FDA will continue until such time as it is evident that companies in all distribution channels have met the intent of Congress to ensure that supplements are not only safe but manufactured according to GMP standards. At the same time, more and more dietary supplement products have invested millions in research to substantiate claims and understand the role these products may play in health promotion.

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Science in the media: explaining science to non-scientists

Crystal Snyder

As lipid researchers, we have all seen headlines that make us cringe. Whether it is the latest warning about dietary fat and heart health or overblown praise for the next big superfood, there seems to be a never-ending barrage of information about foods and our health. It is challenging enough to keep up with traditional journalistic media, but with the deluge of dietary advice from blogs, social media, and other websites, the media landscape has taken on the renegade character of the Wild West—often leaving the public caught in the crossfire.

In my double life as a writer and a scientist, I've spent a lot of time thinking about barriers to public understanding of science. Many

factors are at play, from the volume of information, to the lack of editorial gatekeepers on the Internet, to more traditional tensions between scientists and journalists over what qualifies as a good story. But perhaps the biggest challenge has less to do with the media itself and everything to do with how we, as scientists, understand our audience.

We spend much of our professional lives communicating with other scientists; virtually all of our formal training—theses, peer-reviewed publications, conferences—is geared toward an audience that knows much of what we know. We share a common technical language with other scientists, as well as a common set of values about how science works. It comes as no surprise then, that communicating with the public requires a different approach from communicating with our scientific peers. But what does that mean, exactly, and how do we learn it—and then teach it?

This was the question I had in mind in January 2012 when I was asked to develop a term assignment for Randall Weselake's

CONTINUED ON NEXT PAGE

Student myth busters

Students in the Winter 2012 graduate level lipid science class taught by Crystal Snyder challenged a variety of lipid-related myths, including those surrounding beef, cholesterol, and omega-3 and -6 fats in diet and health. A sampling is available via the digital edition of the November/December issue. To read them, log in at aocs.org/login and click on the supplement tab to the right of the page.

graduate-level lipid science class at the University of Alberta (Edmonton, Canada). [Weslake, an AOCS member, is Chair of the Biotechnology Division and co-editor-in-chief of The AOCS Lipids Library.] In previous years assisting with this course, I'd read dozens of term papers from students, typically a literature review of some topic relevant to lipid science. As an exercise in research skill development, the review papers served their purpose well enough, but given the constant media spotlight on issues related to fats and oils, this class seemed like an ideal environment in which to explore the public as an audience in its own right. I envisioned a new assignment, similar in form to a media backgrounder, that would challenge students to connect with a nonacademic audience.

The goals of the new assignment were threefold. First, I wanted the students to develop an awareness of how science news is delivered to the public, how this differs from scientific communication, and how these differences can lead to misunderstandings. Second, the assignment would force students to actively engage with the course material and demonstrate their understanding by explaining it in nontechnical terms, an exercise that requires persistent attention to audience. Third, I hoped that the assignment would find a useful outlet beyond the classroom, so the students could immediately put their new skills into practice.

Because the assignment would take the class out of the familiar realm of academic writing, it was divided into manageable pieces that would help frame the goals of the assignment and guide the students through the process. The first step was to choose a topic relevant to lipid science that had received recent coverage in the popular press, then to critically analyze a range of coverage from different sources. The analysis drew on the students' understanding of the related material from the course and their own readings from the peer-reviewed literature. The students were required to assess not only the scientific accuracy of various media reports but also their rhetorical underpinnings—how the style and structure of the writing served its intended audience. This included things such as identifying the bias of the article or the publication, recognizing the authors' use of logical, ethical, and emotional appeals, and distilling the articles' key messages in a few sentences.

To sensitize the class to the types of issues they might encounter, I chose an example of a particularly misleading article for the class to analyze during a lab period. Although the students easily recognized the scientific inaccuracies in the article, most were also able to see a hint of truth in even the most distorted facts. This facilitated an in-depth discussion of how such distortion can occur and how, armed with this understanding, scientists can begin to assert more control over how their messages are filtered through the media. Master of Science student Yuning Gao found the critical

analysis eye-opening. "Before this, I didn't doubt the information in the media so much. Now I am pickier when I read about science."

From the analysis, the students were able to identify some of the common myths or misconceptions about their topics. The next step of the assignment was to write a three- to four-page background document, targeted to a nontechnical audience, that explained and dispelled the misinformation. Students were encouraged to be creative in their presentation of the information; most chose a straightforward "mythbusters" approach, first explaining the origin of various misconceptions and then providing a more complete, evidence-based debunking of each one. This was, for most students, the most challenging part of the assignment, but it was also the most popular. "Before this assignment, I knew nothing about the media," said first-year Ph.D. student Lisha Zhao. "I didn't have any opportunity before to write this kind of thing."

The final part of the assignment was to deliver a brief seminar to the class, which was intended to simulate a public outreach activity. Like the backgrounders, the seminar was to be free of jargon and targeted to a nonacademic audience. Here, students were encouraged simply to tell their audience a story. Several students made effective use of personal anecdotes and analogies to explain complex ideas—a strategy that could serve them just as well in teaching as in a discussion with the media, or the public.

From my perspective, the most difficult part of guiding the class through this assignment was encouraging students to untear themselves from their academic perspective and consider the science from the layperson's point of view. It isn't enough merely to simplify the language that we use in describing science to the public; we must be able to frame our message in a context that the public will understand. Often, this requires that we become aware of the knowledge that we take for granted, so that we can more effectively bridge the gap between what we know and what our audience knows.

In watching the students wrestle with this challenge, I also came to realize the value of encouraging students to write in their own voices as a way of simplifying their own thought processes. For all the necessity of jargon in our routine scientific discourse, such a minimalist exercise can help identify fundamental gaps in our own understanding and ensure that we are asking the right questions, of ourselves as well as our audience. This may also be beneficial to students as they begin to tackle more complex issues in their own research.

The entire experience was uncharted territory, as much for myself as for the class, which I think added to the collective excitement about the whole project. In one semester, we may not have tamed all of the unruly myths about fats and oils in the media, but we have all taken away useful lessons from the experiment, which we hope will encourage other instructors to consider integrating nonacademic communication into their courses.

Crystal Snyder is a former laboratory manager in the Department of Agricultural, Food & Nutritional Science at the University of Alberta. She now coordinates undergraduate research at the University of Alberta. She can be reached at crystal.snyder@ualberta.ca. For more information about the lipid science course or the assignment described here, contact Randall Weslake (randall.weslake@ualberta.ca).



The growth of biobased metalworking fluids

KEY CONCEPTS

- Biobased metalworking fluids (MWF) in some cases offer superior performance compared to their conventional counterparts.
- Biobased MWF have a significantly higher flashpoint than conventional MWF, making them a safer choice for work areas at high risk for fire.
- The proper additive package can increase the performance and extend the useful life of an MWF.

Jean Van Rensselar

To keep up with the growing demand for metalworking fluids (MWF) that are made from renewable resources, formulators are developing biobased products that, in many cases, perform as well as or better than conventional MWF at a comparable price.

As straight oils, research shows that biobased MWF can perform significantly better than mineral oils. This is also true to some degree for vegetable stocks emulsified into soluble oil and semisynthetics.

The US Department of Agriculture defines “biobased” this way: “A product determined by the Secretary (of Agriculture) to be a commercial or industrial product (other than food or feed) that is composed, in whole or in significant part, of biological products or renewable domestic

agricultural materials (including plant, animal and marine materials) or forestry materials. [1]"

In the case of biobased MWF, the basestock is most likely soybean, rapeseed (canola), sunflower, or corn. John Hogan, technical service manager for metalworking additives for The Lubrizol Corp. in Wickliffe, Ohio, USA, explains, "When selecting the best vegetable basestock, there are several factors that need to be considered: end-use application of the product, oxidative stability, saturation level, and additive selection. Availability of basestocks in different countries, in different seasons, and over the long term is important to consider in the initial selection of the basestock."

As with most biobased products, in order for MWF to perform as well as their nonbiobased counterparts, chemicals need to be added that compromise the biobased nature of the fluid to differing degrees.

Hogan says, "The use of renewable resources may be desirable, but that does not necessarily make a product green or sustainable. When evaluating the use of renewable materials relative to petroleum products, lifecycle analysis and total environmental impact must be considered. This means the energy consumed and the waste emissions along the entire spectrum of the product must be considered: gathering raw materials, manufacturing the product, distributing the product, product use, and disposal/recycling of an MWF must all be considered in evaluating its environmental impact."

Although biobased MWF were once a poor substitute for conventional MWF (offering little advantage other than safety and significant performance disadvantages), new formulations, genetically modified crops, and additives are leading to performance that is at least comparable and, in some cases, superior to conventional MWF. This means that facilities can finally use biobased MWF to protect their workers and improve performance without incurring significant additional cost.

Craig Mott, executive vice president of Colonial Specialty Chemical in Tabernacle, New Jersey, USA, explains, "Market drivers include OEM [Original Equipment Manufacturer] requirements, federal government mandates, and the green movement. Also, additive/surfactant technology is helping the performance of green fluids; this means there are more vegetable oil options, such as improved oxidation, available for the formulator."

Formulators, distributors, and users are confident that biobased MWF, even with a slew of additives in them, are safer and perform as well as or better than conventional MWF with little price disparity.

In addition to reduced environmental impact, there are genuine performance advantages to using biobased MWF. But there are still a few issues to be worked out before they become generally accepted (and preferred) alternatives to conventional MWF.

Advantages

In addition to helping to protect worker health and the environment, biobased MWF have a number of performance advantages. Viscosity, lubricity, and flashpoint are three areas where biobased MWF really shine. This is especially true for straight biobased oils and less so for solubles and synthetics.

Greater viscosity stability. Vegetable oil has a high natural viscosity. As the machining temperature increases and as ambient temperatures fall, biobased MWF tend to hold their viscosity levels better than conventional MWF.

MWF quick primer

MWF have many applications but all have the basic purpose of lubricating and cooling the work piece–tool interface, flushing scrap and residue from the work area, and improving the surface finish on end products.

MWF are categorized into the following four classes:

- Straight/neat/cutting oil. Ultra-refined oils that are not diluted with water but may contain additives.
- Soluble oil. Composed of anywhere from 30% to 85% severely refined basestock, water, and emulsifiers.
- Semisynthetic fluids. Containing 5% to 30% severely refined oils, 30% to 50% water, and several additives.
- Synthetic fluids. Composed of compounds that do not contain any petroleum oil.

Each of the four classes also may contain additives such as biocides, corrosion inhibitors, extreme pressure chemicals, defoamers, emulsifiers, stabilizers, dispersants, and dyes.

Better lubricity. Unlike conventional MWF, biobased oils have a slight polar charge. This charge naturally attracts the oil to metallic surfaces and is resistant to being wiped off. This polarity also leads to better corrosion protection.

Higher flash, fire, and smoke points. Biobased MWF have a significantly higher flashpoint than conventional MWF—around 200°F (~93°C) higher. This makes them a safer choice for all workspaces, especially those that are tightly enclosed and/or near open flames.

Workers at the US Naval Air Depot at Cherry Point, North Carolina, are seeing the benefits of biobased MWF every day. There are about 160 machines at the facility using MWF. After giving a prototype biobased MWF a fair trial, users came to the following conclusions:

- The flashpoints of straight oils used in the past were 350–400°F (177–204°C). The prototype has a flash point of 640°F (316°C). Because of its high flash point, the biobased prototype provides better heat dissipation and produces less smoke when machining.
- The graphite-like material from the molybdenum disulfide source in the prototype allows metals to be processed with less friction and less torque compared to the MWF they were previously using.
- The prototype provides better tool life compared to the MWF previously used.
- Biobased MWF lead to a safer and healthier environment [2].

Challenges

Poor oxidative stability, poor hydrolytic stability, microbial growth promotion, warranty issues, and higher cost are the five main drawbacks to using biobased MWF. However, with the right formulation expertise, these challenges are easily overcome.

Poor oxidative stability. This is a significant concern because, among other undesirable effects, it causes expensive tools to wear more quickly. Lou Honary, professor and director of the National

Ag-Based Lubricants Center, University of Northern Iowa (UNI-NABL Center) in Waterloo, Iowa, USA, explains, “While oxidation stability remains a big concern in most applications where vegetable oils are used, the oils is carried out with the chips or as a film on the machined part, thus the residency of the oil is shorter in the machine than applications like gear oil or hydraulic oils. Nevertheless, oxidation stability is important because most other attributes like solubility and compatibility with additives are easier to address.”

contacting manufacturers, industry associations, and service professionals to request information about and ultimately resolve warranty issues. As additional information becomes available on the project, the USDA will post it on the bioPreferred website: www.biopreferred.gov [3].

Cost. The level of cost disadvantage depends on the basestock. In addition, additives required for equalizing oxidative/hydrolytic stability and microbial growth issues add to the cost. But as petro-

“NEW FORMULATIONS, GENETICALLY MODIFIED CROPS, AND ADDITIVES ARE LEADING TO PERFORMANCE THAT IS AT LEAST COMPARABLE AND, IN SOME CASES, SUPERIOR TO CONVENTIONAL MWF.”

Poor hydrolytic stability. Conventional MWF are resistant to hydrolytic reactions because they do not contain ester linkages and thus do not hydrolyze (break down). This is not the case with biobased MWF. The good news is that a genetically modified basestock that is naturally more hydrolytically stable is currently being developed. Other ways to overcome the poor hydrolytic stability are through additives and/or chemical modification.

Microbial growth. Unlike conventional MWF, biobased MWF tend to promote microbial growth—the oil biodegrades in the machinery. This compromises performance and creates a noxious odor. The addition of an approved antimicrobial can minimize these problems.

Prohibitive manufacturer warranties. In some cases, using biobased MWF could void the manufacturer’s warranty. The US Department of Agriculture (USDA), in an effort to promote responsible use of biobased products, is working with OEM on the issue of maintenance warranties. Specifically, the USDA has been

leum prices continue to escalate and some countries offer tax incentives for using biobased products, the cost differential is becoming less pronounced.

Mott says, “As far as the cost of biobased MWF, when compared to conventional MWF, there is still a premium, but this gap narrows considerably when petroleum trades at \$120 a barrel. Vegetable oils have also moved up in price but not as much as petroleum.”

Formulation

The development of biobased MWF has been a research focus for scientists at the UNI-ABIL research program since 2002. The center has been formulating alternative, biobased MWF using renewable crop basestock to minimize environmental, health, and safety concerns without compromising performance.

Honary says they have discovered that while certain additives like sulfur used for wear protection are effective regardless of the base oil, it may be necessary to use different antioxidants for vegetable oils than those used for mineral oils. The same logic applies to emulsifiers, antibacterials, and pH improvers.

He adds that vegetable oils have two main areas that need consideration during formulation and use: oxidative stability and cold-temperature performance. “Since most metalworking applications are indoors, the cold temperature issue becomes relatively irrelevant.” Honary explains: “This makes it easier to focus on improving the oxidative stability while formulating MWF.”

Mott says that for water-based systems, emulsion stability is the most common customer concern they hear about. “The end-user cannot tolerate their MWF splitting out in use,” he says. “This is where newer surfactant technology has really helped the formulator. These new surfactants are doing a very good job in keeping the emulsions very stable in both small and large central systems.”

Formulation is a complex science. Hogan explains, “Vegetable basestocks normally require somewhat higher emulsifier content with a different hydrophilic-lipophilic balance for water-extendable products. Selection of emulsifiers and sources of alkalinity

information

- [1] Farm Security and Rural Investment Act of 2002, Section 9001.
- [2] Naval Air Depot Cherry Point Prototypes Alternative Metal Working Fluid, 2006, summary available at: www.denix.osd.mil/sustainability/upload/DoN_Leadership-in-Biobased-Products-Usage.pdf.
- [3] 7CFR Part 2902: Designation of Biobased Items for Federal Procurement; Final Rule, May 14, 2008, www.epa.gov/epp/pubs/guidance/fr73no94.pdf.
- [4] Stear, M., Metalworking fluids—clearing away the mist? *Ann. Occup. Hyg.* 49:279–281 (2005).
- [5] <http://www.epa.ohio.gov/ocapp/p2/fact11.aspx>.



are important to consider in order to prevent interactions with the basestock over time that can degrade emulsion stability.

“Higher quality, high-oleic content basestocks are generally easier to emulsify and yield more stable products,” Hogan says. “Longer fatty chains and increased unsaturation generally require higher emulsifier treat rates. Inclusion of antioxidants is essential to limit oxidation and maintain emulsion stability. Selection of the right antioxidant is critical not only for limiting oxidation but also because it can impact aesthetics of the fluid.”

He also points out that while there is a great deal of emphasis on the use of vegetable basestocks to formulate green MWF, there also are additive technologies based on renewable materials. The new additive technologies are designed to provide the lubrication properties of vegetable oils and are easier to utilize in some water-extendable fluids.

Gene Tripp, sales manager for Performance Biolubes in Cedar Falls, Iowa, USA, says, “Finding the right additives and getting them to remain in the formulation are the real challenges. When it comes to additives, the fewer you use the better. The more ingredients you use and the higher quantity only adds to the cost.”

Fuchs is a formulator and proponent of synthetic esters. The company’s product manager for Cutting & Grinding Fluids, Jonathan Chow, explains: “Variability of supply, hydrolytic stability and oxidation stability have led us to prefer synthetic esters derived from natural vegetable oils. These give much better robustness and consistency. The higher price can be justified by superior performance.”

Recycling, waste treatment, and disposal

While the term “recycling” generally refers to collecting and repurposing, when applied to MWF the term refers to the common practice of continually treating and reusing MWF in the machinery. Once the MWF has reached the end of its useful life, it is almost always treated and disposed of—not recycled in the traditional sense. This is due to the fact that, in most cases, it has already been recycled as much as possible in the machinery.

Recycling

The goal of any MWF recycling program is to maintain a stable fluid at optimal performance as long as possible. No matter whether it involves conventional or biobased MWF, the goal is inherently green since it significantly reduces the amount of waste product in the environment. The same benefits of recycling conventional MWF apply to biobased MWF as well (reduced use, little or no equipment downtime, and the like). But in the case of biobased MWF, aggressive recycling also reduces the strain on natural resources such as crop production.

“If the oil is stable, the recycling of vegetable oil can be done the same as that of the mineral oil-based MWF,” Honary says. “That is, using centrifuge or other means of recovering the base oil for reuse.”

CONTINUED ON NEXT PAGE

He adds that this is assuming that the vegetable oil does not oxidize during use, which could polymerize it and render it unrecyclable.

MWF can only be recycled a limited number of times before they reach the point of disposal. The factors limiting coolant recycling are biological hardness, selective depletion, and tolerance to water hardness minerals.

For operations that generate a relatively small amount of MWF wastewater, contract hauling or evaporation probably is the best disposal method from cost and practicality perspectives.

Before disposal, facilities with spent MWF need to determine whether the MWF are hazardous. A substance is considered hazardous by the US Environmental Protection Agency if it contains any

“IN ADDITION TO REDUCED ENVIRONMENTAL IMPACT, THERE ARE GENUINE PERFORMANCE ADVANTAGES TO USING BIOBASED MWF.”

Treatment

Waste treatment and disposal of spent MWF involve first removing the water and then isolating the hazardous components. The remaining product is then hauled away for incineration or recycling. Costs of fluid handling can account for more than 15% of total machining costs. [4]

When treating biobased MWF, the following three waste treatment processes used for conventional MWF apply.

Physical treatment. Various physical treatment methods are used effectively to treat MWF for disposal. Evaporation is a common treatment for small amounts of oily wastewater—fewer than 3,000 gallons (more than 11,000 liters) a day. The process uses heat to remove water from the used fluid, which has the effect of concentrating the fluid. While this avoids the necessity for sewer discharge, it may require an air discharge permit. Another common physical treatment method that is best for moderate waste fluid volumes is membrane separation via ultra-filtration or reverse osmosis.

Chemical treatment. Chemical treatment uses inorganic or organic chemicals to destabilize or separate emulsions of MWF waste. From a cost perspective, chemical treatment makes more sense for large volumes of MWF waste. Chemical treatment produces a by-product that, with further treatment, will recover some fluid. Unlike physical treatment, it will treat and remove metals.

Biological treatment. The high organic content of biobased MWF makes them excellent candidates for treatment via bacterial degradation. But as with all other MWF, the high oil content of these fluids means that other methods also must be used. Biological treatment for the reduction of organics typically follows either chemical or physical treatment.

Nonhazardous waste can be treated on-site using one of these methods, but ultimately they must be disposed of at a treatment facility or municipal sanitary sewer system (with permission).

Disposal

Even though biobased MWF are biodegradable, because of additives, dissolved metals, and other contaminants, there are still disposal issues. The decision to dispose is usually based on either time in use, loss of key properties, or contamination (including biological contaminants) that exceeds a prescribed limit.

hazardous material. However, metal cuttings removed from MWF are exempt from hazardous waste disposal requirements and can be recycled.

In the case of an inadvertent release into the environment with no intervention, straight mineral oil biodegrades anywhere from 15–35%, and straight vegetable oil biodegrades 70–100%.

The bottom line

What would seem to be a relatively small expenditure for most companies can have a big impact on the bottom line. Researchers at the UNI-NABL research facility estimate that MWF comprise less than 5% of total plant expenditures yet can impact more than 40% of the plant’s operational budget.

Honary does not consider biobased MWF to be a novelty anymore, pointing out that the UNI-NABL Center licensed several biobased MWF and coolants for commercial use in 2000, and the products are currently in the market performing successfully.

Mott also sees mostly blue skies for biobased MWF. “The industry is moving to green fluids to better comply with OSHA [Occupational Safety and Health Administration] standards and improve on best practices,” he says. “Vegetable-based fluids offer improved worker safety and health benefits as well as economics in terms of tool life. From a right-thing-to-do viewpoint, I also believe individuals and companies want to go green when they can because it just makes sense to do so. It is much more a part of the discussion today than it was 10 years ago. I see continued growth for vegetable oils. Year-over-year sales for vegetable-based oils used as basestocks have been a good area of incremental growth in our industry.”

Experts agree that with additives being roughly equal, there is a definite environmental advantage to going green.

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Tourisme Montréal

Attendees will be greeted by a metropolis in full bloom when they arrive at the 104th AOCS Annual Meeting & Expo in Montréal, Québec, Canada, April 28–May 1, 2013. Embraced by the majestic St. Lawrence River, Montréal reawakens each spring as the days get longer, funky cafés and designer restaurants open their outdoor patios, and an increasing number of locals and visitors stroll through the city's charming neighborhoods.



Those who prefer two-wheel travel are in for a treat. Montréal was voted the No. 1 cycling city in North America by *Bicycling* magazine. The city's public, self-service, bike-sharing system, BIXI (a contraction of the words bike and taxi), makes it easy to rent a bike and ride the hundreds of kilometers of bike paths in and around Montréal's central core. With more than 5,000

bikes available to rent, this eco-responsible way of commuting and visiting attractions has been so successful that other major cities, including Boston, Melbourne, and London, have adopted it to enhance their own public transportation networks.

A worldly history

Centuries-old spirit and resolve are what make Montréal the exceptional place it is today. First influenced by Amerindian, French, British, and American settlers, and later by immigrants from all corners of the globe, the city has a rich history that has given rise to the vision, drive, and determination that continues to fuel the progressive attitudes and creativity of its citizens.

Originally called Ville-Marie, or "City of Mary," Montréal takes its present name from Mount Royal, the triple-peaked park and nature reserve designed by Frederick Law Olmstead, the landscape architect behind New York City's famed Central Park.

An important fur trade hub in the early 19th century, Montréal established itself early on as an economic and cultural powerhouse, and it remains an international center for trade, commerce, and exchange. The city has further distinguished itself throughout the decades as a first-rate international host, thanks to major events such as Expo 67 and the 1976 Summer Olympic Games, as well as annual large-scale happenings, such as the Festival International de Jazz de Montréal, the Just For Laughs Festival, and the Canada Grand Prix—Formula 1.

A culture of cool

A home to no less than 120 ethnic groups, Montréal is where people speak not only English and French, but many other languages as well. This cosmopolitanism has resulted in a vast cultural offering ranging from large museums like the Montréal Museum of Fine Art, the Musée d'art contemporain, and the Canadian Centre for Architecture to numerous independent galleries located at, among other places, the Belgo Building downtown, the revitalized Griffintown quarter, the hip Mile End and Plateau Mont-Royal districts, and the city's renowned Golden Square Mile.

Montréal has seen significant transformations in recent decades. Investments made in the picturesque Old Montréal neighborhood have attracted a variety of new boutique hotels, restaurants, galleries, and stores. The revitalization of the Quartier international (International District), which included the convention center expansion project, has earned international acclaim. Equally impressive is the Quartier des spectacles (Entertainment District), which unites an unprecedented number of cultural institutions, performance settings, and public spaces into a well-defined urban area covering one square kilometer. A showcase of Montréal's creative spirit, the Quartier des spectacles brings together innovative lighting design displays, art exhibitions, 80 cultural locales, and 30 performance halls. Within the district is La Vitrine culturelle de Montréal (Montréal's cultural window), an excellent place to gather information, calendars, and tickets for current shows.



Other sites, such as Montréal's Space for Life Museums—also known as the Botanical Garden, the Biodôme, the Insectarium, and the Planetarium (scheduled to open in spring 2013)—reflect the city's commitment to the environment, biodiversity, and sustainable development.

A foodie haven

In 1881, Mark Twain said about Montréal, "This is the first time I was ever in a city where you couldn't throw a brick without breaking a church window." In today's Montréal, you probably couldn't throw a brick without breaking a restaurant window. Here, food is a passion, pursued in anywhere from five-star establishments to Parisian-style bistros to cozy diners. Montréal eateries represent the largest number of restaurants per capita in all of North America, with over 6,000 gastronomic venues within the municipality. Inspired by the culinary traditions of the city's cultural communities, the abundance of local products, and chef ingenuity, Montréal has got the "global village" down pat when it comes to an

epicurean diversity that includes the city's legendary smoked meat and unique fresh-from-the-oven bagels. With no shortage of available culinary delights, you'll end up leaving Montréal knowing that there will be so much more to taste when you return.

Tourisme Montréal is a private, nonprofit organization, founded in 1919, which is comprised of more than 750 members and partners from Montréal's tourism industry. Its mission is to lead the collective effort to position Montréal as a premier destination for business and leisure travel.

AT A GLANCE

104th AOCs Annual Meeting & Expo

April 28–May 1, 2013

Montréal, Québec, Canada

AnnualMeeting.aocs.org

Online registration opens January 7, 2013

Welcome New Members

AOCS is proud to welcome our newest members.

New and reinstated members joined from July 1–September 30, 2012.

Manish C. Agarwal, Petrochem Specialties

Charles Bayer

Ken Bayless, Frito-Lay Inc

Ken T. Bennae, Florida Institute of Technology

Krishnendu Bera, Institute of Genetic Engineering (WBUT)

Gregor C. Burdeos, Tohoku University

Haskell Cooke, Crown Iron Works Co

Leqi Cui, University of Massachusetts, Amherst

⊕ Christopher P. Dooley, Industrial Specialty Chemicals Inc

Luis A. Espinoza, Louisiana State University

Arnetta Fletcher, University of Maryland

Tetyana Forostyan, University of Utah Medical School

Steven Godin, International Fiber Corp

⊕ Steven E. Hill, Kraft Foods Inc

Xinjie Lin, University of Guelph

Concepcion Lopez, Universidad Autonoma de San Luis Potosi

Rashudy F. Mahomedradja, Rijksuniversiteit Groningen

Yingyi Mao, University of Massachusetts

Deborah A. McRoberts, AAK USA

Mutari Musah, Auchi Polytechnic

Stephen M. O'Connor, Lubrizol Advanced Materials

Zhenming Qiu, Chinese Academy of Sciences

Terrin L. Senez, Nutramax Labs Inc

John David Sexton, Clean Control Corp

⊕ David B. Smith, Dilling Group Inc

Allison R. Smither, Louisiana State University

Greg A. Strayer, Bunge North America

Bill Swann, Nuseed Pty Ltd

Lin Tang, Sichuan University

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Arun Kumar Vadamodal, Andhra University

Thidarat Wilaisuwan, Kasetsart University

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All members contribute to the success of the Society while furthering their professional goals.

Ralph T. Holman (1918–2012)

The following reflection on the life and character of Ralph Holman was written for *inform's* periodic Giants of the Past series.

G.R. List and Douglas Bibus

Dr. Ralph Holman was living proof that those who seek God are rewarded. He attributed any success he had during his long and distinguished career to prayer, seeking God's direction, and waiting for answers.

Holman grew up in Minneapolis, Minnesota, USA, as a shy young man. His was a modest hard-working family; and when his father's salary as a streetcar conductor was cut during the Great Depression, the family lost their home. His chances of going to college seemed remote. Thus, taking a job as a janitor at a Minnehaha (now a suburb of Minneapolis) church, which paid \$3–5 per month depending on the season, was providential. The pastor, sensing young Holman's intellect, inquired whether he would be interested in attending Bethel College. Since his grandfather Nils Holman had graduated from Baptist Union Theological Seminary (a precursor of Bethel Junior College, now Bethel University) in 1887, the idea appealed to him. After meeting with Pastor Nelson and Professor C.E. Carlson, the dean of Bethel Junior College, it was decided that school policy gave a free semester to honor students and Holman was allowed to pay off his debts as he went along. Holman remarked, "Thank God for my pastor and the dean who changed my life from black to bright." At Bethel, Holman blossomed as a student.

After the first year, Holman realized he needed more financial help, yet during the summer of 1936, employment was almost impossible. Again, Dean Carlson intervened by allowing Holman to work off the next year's tuition in advance by preparing school buildings for the upcoming school year. Holman gladly accepted the job paying \$0.15 an hour. Not everything was serious work or study. He recounted the day he observed a mouse in a wastebasket and put it to sleep with ether. He tied the rodent to a string, brought it into the classroom, and released it, much to the dismay of the girls shrieking and scrambling for their desktops. Despite this incident, Holman graduated from Bethel in 1937 and two years later received his B.S. degree in chemistry from the University of Minnesota. He then earned a master's degree from Rutgers University (New Brunswick, New Jersey, USA), and finished his graduate work with a Ph.D. in physiological chemistry at the University of Minnesota (1944).

After several post-doctoral years in Sweden, Holman taught at Texas A&M (College Station, USA) and the Mayo Medical School (Rochester, Minnesota) before assuming the Executive Directorship at the Hormel Institute, an affiliate of the University of Minnesota, a position he held for 30 years. During his long and illustrious career,



he published more than 400 publications. He was the founding editor of the journal *Progress in Lipid Research*.

Perhaps his most significant discoveries were in the area of lipid metabolism and the competition between omega-3 and omega-6 fatty acids. These findings are considered to have had a profound effect on diet and nutrition research throughout his lifetime.

Holman received numerous honors and awards. He was the first Bethel graduate to earn a Ph.D., and his work on omega-3 acids earned him membership in the National Academy of Science in 1981. He was quite active in the American Chemical Society and served as AOCS president in 1974. He was the recipient of the AOCS Alton E. Bailey Award (1972) and the Supelco/AOCS Research Award (1978). He was elected as an AOCS Fellow in 2004. Holman also received the AOCS A.R. Baldwin Award (2001) in recognition of his service in bringing biochemists into the society. The health and nutrition division of AOCS honored Dr. Holman with an achievement award named for him and made its first award to him in 2004.

IN MEMORIAM

RALPH THEODORE HOLMAN



Holman

Former AOCS president and National Academy of Sciences member Ralph T. Holman died in Albert Lea, Minnesota, USA, on August 15, 2012, at the age of 94. His wife Karla had preceded him in death in 2003, after 60 years of marriage. Their son, Nils (Ted) Holman, survives, as well as nieces, nephews, cousins, and a host of friends and colleagues.

Holman was a pioneer in the area of essential fatty acid research and nutrition, making several fundamental discoveries about the metabolism of fatty acids. He is regarded as the "Father of Omega-3 Fatty Acids" for his discovery of their essential nature, their metabolism, and competition with omega-6 fatty acids.

His higher education began at Bethel Junior College (now Bethel University) in St. Paul, Minnesota, and he received his B.S. in chemistry from the University of Minnesota in 1939, his M.S. at Rutgers University in physiological chemistry in 1941, and his Ph.D. at the University of Minnesota in 1944. At the end of World War II, he went to Sweden for two postdoctoral years of research; during his time there, he became the first person to crystallize lipoxigenase, a significant enzyme in the inflammatory process.

Holman returned to the United States in 1948 to teach and do research at Texas A&M University (College Station, USA). He joined the Hormel Institute, an affiliate of the University of Minnesota, in 1951, which at the time was pioneering food stabilization technology.

He spent the rest of his working life with the Hormel Institute, serving as its director from 1975 to 1985. There, he conducted and guided research on the essentiality of fatty acids in the diet and the best ways to increase omega-3 and decrease omega-6 fatty acid consumption. Holman retired in 1988, although he continued in the laboratory for several years after that. He also served on the board of directors for Hormel Foods.

During his life, Holman published over 400 journal articles, research reports, and book chapters, as well as editing both journals and textbooks.

Holman joined AOCS in 1946, and shared his skills with the organization. He served as an associate editor for *Lipids* from its first issue in 1966. He became editor in 1974 and continued in that position until 1985; in 1986–1987 he was co-editor of *Lipids*. He also served on the *Lipids* editorial advisory board until 1998.

As a member-at-large, Holman served on the Governing Board of AOCS from 1968 to 1970. He was elected secretary of the organization in 1972, vice president in 1973, and president in 1974. He continued on the Governing Board until 1978.

His work was widely recognized in addition to his election to the National Academy of Sciences. He received the Borden Award in Nutrition in 1966, awarded by the American Institute of Nutrition. From AOCS he received Alton E. Bailey Award (1972), the Award in Lipid Chemistry (1978), and the Lifetime Achievement Award, named after him, in 2004.

Holman practiced what he preached regarding the consumption of omega-3 fatty acids. According to his former student, friend,

and colleague Doug Bibus of Lipids Technologies LLC (Austin, Minnesota), "Ralph had one of the highest omega-3 levels that we have ever measured, likely due to his daily intake of sardines, fish, and vegetables."

Bibus also said, "He taught me science but even more importantly how to be a person."

BRUCE EUGENE McDONALD

Bruce McDonald joined AOCS in 1973 and died on April 27, 2012. He is survived by his wife Judy, with whom he would have celebrated his 50th anniversary in June 2012; five children; seven grandchildren; and four sisters.

McDonald was born on the family farm in Chailey, Alberta, Canada, in 1933. Through involvement with the local 4-H Club, he developed a passion for research. He earned his B.S. in agriculture from the University of Alberta (Edmonton) in 1958, and an M.Sc. in nutrition in 1960. He completed his Ph.D. in nutrition and biochemistry at the University of Wisconsin (Madison, USA) in 1963. He then received a post-doctoral appointment in nutritional biochemistry from the University of Illinois (Urbana-Champaign, USA).

In 1964 he accepted a position with Macdonald College, McGill University (Montréal, Québec, Canada). When McDonald's department head at McGill moved to the University of Manitoba (Winnipeg, Saskatchewan, Canada) in 1968, McDonald followed. He continued there for the next 30 years.

McDonald was the author of several books, many book chapters, and close to 60 publications. His research on the nutritional characteristics of canola, conducted with Vivian Bruce, was critical to its successful development throughout the world. Following his retirement, he became executive director of the Manitoba Health Research Council, a position he held until 2004. McDonald also served in a number of appointments for Health Canada/Heart and Stroke Canada and the Canadian Agri-Food Policy Institute.

From 1988 to 1998, McDonald chaired Dietitians of Canada committees that developed guidelines for undergraduate education in nutrition and dietetics in the 21st century. He was known for his research on the nutritional properties of canola oil, in particular the effect of its fatty acid composition on cardiovascular risk factors and in 2005 served as secretary to the National Trans Fat Task Force.

TOSHIRO NISHIDA

AOCS emeritus member Toshiro Nishida died on August 15, 2012, in Urbana, Illinois, USA. He joined AOCS in 1964 under the sponsorship of Fred A. Kummerow and Edward G. Perkins.

Nishida served on the faculty of the Department of Food Science and Human Nutrition at the University of Illinois at Urbana-Champaign for more than 40 years. During that time, he made fundamental research contributions to our understanding of the pathways, enzymes, and proteins associated with lipid metabolism and transport. He was internationally recognized for his research contributions and was a recipient of the National Institutes of Health Award of Merit and the College of Agricultural, Consumer and Environmental Sciences Paul A. Funk Award. He taught courses related to food chemistry and nutrition, as well as a graduate course on the metabolism of lipids. He also was an active part of the off-campus M.S. program in food science

in Chicago. Nishida trained many students who went on to successful and productive careers in science.

He received his Ph.D. at the University of Illinois in Food Science in 1956 under the direction of Fred Kummerow. Before that, he was a research assistant at the Osaka Prefectural University (1947–1949) and carried out industrial research for the Osaka Soda Co. (1952–1953).

When Nishida retired in 1998, his friends, family, and colleagues established the Toshiro Nishida Fellowship for Excellence in Research. He and his wife and research associate, Hiro, continued to support that fund, which was intended to further opportunities for both undergraduate and graduate students to excel in research.

TIMOTHY A. KELLY

Tim Kelley died unexpectedly on August 3, 2012, at the age of 50 in Maineville, Ohio, USA.

He received his B.S. in chemical engineering from the University of Michigan in 1984. He worked for Procter & Gamble (Cincinnati, Ohio) for almost 20 years, primarily in Bar Soaps. His roles over the years included technical engineer, operations manager, quality assurance manager, and technology director.

Kelly joined VVF, a contract manufacturer of personal care and oleochemical products, in 2009, where he served as vice president of new business development. In this position, he was responsible for implementing the growth of the VVF oleochemical business (fatty acids, fatty alcohols, glycerin, and soap chips) and private-label bar soaps and antiperspirant/deodorant products in North America.

He was recognized as an expert on bar soap technology and wrote the chapter "Continuous saponification and neutralization systems" in *Soap Manufacturing Technology*, edited by L. Spitz, which was published by AOCS Press in 2009.

Kelly joined AOCS in 2007. He was also a member of the American Institute of Chemical Engineers and the American Cleaning Institute.

He is survived by his wife Miriam, his children Michael, Timothy, and Kristi; his parents; and his six siblings.

FRANK HENRY PASSALAQUA

AOCS Emeritus Member Frank H. Passalaqua, of Conroe, Texas, USA, died on August 21, 2012, at the age of 92. His daughter Lynda, three grandchildren, four great-grandchildren, and two siblings survive. His wife, Anne, and daughter Sandra preceded him in death.

Passalaqua was born in McComb, Mississippi, USA, on November 18, 1919, the son of Sicilian immigrants. He served in the US Army during World War II as a paratrooper.

The Passalaquas moved to Conroe in the early 1960s, where he served as executive vice president of Sparkler Filters Inc., which is devoted to the liquid filtration industry. He developed three US patents while he was with Sparkler, and later began working independently for Industrial Filter and Pump Co. (Chicago, Illinois, USA).

He joined AOCS in 1963, and was also a member of the International Oil Mill Superintendents Association.

Holman's laboratory at the Hormel Institute was a beacon for scientists wishing to do post-doctoral work, and indeed many outstanding scientists of today at some point collaborated with him during their careers.

In retirement, Dr. Holman and his wife Karla lived in a neatly furnished home in Austin, Minnesota, where he grew orchids and she grew African violets. As Karla's health deteriorated, Ralph cared for her until she entered St. Mark's Lutheran Home. When not visiting her, Holman spent his latter years writing his memoirs.

Perhaps the best testimony to Holman came in 1998 when Bethel College honored him as the Distinguished Alumnus of the Year. After speaking for 15 minutes, he was given a five-minute standing ovation. Following the ceremony, Dr. and Mrs. Holman were taken to see the excavation for a new chemistry lab. Signs at the door read: "The Holman Chemistry Laboratory."

In 1991, Ralph and Karla Holman established the Endowed Program in Chemistry at Bethel. He said, "Bethel Junior College has played a major role in my early education, teaching me the foundations of a broad range of knowledge. I learned from Bethel's faculty and students that one can be a Christian in any walk of life, and they demonstrated to me the fundamentals of such a life. The foundations of chemistry which I learned there have served me well for many years, and I am grateful to Bethel for getting me started."

When asked the secret to a long and productive life, Holman replied, "Eating fish and omega-3 acids, vegetables, and fruit is the Gospel I preach." He concluded by saying that much time in prayer and academic preparation turned the nebulous hope of a young boy from Minneapolis to the concrete realities of an accomplished biochemist. Holman attributed a study of history, needed for graduation from Bethel, as instrumental in forging his understanding of life and as such, hoped his memoirs would be an inspiration to others.

When informed that he had been elected to the National Academy of Science, as was his hero, Thomas Edison (1927), Holman remarked, "I picked the right role model." As we see it, Dr. Ralph Holman was an excellent role model as well. After 75 years of studying family history, Holman contended that love of family is more important than the science of omega-3 acids.

Gary List is the primary contributor to inform's popular Giants of the Past series. He currently works as a consultant after retiring from the US Department of Agriculture, Agricultural Research Service, National Center for Agricultural Utilization Research in Peoria, Illinois, USA. He can be contacted at glist@telestar-online.net.

Doug Bibus is a scientist in the field of fatty acid nutrition. He spent 16 years with Dr. Holman at the Hormel Institute, initially as a student and then running his laboratory. Doug and Ralph enjoyed many years of defining omega-3 status in populations around the world. Doug currently runs a fatty acid and lipid analysis group, Lipid Technologies, LLC in Austin Minnesota, USA. He can be contacted at doug@lipidlab.com.

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Extracts & Distillates

Analysis of EPA and DHA and distinction between fish oils and concentrates

Pan, B., *Lipid Technol.* 24:178–180, 2012.

Fish oil is the major dietary source of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). Adequate dietary intake of these n-3 PUFA is beneficial to reduce the risks of cardiovascular mortality, prostate cancer, and neurological disorders in children and adults. There is a surge in demand for fish oil in the functional food market. Microencapsulation of fish oil is the trend to improve its stability and sensory quality. The EPA and DHA content of the fish oil products may vary markedly from the label due to their susceptibility to oxidation. Quick and reliable methods other than the AOAC BF3 method have been exploited to quantify EPA and DHA in the encapsulated fish oil and the microencapsulated powdered products such as infant formula. This article describes a method to differentiate the ethyl-ester form from the TAG form of EPA and DHA in encapsulated fish oil which may be a mixture of natural triacylglycerol enriched with ethyl esterified EPA and DHA. A method is recommended due to the difference in apparent potency of these two esterified forms, which may be a concern in infant formula and elsewhere.

Lipase-catalyzed transesterification to remove saturated MAG from biodiesel

Padhi, S.K., *et al.*, *Eur. J. Lipid Sci. Technol.* 114:875–879, 2012.

Saturated MAG (SMG) are known to be present in FAME intended to be used as biodiesel. These SMG can strongly affect the properties of biofuels such as the cloud point (CP), and they have been implicated in engine failure due to filter plugging. It is shown here that lipase G from *Penicillium camemberti* can be efficiently used for the transesterification of SMG to fatty acid methyl ester and glycerol even in the presence of the bulk biodiesel. Thus, in samples

of commercial biodiesel to which glycerol monostearate (GMS) and glycerol monopalmitate (GMP) had been added, their levels were enzymatically reduced from 2% (w/v) to 0.22% (w/v) for GMP and 0.14% (w/v) for GMS as confirmed by GC-MS analysis.

Effects of oxygen and antioxidants on the lipid oxidation and yellow discolouration of film from red tilapia mince

Tongnuanchan, P., *et al.*, *J. Sci. Food Agric.* 92:2507–2517, 2012.

Generally, biodegradable films from fish muscle protein become yellow after preparation. This discoloration is more likely associated with lipid oxidation and can be prevented by minimizing the oxidation in the films. Thus, the effects of oxygen and antioxidants on lipid oxidation and yellow discoloration of film from red tilapia mince during storage were investigated. Both films prepared at pH 3 and 11, and kept under atmosphere containing 100% N₂ had the lowest TBARS value with the concomitant lowest *b** and ΔE^* values during storage ($P < 0.05$), when compared with other films kept in air and a 100% O₂ atmosphere. Films prepared at pH 3 and incorporated with antioxidants (Trolox and catechin) at all levels (100, 200 and 400 mg L⁻¹ film-forming solution) had the lowest TBARS value, *b** and ΔE^* values during storage, indicating the retardation of lipid oxidation and yellow discoloration in films. Nevertheless, films prepared at pH 11 had no difference in TBARS values, in comparison with control film, regardless of antioxidant incorporation. Coincidentally, increases in *b** and ΔE^* values were observed in those films. Lipid oxidation was the main factor inducing yellow discoloration of film exposed to oxygen and the incorporation of antioxidants in film prepared at acidic pH was able to prevent yellow discoloration of resulting film.

Serum fatty-acid composition and the risk of Alzheimer's disease: a longitudinal population-based study

Rönnemaa, E., *et al.*, *Eur. J. Clin. Nutr.* 66:885–890, 2012.

It is unknown if a specific fatty-acid composition influences the development of Alzheimer's disease (AD). Nutrition is a possible target for prevention of dementia and

especially omega-3-based fatty acids (n-3 FAs) have previously been suggested to be beneficial for cognition. The objective was to ascertain whether serum FAs predict the risk of incident AD and dementia in a longitudinal population-based cohort. Uppsala Longitudinal Study of Adult Men started in 1970. The proportions of FAs in serum cholesteryl esters were estimated in men ($n = 2009$) who were 50 years old at baseline. During a 35-year follow-up time, 213 men had developed dementia, out of which 91 had AD. The associations were analyzed with Cox proportional hazards and logistic regression; adjusted for age, education and vascular risk factors. Subjects with a higher proportion of saturated FAs had a decreased risk of AD in crude and multi-adjusted models (hazard ratio for 1-s.d. increase in palmitic acid 0.72; 95% confidence intervals: 0.59–0.89). These associations persisted even in the group of approximately 85-year-old survivors. n-3 FAs were not associated with decreased risk of AD or dementia. In contrast to experimental studies, saturated FAs were inversely associated with risk of AD. No evidence of a protective effect of n-3 FAs against dementia was found. The results remained essentially unchanged if competing risk from mortality was taken into account.

Differences in lipid parameters among statins treated patients with coronary arteriosclerosis—a pilot study

Burchardt, P., *et al.*, *Eur. J. Lipid Sci. Tech.* 114:869–874, 2012.

Low-density lipoprotein (LDL), total cholesterol (TC), and high-density lipoprotein (HDL) are poor predictors of the cardiovascular risk among patients undergoing hypolipidemic therapy with statins. Thus, in this pilot study we have attempted to determine, on the basis of routinely used assessments of lipid profiles, sensitive and inexpensive parameter which would associate with the severity of coronary artery disease in patients undergoing hypolipidemic treatment who achieved LDL goal. Apolipoprotein (apo) B100, apoA1, LDL, triglycerides, HDL, lipoprotein (a) and TC levels were assessed in 140 patients referred for coronary angiography. The various ratios based on lipid parameters were calculated and compared to patients taking statins. Coronary arteriosclerosis was determined by the degree of single stenosis

CONTINUED ON NEXT PAGE

and quantitatively by applying the Gensini score. Using multivariate analysis we have found that in the group with hypolipidemic therapy and/or with treatment LDL target (70–100 mg/dL) the TC/apoB100 ratio was associated with coronary artery stenosis. Additionally, univariate analysis showed that the TC/apoB100 ratio (among treated subjects) was significantly lower in patients with hemodynamically significant stenosis of coronary arteries than in matched patients without coronary artery lesions.

Low-quality vegetable oils as feedstock for biodiesel production using K-pumice as solid catalyst. Tolerance of water and free fatty acids contents

Díaz, L., and M. E. Borges, *J. Agric. Food Chem.* 60:7928–7933, 2012.

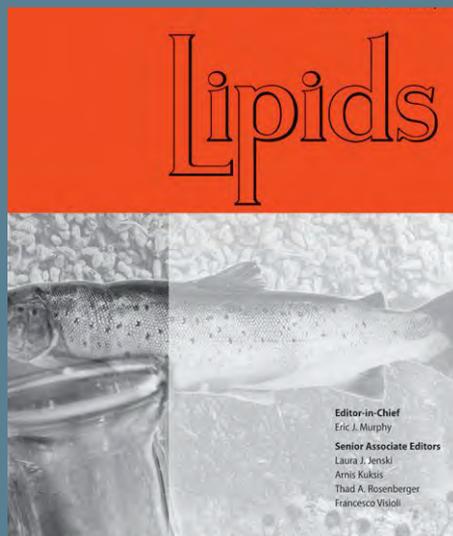
Waste oils are a promising alternative feedstock for biodiesel production due to the decrease of the industrial production costs. However, feedstock with high free fatty acids (FFA) content presents several drawbacks when alkaline-catalyzed transesterification reaction is employed in biodiesel production process. Nowadays, to develop suitable processes capable of treating oils with high free fatty acids content, a two-step process for biodiesel production is being investigated. The major problem that it presents is that two catalysts are needed to carry out the whole process: an acidic catalyst for free fatty acids esterification (first step) and a basic catalyst for pretreated product transesterification (second step). The use of a bifunctional catalyst, which allows both reactions to take place simultaneously, could minimize the production costs and time. In the present study, the behavior of pumice, a natural volcanic material used as a heterogeneous catalyst, was tested using oils with several FFA and water contents as feedstock in the transesterification reaction to produce biodiesel. Pumice as a bifunctional solid catalyst, which can catalyze simultaneously the esterification of FFA and the transesterification of fatty acid glycerides into biodiesel, was shown to be an efficient catalyst for the conversion of low-grade, nonedible oil feedstock into biodiesel product. Using this solid catalyst for the transesterification reaction, high FAME yields were achieved when feedstock oils presented a FFA content until approximately 2% wt/wt and a water content until 2% wt/wt.

AOCS Journals



Journal of the American Oil Chemists' Society (October)

- Identification of minor acylglycerols less polar than tricinolein in castor oil by mass spectrometry, Lin, J.-T., and G.Q. Chen
- Ratios of regioisomers of minor acylglycerols less polar than tricinolein in castor oil estimated by mass spectrometry, Lin, J.-T., and G.Q. Chen
- Surface-enhanced Raman spectroscopy of tribochemically formed boundary films of refined and unrefined canola oils, Chua, W., P. Chapman, and G.W. Stachowiak
- The toxic aldehyde, 4-hydroxy-2-trans-nonenal (HNE) formation in natural and imitation mozzarella cheeses: heat treatment effects, Han, I.H., and A.S. Csallany
- Detection of olive oil adulteration using FT-IR spectroscopy and PLS with variable importance of projection (VIP) scores, Oussama, A., F. Elabadi, S. Platikanov, F. Kzaiber, and R. Tauler
- *cis*-, *trans*- and saturated fatty acids in selected hydrogenated and refined vegetable oils in the Indian market, Amrutha Kala, A.L.
- Determining frying oil degradation by near infrared spectroscopy using chemometric techniques, Öğütçü, M., B. Aydeniz, M.B. Büyükcan, and E. Yılmaz
- Aldehydes from oxidized lipids can react with 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radicals in isoctane systems, Jeong, M.K., J. Yeo, E.Y. Jang, M.-J. Kim, and J. Lee
- Bioimprinted immobilization of *Candida antarctica* lipase A for concentration of omega-3 polyunsaturated fatty acids, Kahveci, D., and X. Xu
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- Rheology of cream-like emulsions prepared with soybean milk and low *trans* vegetable fat, Márquez, A.L., and J.R. Wagner
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Lipid characterization and antioxidant status of the seeds and meals of *Camelina sativa* and flax

Quezada, N. and G. Cherian, *Eur. J. Lipid Sci. Technol.* 114:974–982, 2012.

Four different antioxidant activity assays including 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid (ABTS), 2,2-diphenyl-1-picrylhydrazyl (DPPH), ferric reducing antioxidant power (FRAP) and oxygen radical absorption capacity (ORAC), and thiobarbituric acid reactive substances were performed on the methanolic and ethyl acetate extracts of *Camelina* seeds (CS), flaxseeds (FS), *Camelina* meal low fat (CMLF, 9.9% fat), *Camelina* meal high fat (CMHF, 24.6% fat), and flaxseed meal (FSM, 2.7% fat). In addition, the fatty acid profile, and phenolic, tocopherol, flavonoid, and glucosinolate contents of CS, FS, CMLF, CMHF, and FSM were studied. The major fatty acid was α -linolenic acid (C18:3 n-3) which was 33.2, 29.4, 30.2, 60.1, and 39.3% in CS, CMLF, CMHF, FS, and FSM, respectively. The methanolic extract of CMLF showed the highest values of ABTS, DPPH, and FRAP and the highest content of phenolic compounds, flavonoids, and glucosinolates. The methanolic and ethylacetate extracts of CMHF showed the highest values for ORAC and α - and γ -tocopherols. The ethylacetate extracts of seeds and meals of *Camelina sativa* and flax showed lower values for antioxidant activity, phenolic compounds, and flavonoids than the methanolic extracts. In general, *Camelina* and FS meals showed higher antioxidant activities, and phenolic and flavonoid contents than their respective seeds.

Dietary fat quality and risk of sudden cardiac death in women

Chiuvè, S.E., *et al.*, *Am. J. Clin. Nutr.* 96:498–507, 2012.

Dietary n-3 PUFAs are inversely associated with risk of sudden cardiac death (SCD); however, little is known about other fats and SCD. Furthermore, concerns have been raised that high n-6 PUFA intake may attenuate the benefits of n-3 PUFAs. We examined associations and selected interactions between dietary fatty acids, expressed as a proportion of total fat and SCD. We conducted a prospective cohort study among 91,981 women aged 34–59 y from the Nurses' Health Study in 1980. Over 30 y, we documented 385

SCDs. In multivariable models, women in the highest compared with the lowest quintile of SFA intake had an RR of SCD of 1.44 (95% CI: 1.04, 1.98). Conversely, women in the highest compared with the lowest quintile of PUFA intake had a relative risk (RR) of SCD of 0.57 (95% CI: 0.41, 0.78). Intakes of n-6 and n-3 PUFAs were both significantly associated with a lower risk of SCD, and n-6 PUFAs did not modify the association between n-3 PUFAs and SCD. MUFAs and trans fats were not associated with SCD risk. After further adjustment for coronary heart disease (CHD) and CHD risk factors potentially in the causal pathway, the association between PUFAs and SCD remained significant, whereas the association for SFAs was no longer significant. Intake of PUFAs as a proportion of fat was inversely associated with SCD risk, independent of traditional CHD risk factors. These results support dietary guidelines to improve dietary fat quality by replacing intake of SFAs with n-6 and n-3 PUFAs.

Protein and oil composition predictions of single soybeans by transmission raman spectroscopy

Schulmerich, M.V., *et al.*, *J. Agric. Food Chem.* 60:8097–8102, 2012.

The soybean industry requires rapid, accurate, and precise technologies for the analyses of seed/grain constituents. While the current gold standard for nondestructive quantification of economically and nutritionally important soybean components is near-infrared spectroscopy (NIRS), emerging technology may provide viable alternatives and lead to next-generation instrumentation for grain compositional analysis. In principle, Raman spectroscopy provides the necessary chemical information to generate models for predicting the concentration of soybean constituents. In this communication, we explore the use of transmission Raman spectroscopy (TRS) for nondestructive soybean measurements. We show that TRS uses the light scattering properties of soybeans to effectively homogenize the heterogeneous bulk of a soybean for representative sampling. Working with over 1000 individual intact soybean seeds, we developed a simple partial least-squares model for predicting oil and protein content non-destructively. We find TRS to have a root-mean-standard error of prediction (RMSEP) of 0.89% for oil measurements and 0.92% for protein measurements. In both calibration and validation sets, the predictive capabilities

of the model were similar to the error in the reference methods.

Uptake of natural and synthetic estrogens by maize seedlings

Card, M.L., *et al.*, *J. Agric. Food Chem.* 60:8264–8271, 2012.

Runoff from manure-fertilized crop fields constitutes a significant source of natural estrogens (e.g., estradiol [17β -E2] and estrone [E1]) and synthetic estrogen mimics (e.g., zeranol [α -ZAL] and zearalanone [ZAN]) in the environment. However, processes such as sorption to and uptake by plants may inhibit the environmental mobility of hormonally active compounds. Sorption to dried root tissue was assessed in batch sorption tests, and resulting sorption isotherms were nonlinear at aqueous concentrations below 0.1 μM and linear above that limit. To evaluate the role of crop plants in the environmental fate of such compounds, we exposed hydroponic solutions containing 2 μM 17β -E2, E1, α -ZAL, or ZAN to maize seedlings. After 22 days of exposure, α -ZAL and ZAN concentrations decreased by more than 96%, and 17β -E2 and E1 were undetectable. The decrease in α -ZAL and ZAN concentrations in maize-exposed solutions was initially slow, but the observed uptake exceeded that predicted by sorption alone within 3 d. All four estrogens were detected in root tissues at concentrations up to 0.19 $\mu\text{mol g}^{-1}$, with concentrations peaking after 1–3 days of exposure. Only 17β -E2 and α -ZAL were detected in shoots, and maximum concentrations were measured after 2 days for 17β -E2 (0.02 $\mu\text{mol g}^{-1}$) and 16 days for α -ZAL (0.8 nmol g^{-1}). Concentrations measured in root and shoot tissues were 82% or less than those predicted by a partition-limited uptake model, which is attributed to transformation and possibly irreversible binding processes.

Coconut oil enhances tomato carotenoid tissue accumulation compared to safflower oil in the Mongolian gerbil (*Meriones unguiculatus*)

Conlon, L.E., *et al.*, *J. Agric. Food Chem.* 60:8386–8394, 2012.

Evidence suggests that monounsaturated and polyunsaturated fats facilitate greater absorption of carotenoids than saturated fats. However, the comparison of consuming a polyunsaturated fat source versus a saturated fat source on tomato carotenoid

bioaccumulation has not been examined. The goal of this study was to determine the influence of coconut oil and safflower oil on tomato carotenoid tissue accumulation in Mongolian gerbils (*Meriones unguiculatus*) fed a 20% fat diet. Coconut oil feeding increased carotenoid concentrations among many compartments including total carotenoids in the serum ($p = 0.0003$), adrenal glandular phytoene ($p = 0.04$), hepatic phytofluene ($p = 0.0001$), testicular all-*trans*-lycopene ($p = 0.01$), and *cis*-lycopene ($p = 0.006$) in the prostate–seminal vesicle complex compared to safflower oil. Safflower oil-fed gerbils had greater splenic lycopene concentrations ($p = 0.006$) compared to coconut oil-fed gerbils. Coconut oil feeding increased serum cholesterol ($p = 0.0001$) and decreased hepatic cholesterol ($p = 0.0003$) compared to safflower oil. In summary, coconut oil enhanced tissue uptake of tomato carotenoids to a greater degree than safflower oil. These results may have been due to the large proportion of medium-chain fatty acids in coconut oil, which might have caused a shift in cholesterol flux to favor extrahepatic carotenoid tissue deposition.

Tracing the source of cooking oils with an integrated approach of using stable carbon isotope and fatty acid abundance

Liu, W., *et al.*, *J. Agric. Food Chem.* 60:8069–8073, 2012.

We report a new approach to identify swill-cooked oils that are recycled from tainted food and livestock waste from commercial vegetable and animal oils by means of carbon isotope values and relative abundance of fatty acids. We test this method using 40 cooking oil samples of different types with known sources. We found significant differences in both total organic carbon isotope as well as compound-specific isotope values and fatty acid C_{14}/C_{18} ratios between commercial vegetable oils refined from C_3 plants (from -35.7 to -27.0‰ and from 0 to 0.15) and animal oils (from -28.3 to -14.3‰ and from 0.1 to 0.6). Tested swill-cooked oils, which were generally refined by mixing with animal waste illegally, fall into a narrow $\delta^{13}\text{C}$ /fatty acid ratio distribution: from -25.9 to -24.1‰ and from 0.1 to 0.2. Our data demonstrate that the index of a cross-plotting between fatty acid $\delta^{13}\text{C}$ values and C_{14}/C_{18} ratios can be used to distinguish clean commercial cooking oils from illegal swill-cooked oils. ■



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Patents

Published Patents

Sheet-fed offset printing inks and varnishes comprising new solvents

Pulina, T., and C. Johnke, Sun Chemical Corp., US8168708, May 1, 2012

An offset printing varnish, comprising a solvent, is provided. The solvent comprises at least one triglyceride with saturated monocarboxylic acid moieties, which solvent is preferably food safe.

Method for producing at least one of α,β -unsaturated aldehyde and α,β -unsaturated carboxylic acid

Takeda, A., *et al.*, Mitsubishi Rayon Co., Ltd., US8173838, May 8, 2012

Disclosed is a method for producing at least one of an α,β -unsaturated aldehyde and an α,β -unsaturated carboxylic acid from an alcohol in a liquid phase through a simple process. Namely, at least one of an α,β -unsaturated aldehyde and an α,β -unsaturated carboxylic acid is produced by dehydrating and oxidizing an alcohol in a liquid phase at 110 to 250°C in the presence of molecular oxygen and a noble metal-containing catalyst. Alternatively, at least one of an α,β -unsaturated aldehyde and an α,β -unsaturated carboxylic acid is produced by dehydrating and oxidizing an alcohol in a liquid phase in the presence of molecular oxygen, a noble metal-containing catalyst, and an acidic substance.

Production of fatty acid and fatty acid ester

Pastinen, O., *et al.*, Aalto University Foundation, US8178706, May 15, 2012

The present invention concerns a process for forming a fatty acid, a fatty acid ester or a mixture thereof from a soap-comprising starting material, in which process a metal-ion forming agent is added to the starting material, whereby a mixture is formed, which contains an insoluble phase and a liquid phase, the insoluble phase is separated from the liquid phase, and an acid is added into the insoluble phase to form a fatty acid, or a monohydric alcohol and an acid catalyst are added to form a fatty acid ester, whereby two phases are formed, an aqueous phase and an organic phase, or, first, an acid is added and then a monohydric alcohol and an acid catalyst are added into at least a portion of the formed fatty acid to esterify the fatty acid.

Ruminant feedstock dietary supplement

Roman, E.A., *et al.*, Church & Dwight Co., Inc., US8182851, May 22, 2012

This invention provides a control release formulation or rumen-bypass dietary supplement in compacted form. The formulation or supplement has the capability to transport fatty acid calcium salt and between about 1–75% of one or more rumen-protected undegraded biologically active agents to the post-ruminal digestive system of a ruminant.

A feedstock containing the formulation or supplement for ruminants beneficially improves feed efficiency and body growth. The feedstock also is adapted to improve the lactational performance of dairy cattle.

Edible products with low content of saturated and trans unsaturated fats

Cleenewerck, B., *et al.*, Fuji Oil Co., Ltd., US8182857, May 22, 2012

The present invention relates to a structured, fat continuous edible product, wherein the edible product contains, expressed on total product basis, less than 35 wt% of saturated fatty acids, between 20 and 100 wt% of a triglyceride composition, between 0 and 80 wt% of a filler material, and less than 15 wt% of water. The triglyceride composition contains less than 50 wt% of saturated fatty acids, less than 10 wt% of trans unsaturated fatty acids, at least 10 wt% of POP triglycerides, wherein P is palmitic fatty acid, O is oleic acid, a ratio SUS/SUU of at least 1.3 [where S = saturated fatty acid; U = unsaturated fatty acid], a ratio SUS/S3 of at least 15, at least 90 wt% of C8–18 fatty acids, a ratio C16/C18 saturated fatty acids of at least 1. The triglyceride composition has an SFC [solid fat content] at 20°C of between 3 and 55%. The present invention also relates to a process for producing such a product and to triglyceride compositions suitable for use in such a product.

Phenyl acetic acid derivatives

Grillo, M., *et al.*, Amgen Inc., US8183293, May 22, 2012

Compounds, pharmaceutical compositions, and methods are provided that are useful in the treatment of inflammatory and immune-related diseases and conditions. In particular, the invention provides compounds which modulate the function and/or expression of proteins involved in atopic diseases, inflammatory conditions, and cancer. The subject compounds are carboxylic acid derivatives.

Base agent for electrical insulating oil

Kanoh, T., *et al.*, Lion Corp.; Japan AE Power Systems Corp., US8187508, May 29, 2012

Disclosed is a base agent for electrical insulating oils, which mainly contains an esterified product of glycerin and a linear or branched, saturated or unsaturated fatty acid having 6–14, preferably 8–12 carbon atoms. This base agent for electrical insulating oils is excellent in electrical characteristics, oxidation stability, cooling characteristics, flame retardance, and safety. In particular, this agent for electrical insulating oils can meet energy/environmental problems by using an edible oil and fat, which is obtained by using a fatty acid derived from a vegetable oil as a raw material, as the linear or branched, saturated or unsaturated fatty acid having 6–14 carbon atoms.

Recombinant microalgae cells producing novel oils

Franklin, S., *et al.*, Solazyme, Inc., US8187860, May 29, 2012

Disclosed herein are obligate heterotrophic microalgae cells containing an exogenous gene. In some embodiments the gene is a sucrose utilization gene, and further disclosed are methods of manufacturing triglyceride oils using sugar cane or sugar beets as a feedstock in a heterotrophic fermentation. In other embodiments the feedstock is depolymerized cellulosic material. Also disclosed are cells that produce

medium-chain fatty acids at levels not produced in nonrecombinant cells of the same species and genus.

Additive and vehicle for inks, paints, coatings and adhesives

Cook, L.J., and R.T. Skov, Omnitech Environmental, LLC, US8188184, May 29, 2012

An environmentally safe additive and vehicle system are provided for water-based and oil-based printing inks, paints, coatings, and adhesives, which can be rapidly transferred, dispersed, dispensed, spread, dried, and cured. The low cost, stable additive and vehicle system enhance multiple-color, high-speed printing with sharp, highly defined images and superior quality, and can be used on many different types of substrates, such as paper, paperboard, cardboard, clay-coated board, foil, plastic, glass, metal, wood, and composites. The additive may be formed by the reaction product of a photoinitiator, such as an ultraviolet light-activated polyelectrolyte, and a monomer, such as an acrylate or a methacrylate in an aqueous solution. In other embodiments, the additive is formed from a carboxylic acid or anhydride and alkylalkanolamine monomer or a dialkylaminoalkyl acrylate or methacrylate.

Process for producing triglycerides

Cain, F.W., and U. Schmid, Loders Croklaan B.V., US8183021, May 22, 2012

A process for producing triglycerides comprises: (i) subjecting a first triglyceride comprising at least 40% by moles of oleic acid residues, based on total acyl groups in the triglyceride, to a reaction with stearic acid, at least one ester of stearic acid or a mixture thereof, to obtain a composition comprising 1,3-distearoyl 2-oleoyl glyceride and trioleoyl glyceride; (ii) treating the composition to form a first fraction having an increased amount by weight of oleoyl groups compared to said composition and a second fraction having an increased amount by weight of stearoyl groups compared to said composition; (iii) hydrolyzing the first fraction to form oleic acid; and (iv) reacting said oleic acid or an ester thereof with a triglyceride comprising at least 50% by moles of palmitic acid residues, based on total acyl groups, to form a composition comprising 1,3-dioleoyl 2-palmitoyl glyceride.

Process for the preparation of cyclopropyl carboxylic acid esters and derivatives

Clark, A., et al., AstraZeneca AB, US8183412, May 22, 2012

The invention relates to a novel process for the preparation of certain cyclopropyl carboxylic acid esters and other cyclopropyl carboxylic acid derivatives; a novel process for the preparation of dimethylsulfoxonium methylide and dimethylsulfonium methylide; to the use of certain cyclopropyl carboxylic acid esters in a process for the preparation of intermediates that can be used in the synthesis of pharmaceutically active entities; and to certain intermediates provided by these processes.

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



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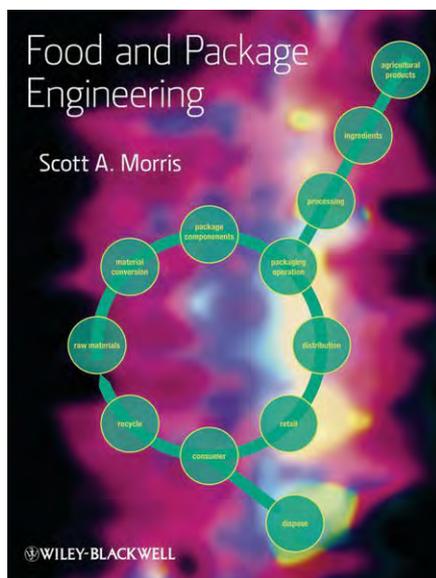
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Book Review

Food and Package Engineering
 Scott A. Morris, Wiley-Blackwell,
 John Wiley & Sons, Inc., 2011
 480 pages, \$199.99, ISBN 978-0-8138-1479-7

Symon Mahungu



Food and Package Engineering intends to fill a large gap in the information available on the subject. It is directed toward food scientists and those with modest food science training.

In that respect, the book scores very well. The information is presented in a format that encourages one to continue reading as the more technical concepts are introduced. The book makes the reader appreciate engineering concepts without being

overwhelmed with excessive mathematics. It is a great book for beginners in food science, particularly for a food and package engineering course.

The text is divided into 12 chapters. However, an analysis of the book indicates that it can be divided into four sections: introductory material (Chapters 1–2), processing (Chapters 3–7), management and distribution (Chapters 8–11), and future development and technologies (Chapter 12).

Chapter 1 presents a brief overview of food processing and packaging and how they are interrelated. It successfully explains how food and packaging impact positively on economics as well as discussing marketing issues in the food industry.

Basic engineering concepts critical to food processing and packaging are presented in Chapter 2. This chapter is essentially an overview of Chapters 3–7, and for nonstudents of food engineering, this may be enough reading material to impart working knowledge in this area of food science. The book manages to introduce and explain in very basic language the technical aspects and applications of mass and energy balances; nonthermal energy, such as mechanical and electrical; the mechanics of materials; fluid-flow systems; rheology; heat-transfer systems; and psychrometry. These concepts are critical and play a significant role in food processing and preservation.

The other 10 chapters are dedicated to specific aspects of the subject. These include various types of raw materials, their processing

into finished products, food preservation and shelf life, plant operations, and food regulations. Chapter 3 presents a glossary of raw materials used in packaging, while Chapter 4 describes processes for converting these raw materials into packaging materials. The chemical principles utilized during the raw material conversion are briefly presented for better understanding of basic principles. For example, cellulose and lignin chemistry is presented in order to understand paper production from wood.

The manufacture of steel and aluminum cans as well as glass bottles is covered in reasonable detail in Chapter 4. Production of various types of plastics and additives such as color, plasticizers, and antioxidants is presented in detail. Production processes such as extrusion, film metallization and microperforation, and lamination are presented as examples of packaging material production.

Chapter 5 is dedicated to secondary packaging components such as closures (caps, liners, etc.), glues and adhesives, tapes, aerosols, and types of inks for package printing and coding. Chapter 6 is dedicated to the engineering processes involved in food processing. The thermal issues and related calculations, sterilization, pasteurization, drying (drum- and spray-drying), freeze-drying, and concentration and separation of food products are well presented with relevant real-life examples.

Chapter 7 is dedicated to food preservation and shelf life. The author briefly discusses the chemical kinetics of food deterioration, followed by their application in shelf life testing. Interaction of environmental agents, water activity and mobility, packaging permeability, microbial product changes, preservative agent utilization, and package–product interactions as they affect shelf life of the packaged food is exhaustively explained.

Chapter 8 details issues regarding packaging machinery, filling equipment, general plant operation procedures, and factory safety, while Chapter 9 is an analysis of product damage control/prevention during transportation and distribution. Chapter 10 highlights food regulations and safety systems *vis-à-vis* regulatory agencies such as the US Food and Drug Administration and Environmental Protection Agency, as well as safety systems such as HACCP (Hazard Analysis Critical Control Point). Chapters 11 and 12 act as concluding chapters, with brief presentations on recycling and future technologies, respectively.

The book is a good course or textbook for undergraduate (seniors) pursuing food science with an interest in food processing. The information is well presented in an easy-to-comprehend format. However, I recommend an increase in the number of examples, especially calculations, in future editions so that every chapter has real-life examples from industry and information as to how industry deals with that particular problem. Alternatively, the author could prepare a students' manual incorporating case studies from industry with the appropriate solutions.

Symon Mahungu is a professor of food chemistry at Egerton University in Kenya. He holds a Ph.D. in Food Science from the University of Illinois, with M.S. and B.S. degrees in chemistry. He was appointed in 2008 by the United Nations Food and Agriculture Organization to the Joint FAO/WHO Expert Committee on Food Additives for a four-year term. He can be reached at smahungu@yahoo.com.

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Science in the media

The three articles that follow were adapted from an assignment for a graduate level lipid science course at the University of Alberta in Edmonton, Canada, in which students were asked to write about a lipid-related topic for the general public. Read the main article on page 655.

» **Beef is healthy, beef is safe**

» **Omega fats in diet and health**

» **Cholesterol, the misunderstood molecule**

“Super phos” esters: the key to higher-performance products

View the complete PowerPoint presentation given by the 2012 Samuel Rosen Memorial Award winner, Robert L. Reiersen, at the 103rd AOCS Annual Meeting & Expo in Long Beach, California, USA. Read the main article on page 643.

Beef is healthy, beef is **SAFE**

One day, about 12 years ago, my father came home from work and placed a ban on eating beef in our household. He worked at the seaport as a clearing officer, and his colleagues had talked about how beef would cause one to die from heart disease and stroke. In my father's own words, he was "not going to keep buying death with his money," so beef was struck off the menu and was replaced with chicken. My father recently suffered a partial stroke after several years of not eating beef. How ironic!

There are lots of myths and misunderstandings making rounds in the media about beef. Some of the newspaper headlines read, "Proven: Eating meat raises death risk from cancer and other disease," "Study shows how red meat increases the risk of death," etc. These distortions have scared consumers away from eating beef, causing them to miss out on the many vital nutrients beef has to offer.



Chinyere
Ekine-Dzivenu

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Beef has many health benefit

Beef is packed with nutrients that are essential for our general well-being. It is a great source of protein, B-vitamins, essential fatty acids, iron, and zinc. In beef, these minerals exist in a form that is much easier for the body to absorb compared to the same minerals in grains and pulses. Vitamin B-12 is found only in animal products and plays a key role in the normal functioning of cells in the muscle, brain and nervous system, and is important in the formation of blood. Niacin and riboflavin are other B-vitamins found in beef. Riboflavin helps the body use energy. It promotes healthy skin and good vision. Niacin promotes healthy skin and nerves, helps digestion and stimulates normal appetite.

Amino acids are the building blocks of proteins. The body needs 22 of these amino acids, 8 of which must come from the diet because the body cannot make them. These are called essential amino acids. Proteins containing these amino acids in the right proportions needed by the body are called complete or high quality proteins. Beef is a high quality protein and it helps the body to maintain, repair or build body tissues and also increases resistance to diseases and infection.

A very important nutrient in beef is iron. Red blood cells need iron to carry oxygen to and away from body cells. Iron is important for intellectual performance, work performance, immune defence and a healthy pregnancy. Iron deficiency is a common nutritional deficiency in young children and women of child bearing age. Beef is one of the best sources of iron. Beef also contains zinc. The body needs zinc to make enzymes and insulin, heal wounds and resist infections. When beef is not included in the diet, zinc and iron are particularly difficult to obtain. Following are some common misconceptions about beef.

Myth 1: Beef is unhealthy because it is high in saturated fat

It is widely believed that diets high in saturated fatty acid increase the risk of cardiovascular diseases but this is not the case with beef. 51% of the fat in beef is monounsaturated





of which about 90% is oleic acid. Saturated fat makes up about 45% and stearic acid, which has been found to have a neutral effect on cardiovascular risk, makes up a third of this proportion. Stearic acid is quickly converted to oleic acid, which is a beneficial monounsaturated fatty acid. Dietary stearic acid also reduces the absorption of cholesterol.

The remaining 4% of beef fat is made up of polyunsaturated fatty acids, which improve cholesterol levels. In sum over 70% of fatty acids in beef have a beneficial impact on cholesterol levels.

Myth 2: Beef causes cancer and other diseases

A common myth is that beef causes cancer and several other diseases. In fact, unprocessed beef is packed full of nutrients that protect your body. Beef also contains con-

jugated linoleic acid (CLA) which is a class of fat derived from ruminant animals that is thought to have beneficial effects on health. CLAs are believed to reduce the risk of cancer, atherosclerosis and diabetes. They are also considered to have anti-obesity effects. Beef also contains unsaturated fatty acids, omega 3 fatty acids, selenium, and the B-vitamins which protect against cardiovascular diseases.

Myth 3: Beef contains antibiotics, unsafe hormones and pesticides

Some fear that beef contains antibiotics, hormones, and residues that are harmful to the body. In fact, antibiotics are removed by the body of the animal over time just as in human beings. A withdrawal period is allowed between the time the antibiotics are administered and time when

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the animal is slaughtered, so that no residues are left in the animal.

All animals, including humans, produce hormones. Animals are given hormones for growth and to correct for the lack of hormones caused by the castration of bulls. Hormones are used to suppress estrus, improve weight gain and feed efficiency. These hormones are naturally occurring hormones that are also found in humans and other mammals. Their use is heavily regulated, and they are not allowed to be of a nature or quantity that would pose a risk to people.

Beef may get pesticides from feed like corn and hay but the animal's liver removes pesticides the same way it does in humans. Pesticides are unlikely to be present in the animals at the time the animal is eaten.

Myth 4: Beef contains heterocyclic amines and nitrites which are known to cause cancer

Heterocyclic amines (HCA) are formed when muscle meats such as fish, chicken, beef and pork are cooked at very high temperatures (frying, barbecuing). Cooking any kind of meat, not only beef, at high temperatures will cause amino acids and creatine (a compound found in muscles) to react and form HCA. When meat is cooked at lower temperatures (boiling, stewing), negligible amounts of HCA are formed, and associated risk is reduced. HCAs are not related to beef per se, but to the cooking method.

Similarly, nitrites are introduced during the processing of meats and are not present in unprocessed beef.

Part of a nutritionally balanced diet

Beef and red meat as a whole are not bad in and of themselves and so should be part of a nutritionally balanced diet. I do research on beef genomics and our focus is to develop a tool for selecting and breeding cattle with increased amounts of beneficial fatty acids. Over 50% of the fatty acids in beef are beneficial, and it may be possible to improve on this by selecting cattle with favourable genes which promote the accumulation of beneficial fatty acids.

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What should I stay away from?

Trans fat

Trans fat is abundant in processed food. It is produced by partially hydrogenating oil. These fats raise low density lipoprotein (LDL) (bad cholesterol) and lower high density lipoprotein (HDL) (good cholesterol). This is because it narrows arteries and restricts blood flow through the arteries. This condition increases the risk of a heart attack or a stroke. On the other hand, HDL helps clean up LDL from the blood stream.

Processed meats

Processed meats contain nitrites, which are considered carcinogenic. They also contain a lot of salt, fillers, artificial colours, and additives to prolong shelf life. These chemicals are not necessarily good for you.



Omega fats in diet and HEALTH

Every day, we are flooded with information about fats in our diet, and much of this information can be confusing to consumers. For example, although many media reports describe the health benefits of omega-3 fatty acids, other headlines draw those benefits into question. Such articles often focus on the results from one or two research studies and do not cover the full spectrum of information about omega fatty acids. In the process of simplifying the research outcomes, they sometimes become misleading. Our goal is to describe and debunk some of the persistent myths about omega-3 and omega-6 fatty acids in the human diet.

Omega-3 and omega-6 fatty acids are two families of fatty acids with different structural properties, which help determine their metabolic fate and biological functions in the body. The terms “omega-3” and “omega-6” (sometimes also known as n-3 or n-6) refer to the position of the final double bond in the fatty acid chain. Both families are derived from the essential fatty acids, linoleic acid (LA, omega-6) and alpha-linolenic acid (ALA, omega-3), which are abundant in plants but cannot be synthesized by animals. Both LA and ALA must be consumed in the diet, and are the building blocks for other omega-6 or omega-3 fatty acids, respectively. It is important to understand that the terms omega-3 and omega-6 refer to families of fatty acids, and that not all members of each family have the same biological functions. Media reports that emphasize the health benefits of “omega-3 fatty acids” can sometimes be confusing because they aren’t specific to a particular member of the omega-3 family.

Myth 1: All omega-3 fatty acids are the same

There is a key difference between omega-3 fatty acids obtained from plant sources and those obtained from animal sources such as fatty fish. Many plants, including flaxseed, walnut and spinach contain abundant amounts of omega-3 fatty acids, but mostly in the form of ALA, the dietary precursor to longer chain omega-3s such as eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). Many of the health benefits of “omega-3 fatty acids” are actually attributable to these longer chain omega-3s rather than to ALA itself. Although ALA can be converted in limited amounts by the body to EPA and DHA, this conversion is not very efficient (about 8% for EPA and 0.1% for DHA), and is greatly affected by other dietary and lifestyle factors. A large intake of ALA does not necessarily translate into a large increase in EPA or DHA levels. A direct dietary source of EPA and DHA is therefore recommended.

Besides fatty fish, which are a common dietary source of EPA and DHA, marine microalgae provide an effective DHA and EPA supplement that is also suitable for vegan diets. Microalgae occupy the bottom of the food chain, where fish originally

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Lisha Zhao, Sandeep Nain, and Yuning Gao

Lisha Zhao, Sandeep Nain, and Yuning Gao (from top to bottom) are graduate students in the Department of Agricultural, Food & Nutritional Science at the University of Alberta (Edmonton, Canada). For more information about the Lipid Science course or the assignment from which this article was adapted, contact Randall Weselake at randall.weselake@ualberta.ca.

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Omega-3's in the future

Genetically modified (GM) crops are currently being developed as a potential alternative land-based source of long-chain omega-3s. While the ultimate goal would be to produce EPA and DHA in a plant by introducing genes from microalgae, another strategy involves producing omega-3 intermediates that can be more efficiently converted into EPA and DHA in the body. Stearidonic acid (SDA) is an 18-carbon omega-3 fatty acid that can be converted to EPA more effectively in the body than ALA. The production of SDA in plants requires the insertion of only a single gene, and it is likely we will soon see SDA-enriched soybean oil on the market.

get their omega-3. Several omega-3 products (e.g. capsules) from microalgae are available on the market. It should be mentioned, however, that even these commercial products may not give a complete EPA and DHA supplement. Some microalgae species contain only EPA or DHA, not both. While each fatty acid carries its own health benefits, EPA and DHA are believed to be more beneficial in combination with one another. Therefore, it is important to choose a product (or combination of products) that provides adequate amounts of both EPA and DHA. In the future, plant-based sources of EPA/DHA may be developed through biotechnology. Some genetically modified crops are currently under development (see **Omega-3's in the future** on page S6.)

Myth 2: All omega-6 fatty acids are bad

Much of the media coverage emphasizes the importance of omega-3 fatty acids for optimal health, while suggest-

ing that too much omega-6 consumption is associated with certain health risks. For example, omega-3s are often described as anti-inflammatory, while omega-6s are pro-inflammatory. In fact, both of these activities are essential to our health and must exist in a balance. Omega-6 fatty acids are not bad for us, but rather play very important roles in maintaining proper physiological functions. Both LA (omega-6) and ALA (omega-3) are considered to be essential fatty acids. Some omega-6 fatty acids, such as gamma-linolenic acid (GLA) and di-homo gamma linolenic acid (DGLA) have even been reported to have health benefits of their own, when consumed in the proper balance with omega-3 fatty acids.

The problem is that most of us do not maintain a good balance of omega-6 and omega-3 fatty acids. Most of us already obtain enough LA in our diet from vegetable oil sources such as canola, corn, and soybean oil. On the other hand, omega-3 consumption tends to be lower, leading to an imbalance in our omega-6/omega-3 ratio. Recent surveys have shown that the ratio of omega-6 to omega-3 in most western diets ranges between 10 and 20 to 1, which is much higher than what our bodies are adapted to. This imbalance can actually have detrimental effects on the body's ability to use the omega-3 fatty acids that we do consume.

Both omega-6 and omega-3 fatty acids are metabolized in the body using the same biochemical pathway. In other words, they compete with each other for the enzymes to synthesize longer chain fatty acids, such as arachidonic acid (omega-6) or EPA and DHA (omega-3s). As a result, overconsumption of omega-6s can make the conversion rate of omega-3s even lower, which means we are getting much more severe imbalance ratio of long chain omega-6s to omega-3s. Accumulating evidence indicates that EPA and DHA are essential for retinal development of the eye, maintaining normal functions of brain and neural development, as well as decreasing the risk of cardiovascular diseases, cancer, inflammation, and a variety of other conditions. Unbalanced consumption of omega-6s and omega-3s has been reported to be associated with many negative effects to human health, such as heart disease, asthma, cancer, arthritis and depression.

Myth 3: There is one ideal omega-6/omega-3 ratio

Studies on human evolution have shown that our ancestors lived on an omega-6 to omega-3 ratio of around 1:1,



whereas our western diets are far in excess of that, up to around 20:1. This has been a huge and rapid change in our diet, but there has been relatively little change in our genetic make-up over the years. Therefore, we notice an increased incidence of lifestyle diseases such as obesity, cancer and heart disease. In response to this, many media reports have suggested various recommended ratios of omega-6 to omega-3 in our diets, which can be confusing to the public.

Clinical evidence has suggested a range of ratios of omega-6 to omega-3 as preventive measures in various disease conditions. A ratio of 4:1 was shown to reduce total mortality due to cardiovascular disease by 70%. A ratio of 2.5:1 has been associated with the reduction of rectal cell proliferation in patients with colorectal cancer. A ratio of 2-3:1 suppressed inflammation in patients with rheumatoid arthritis, and a ratio of 5:1 has a beneficial effect on patients with asthma. These studies indicate that the optimal ratio varies with the nature and severity of disease and from genetic predisposition. It is not possible to recommend a single "best" possible ratio on the basis of individual studies. The "ideal" ratio of omega-6 to omega-3 varies from individual to individual with changes in their food

habits, health conditions, differences in genetic predisposition, activity level, age and gender. It is clear, however, that a lower ratio of omega-6/omega-3 in range of around 3:1 is more desirable for reducing the risk of many of the chronic diseases.

Our responsibility as scientists

Recent research has emphasized the importance of maintaining a healthy balance of omega-3 and omega-6 fatty acids in our diet, which usually means decreasing our omega-6 consumption while increasing and diversifying our intake of omega-3 fatty acids. However, the sheer volume of information being disseminated to the public can be overwhelming, especially when studies appear to contradict one another. When we observe misleading information spreading through the media, it is important for experts to come forward and communicate the right information in an accessible way. The media plays an excellent role in spreading information to the general public; however, as scientists, we have a responsibility to help deliver the correct message and educate people to be aware of misleading information.

Cholesterol, the misunderstood MOLECULE

Cholesterol is a molecule that is often misunderstood. While cholesterol is often discussed in the media and many people are concerned about cholesterol and heart health, researchers still are learning more about cholesterol all the time, and there may be better ways to protect our hearts.

Cholesterol molecules are not tiny bits of evil that float around in your blood; cholesterol is important for your cells to function properly. Cholesterol is required for nerve function, is important for the production of several important hormones such as testosterone and estrogen, and regulates cell membrane fluidity. While cholesterol is an important molecule in the body, people are usually not concerned with having too little cholesterol. Instead, high blood cholesterol is considered to be one of the risk factors for heart disease and stroke.

Cholesterol, like other lipids, is a waxy molecule that is not soluble in water. Since cholesterol is not water soluble, it is transported in the bloodstream by way of larger, water soluble particles called lipoproteins. Two common lipoprotein particles that shuttle cholesterol to and from cells are low density lipoproteins (LDL) and high density lipoproteins (HDL). A high concentration of LDL relative to HDL is often considered to indicate a higher heart risk. As a result, LDL is often called “bad cholesterol” and HDL particles are often referred to as “good cholesterol”, although neither are themselves cholesterol.

What is often overlooked by the media is that the size of the LDL particles also has a profound effect on risk factors. Some people have LDL particles that are very large while others have LDL particles that are very small. The size of LDL particles may matter more than the numbers of LDL particles. Small LDL particles may be worse for your health, and people with large particles usually have lower particle numbers too. The difference can be illustrated by analogy: imagine you have a fine sieve, and two buckets. In one bucket you have coarse gravel, in the other bucket, you have sand. If you pour the gravel in, the sieve will keep the gravel out. But sand particles are a lot smaller, and there are more particles in the same volume, so they are able to get stuck in between the mesh of the sieve and clog it. Your arteries are similar to the sieve; both the size and number of LDL play a role in determining heart risk. Most tests for blood cholesterol report only three numbers: the levels of HDL, the levels of LDL, and the total blood cholesterol. These tests do not take particle size into account. Because of the importance of the size of the LDL particles, an NMR test, which shows the size breakdowns of the particles, may be best. While LDL size is partially determined by genetics, LDL levels are also influenced by exercise and are least affected by diet.

It is generally believed that cholesterol is involved in the build up of plaques within artery walls, blocking the flow of blood in the heart (atherosclerosis). These plaques can rupture and cause heart attacks and strokes. The exact mechanisms of plaque formation



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and cholesterol's role are still unknown, although there is evidence that atherosclerosis may partially be caused by LDL particles that are trapped in the artery walls. The cholesterol contained in the LDL particles can oxidize and cause inflammation and lead to plaque formation. This oxidation process can perhaps be prevented by having enough anti-oxidants, such as vitamin E, in your bloodstream, and the level of these anti oxidants can be affected by your diet. Also, rancid fats are able to oxidize other molecules in the body, so ensure that oils (especially encapsulated oils you like omega-3 supplements) are stored properly.

It was previously thought that you could reduce cholesterol through diet modification by avoiding high cholesterol foods such as eggs, dairy products, and fatty meats. It is now understood that while diet plays a role,

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a healthy lifestyle that incorporates appropriate exercise is more important.

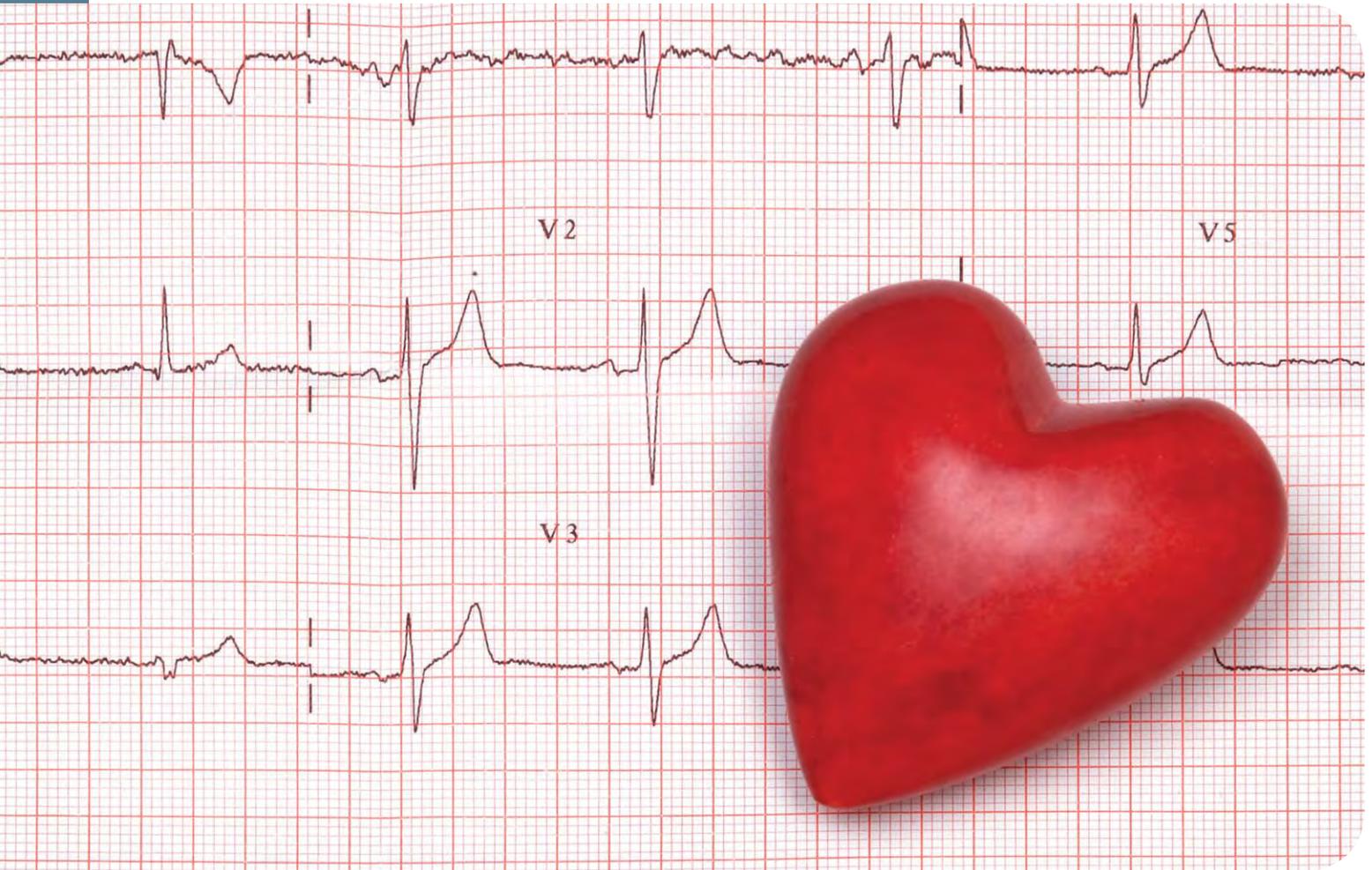
It is common for people with high concentrations of LDL to take medication to reduce LDL in the bloodstream. One group of these drugs, statins, are known to reduce heart attacks, and are the number one selling drugs in the world. Other compounds called phytosterols are cholesterol-like molecules produced by plants, and can also reduce LDL concentrations in the blood, but they do not reduce the risk of heart attacks. This is one of the reasons that there is controversy about whether LDL concentrations are a cause of heart disease. However, both statins and phytosterols have positive effects on health and are often appropriate health measures.

There are currently many ongoing scientific studies related to cholesterol and its effect on the body. However, many of the results from the studies are conflicting. This contributes to the difficulty recognizing and understanding the most important factors. Furthermore, the tendency in the media toward oversimplifying the reporting of mechanisms (for example “good and bad cholesterol”)

There are many factors that influence your risk of heart attack. It is never too early to start preventing heart disease. Here are some tips to protect your heart.

- Lead an active lifestyle
- Avoid rancid fats and do not take expired or improperly stored omega-3 supplements
- Do not smoke
- Get your cholesterol tested (NMR test) to determine the size and amount of your LDL particles
- If you have diabetes, manage your blood sugar closely
- Monitor your blood pressure with your doctor
- Eat a balanced diet (include plenty of fruits and vegetables)
- Avoid venereal diseases

results in incomplete presentation of the facts to the public.



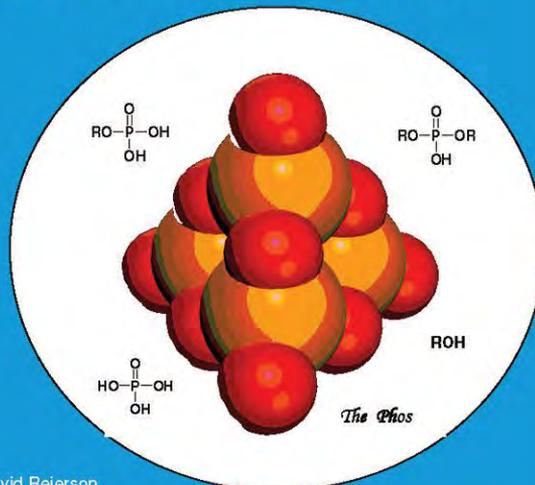
“Super phos” esters: the key to higher-performance PRODUCTS

Robert L. Reiersen, winner of the 2012 Samuel Rosen Memorial Award recognizing accomplishments in surfactant chemistry, made the following presentation at the 103rd AOCs Annual Meeting & Expo in Long Beach, California, USA. His article appears on page 643.



“Super Phos” Esters – A Key to Higher Performance Products

R. L. Reiersen
AOCs Annual Meeting
May 1, 2012



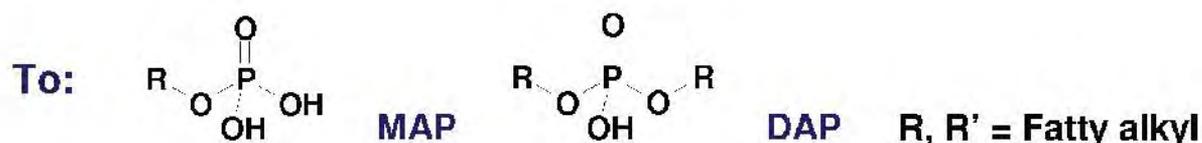
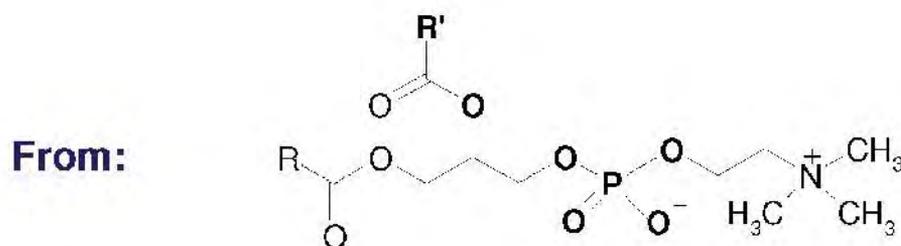
P₄O₁₀ Molecule Artwork by R. David Reiersen

Outline

- Nature's Surfactant, Industry's Opportunity
- Phosphation Processes
 - Polyphosphoric Acid
 - Phosphoric Anhydride
 - Hybrid
- Functional Performance Features
 - Adhesion Enhancement
 - Actives Deposition
 - Mildness
- Selected Applications
 - Industrial
 - Personal Care
- Summary

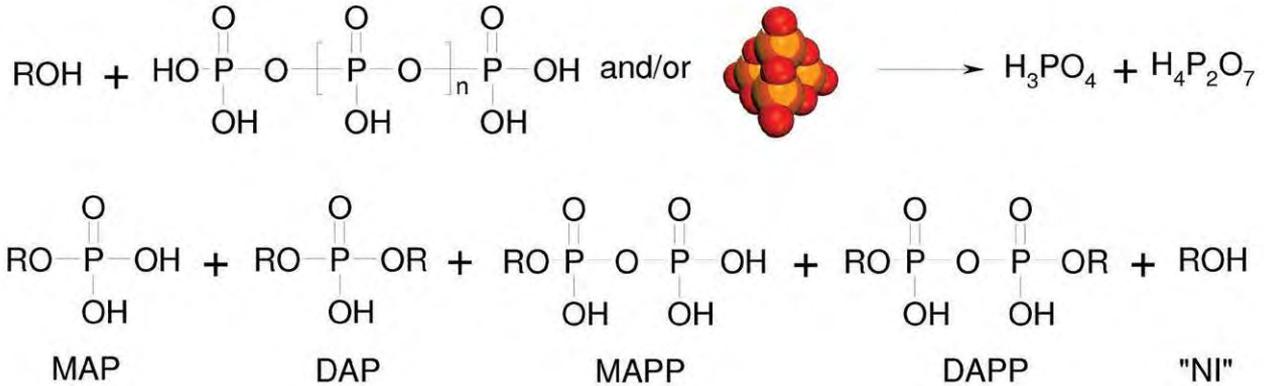
Nature's Surfactant – Industry's Opportunity

Designed by Nature – Complex Processes – Critical to Life
Industry: Lacks catalysts, needs broader utility, low cost → simplify!



If based on renewable, natural RM, industrial phosphate esters similarly should be regarded as sustainable and “green”.

Commercial Phosphate Processes, Products



- Poor selectivity; difficult separation
- Each reagent produces a characteristic mixture
- Product properties influenced by MAP : DAP ratio
- Composition affected by "impurities"

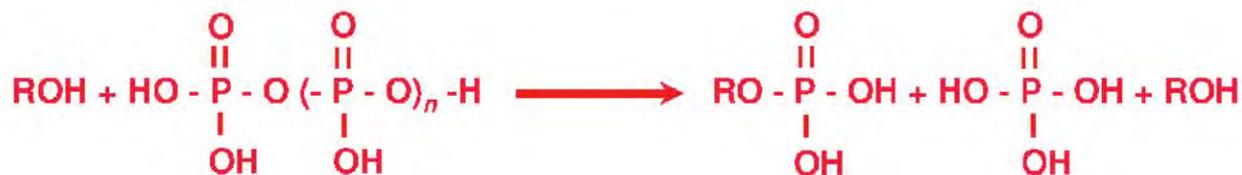
Properties of Alkyl Phosphates

Properties, Performance Influenced by MAP : DAP Ratio

$\begin{array}{c} \text{O} \\ \parallel \\ \text{ROP}(\text{OH})_2 \text{ [MAP]} \end{array}$	<u>Property</u>	$\begin{array}{c} \text{O} \\ \parallel \\ (\text{RO})_2\text{POH [DAP]} \end{array}$
High	Water Compatibility	Low
High	Detergency	Low
<i>High</i>	<i>Foam</i>	<i>Low</i>
High	Rinsability	Low
Low	Oil Compatibility	High
Low	Emulsification	High
Very Low	Skin Irritancy	Low
Unique	Skin Feel	



Theoretical Polyphosphoric Acid Reactions



Weight % of Each Component in 116% Polyphosphoric Acid*

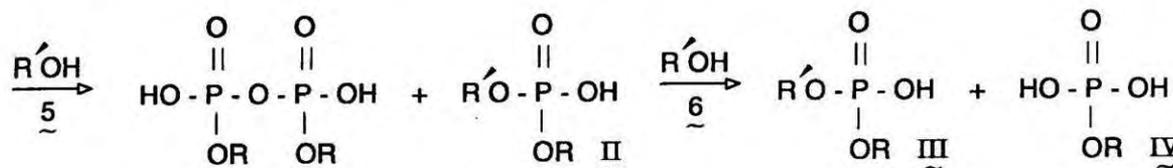
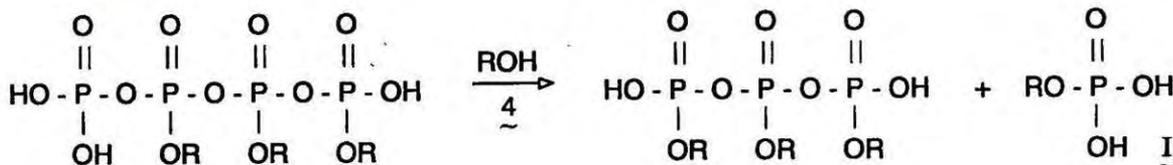
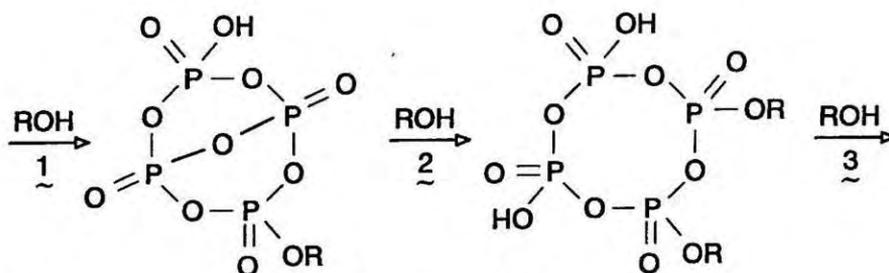
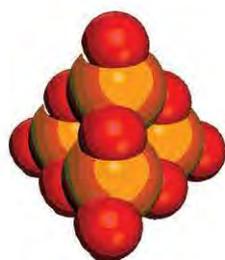
n	0	1	2	3	4	5	6	7	8	9	10	11	12	13	>13
%	3.9	11.8	12.7	12.0	10.5	9.0	8.0	6.6	5.6	4.5	3.7	3.0	2.5	1.7	4.5

Terminal Phosphate Groups (117% PPA) Yield 23 Mole % H_3PO_4 **

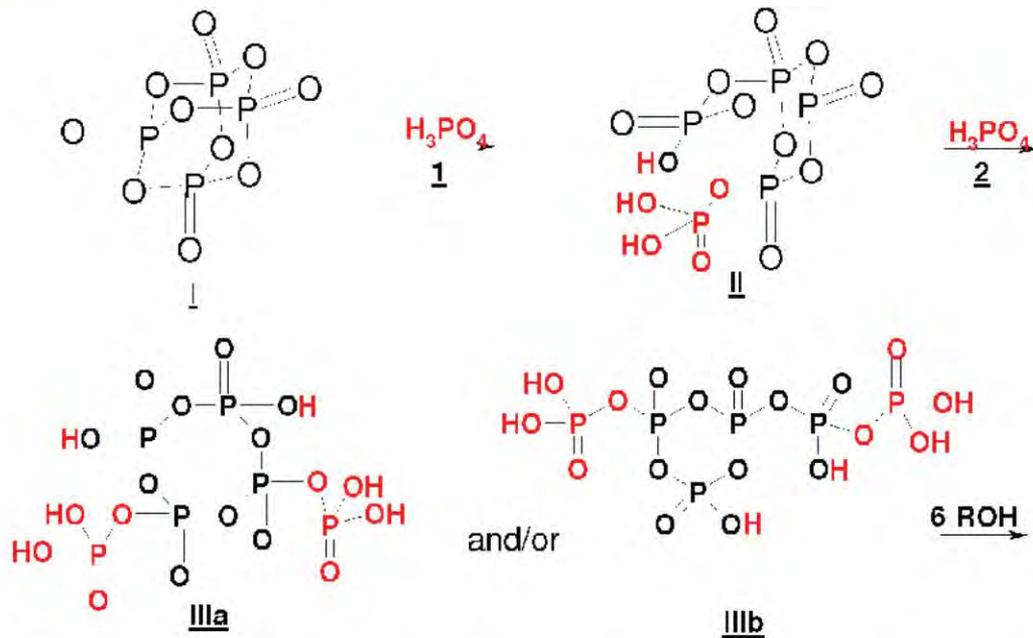
* R. Hudson, M. Dolan, *Ency. Chem. Tech.* 3rd Ed., Vol. 17, 450, (1982).

** F. Clarke, J. Lyons, *J. Am. Chem. Soc.* 88, 4401 (1966).

Theoretical P_4O_{10} Reactions

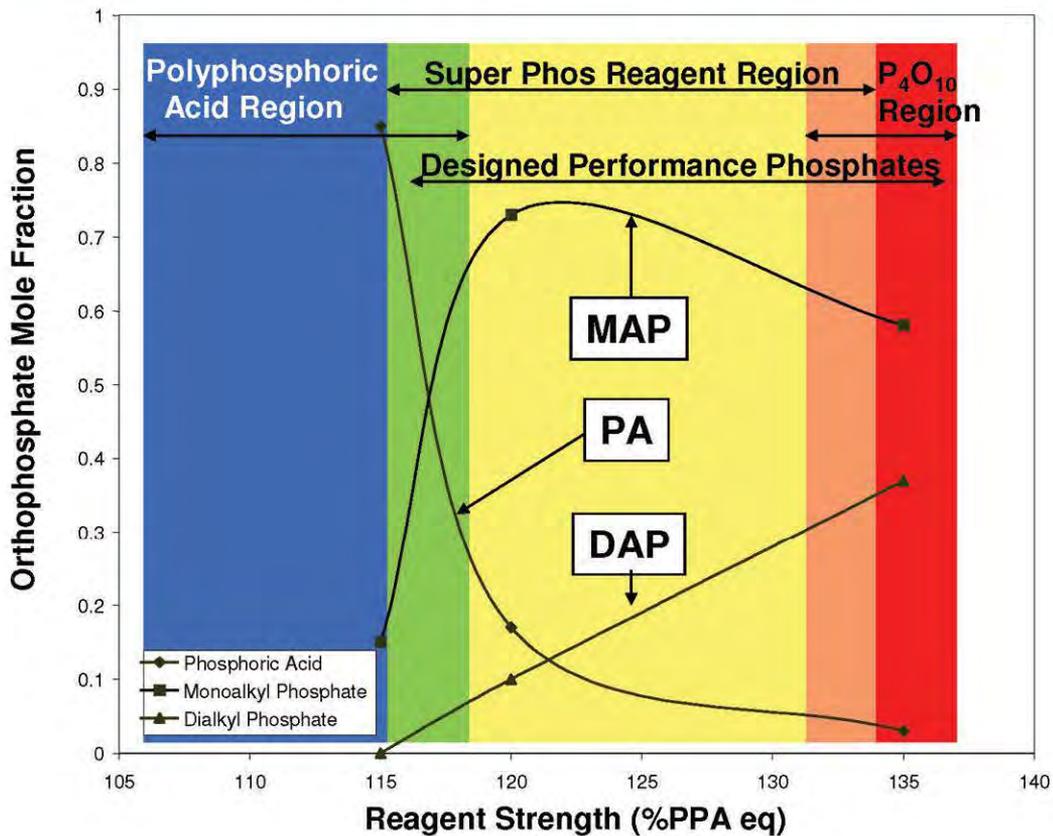


Super Phos Reagent



Orthophosphate acid and ester distribution determined by SP Reagent composition

Super Phos Process: Reagent Controls Composition



Designed Performance Phosphate Products

Need ↔ **Composition** ↔ **Process**

- Define compounds/compositions by Structure-Property Relationships
- Reliably produce optimum composition through Precision Phosphation
- Applications no longer limited to only PPA or P_4O_{10} compositions
- “Me-too” products are options, if required

Possible Structure Variation:

<u>Element</u>	<u>Range</u>
MAP/DAP Molar Ratio	60/40 to 93/7
Hydrophobe	$C_4 - C_{30}$, Aliphatic, olefinic, etc.
Degree of Ethoxylation	0 - 50; Especially 1 -3
Counter Ion (Salt)	Na, K, TEA, or customer choice
Other Components	Residual alcohol, H_3PO_4 , <u>≤ 6 wt.% ea.</u>

Definition: FUNCTIONAL Surfactant

A compound that offers good Surfactant properties **AND** additional, useful, Functional properties.

MAP Surfactant Properties (Function of Structure):

- Excellent detergency, low surface tension
- Mildness; essentially non-irritating to skin
- Good lubrication
- Excellent emulsifier

MAP Biological Properties (~ Hydrophobe):

- Biodegradable, bio-compatible
- Chemically stable but hydrolyzed by enzymes

MAP FUNCTIONAL Properties (Enhanced by Phosphate):

- ADHESION to organic and inorganic surfaces
- Film Formation

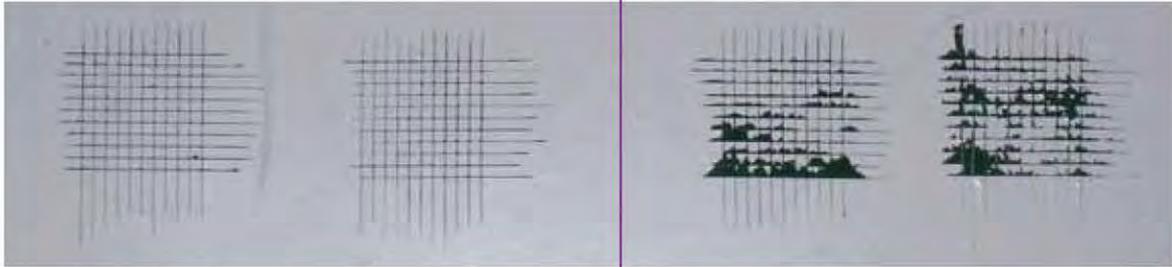
Phosphate Esters in Paint Formulations

Phosphate Ester

Blank

Dry Adhesion Wet Adhesion

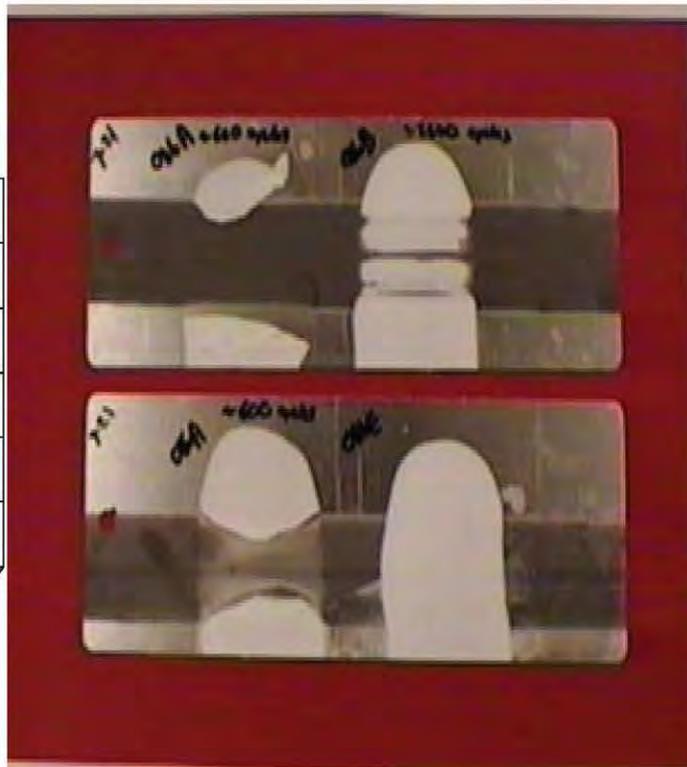
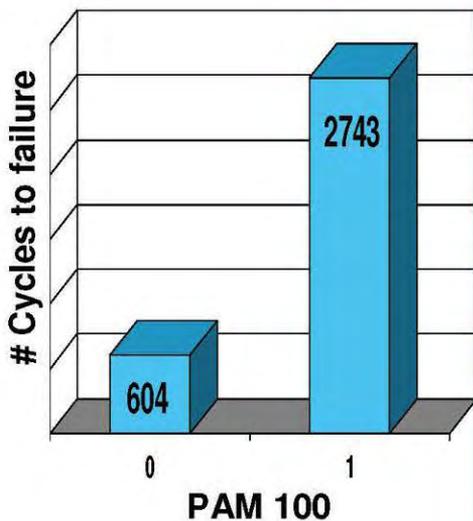
Dry Adhesion Wet Adhesion



Paint tested after drying 7 days

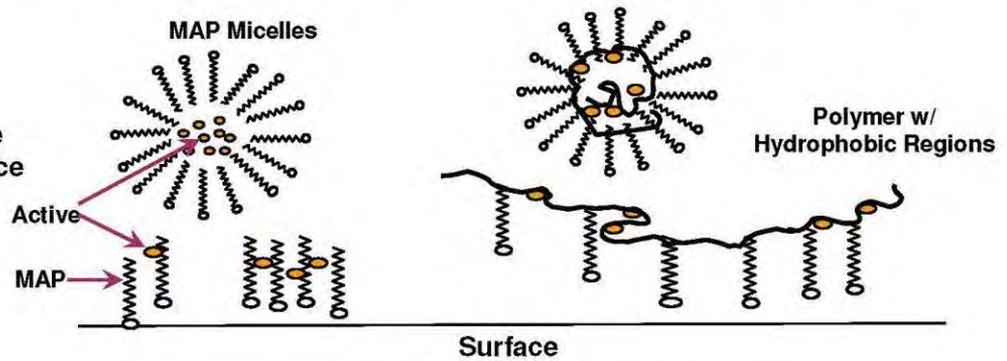
Phosphate ester enhanced adhesion to alkyd substrate

Improved Polymer Adhesion to Metal Substrates

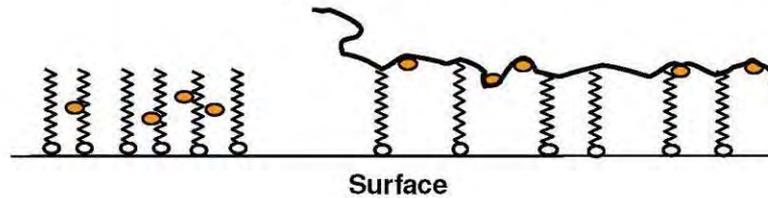


Actives Depositon; Controlled Release for PC

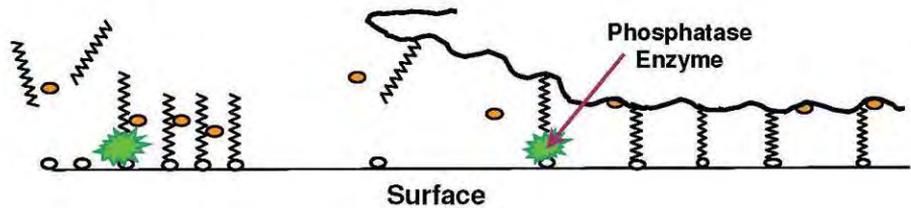
MAP Assisted Adsorption of Active Ingredient Onto Surface



Active Ingredient Remains Trapped In Adsorbed MAP Or MAP/Polymer Layer On Surface

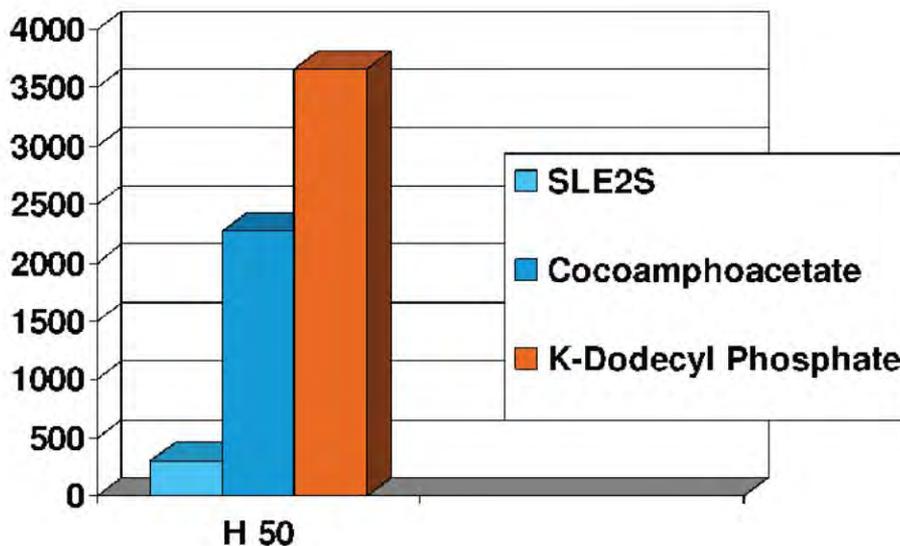


Enzyme Activity Cleaves Phosphate Anchor Over Time Releasing Active Ingredient



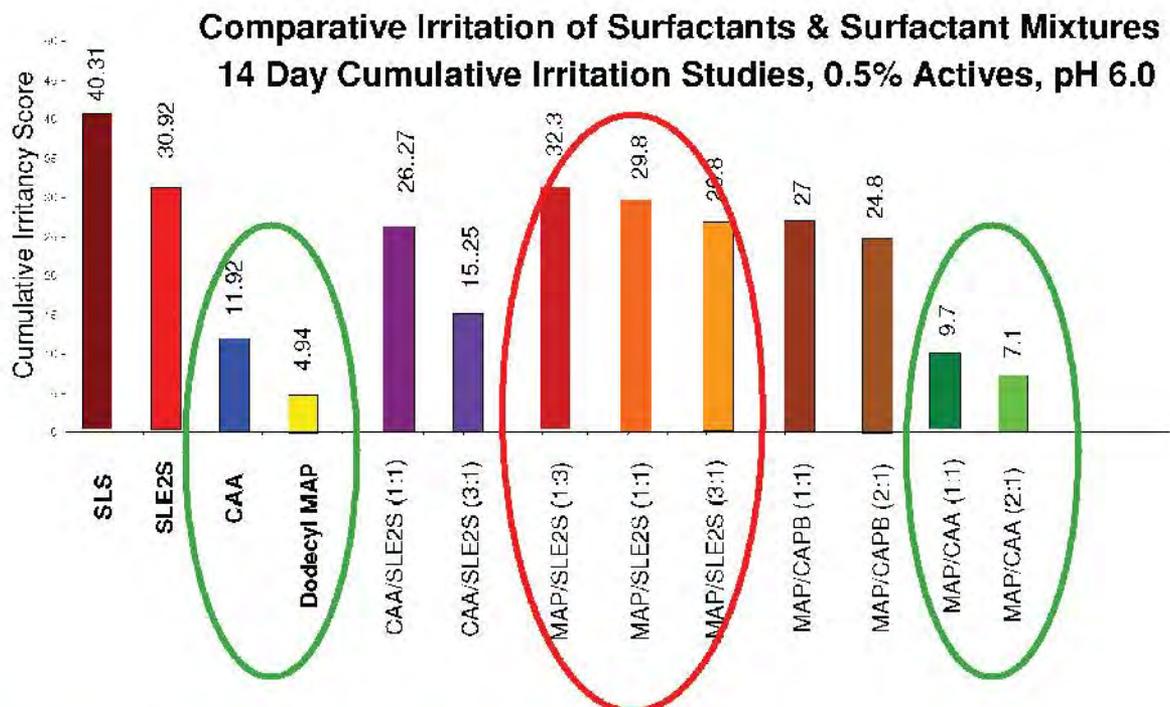
Red Blood Cell Test (Eye Irritation Indicator)

H50 is the concentration, in mg/L, of surfactant inducing 50 % hemolysis and is thus related to the degree of ocular safety



Outstanding Skin Compatibility

- MAPs are naturally mild and skin compatible
- Blends with true amphoteric surfactants give mild performance concentrates



Industrial Applications

- **In Latex Resins and Coatings (Surfactant or Monomer)**
 - Enhanced coating cohesion and substrate adhesion
 - Metal; also corrosion inhibition
 - Inorganic powders; as dispersant, stabilizer for pigments
 - Effective emulsifier; improves latex stability
 - Good wetting agent; increases gloss, color acceptance and durability
 - Improves water, blocking, abrasion, staining and scrub resistance
- Chemical mechanical planarization of silicon wafers
- Emulsifier for clinical diagnostic kits
- Reversible gellant (EOR)
- Lubricant additive
- Asphalt anti-stripping agent (aggregate)
- Herbicide adjuvant (formulation stability and bioefficacy)

Personal Care Applications

• Surfactant Applications

- Skin cleanser (infants to elderly)
- Baby Wet-wipes (lotion irritation potential ~ D.I. water)

• Protective Film Applications

- Harsh hair permanent chemicals
- Tooth erosion (acidic beverages), anti-stain
- Enamel renewal
- Dentinal hypersensitivity
- Residue free antiperspirant

• Actives Deposition, Retention

- Skin lotion fragrance retention aid
- Sunscreen lotion
- Anti-bacterial agents
- Hair dye (color enhancement and retention)

Summary

- “Super Phos” esters offer tremendous structural diversity; deliberate MAP:DAP control for improved; product performance.
- “Super Phos” process bridges the PPA – P_4O_{10} composition gap.
 - Predictable correlation between Reagent strength and product composition
 - Optimum composition reliably produced via Precision Phosphation
- Green MAPs provide unique combinations - anionic surfactant power with exceptional mildness and good skin feel.
- Phosphate group provides ADHESION for film formation; improves coating toughness, durability.

As long as problems exist, new opportunities wait to be discovered!

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