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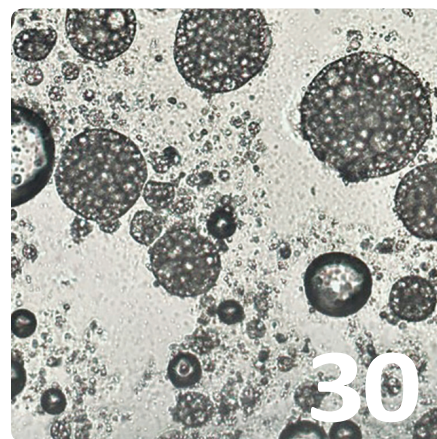
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Science beyond borders: international student exchange

Laura Cassiday

As an undergraduate student at the University of Arkansas, in Fayetteville, USA, Sarah Mayfield received an opportunity that most food scientists can only dream of: the chance to study chocolate in Belgium, a country famous for that confection. As part of her undergraduate honors thesis, Mayfield prepared and analyzed chocolate products containing conjugated linoleic acid (CLA)-rich soybean oil at the University of Ghent's renowned Cacaolab. "The Cacaolab has all the equipment they would have in a company that makes chocolate, but at a lab scale instead of an industrial scale," says Mayfield. "I was able to do research that I wouldn't have been able to do in Arkansas."

- International student exchange programs offer undergraduate and graduate students the ability to study or conduct research at a university in a different country.
- Exchange programs offer many benefits to students, such as improved responsibility, flexibility, and leadership skills; laboratory facilities, equipment, or expertise not available at home; and scientific collaborations.
- The most successful research exchanges are built upon relationships between professors at home and host universities.

In addition to research opportunities, international student exchange programs offer the ability to immerse oneself in another culture, learn a foreign language, take classes from experts in a particular field, and forge collaborations. With increasing globalization and communication capabilities, arranging an international student exchange has never been easier.

INTERNATIONAL AFFAIRS

AOCS member Roland Verhé, professor emeritus at the University of Ghent, has been instrumental in facilitating the exchange of thousands of students across the globe. In 1988, Verhé became a scientific coordinator for the newly formed Erasmus Programme, a European student exchange program established by the European Commission. The program was named for the Dutch philosopher Desiderius Erasmus of Rotterdam (1465–1536), an early proponent of international education. The Erasmus program provides scholarships and support for EU students to study at one of more than 4,000 partner institutions in 37 countries. A key aspect of the program is that students do not pay tuition fees to the host university. Grants are available to help cover travel and living expenses in the foreign country.

In 2004, Verhé and fellow AOCS member Andrew Proctor, professor of food science at the University of Arkansas, applied for a grant from the Atlantis Program, a student and faculty exchange program funded jointly by the EU and the US. They received a grant to fund student research exchanges in the area of biorenewable resources. Students from the EU could receive financial assistance to study at one of three US universities (University of Arkansas, Iowa State University, and Kansas State University), while US applicants could choose among three European universities (University of Ghent; University of Graz, in Austria; and National Polytechnic Institute of Toulouse, in France). Although the Erasmus program continues, US funding for the Atlantis program ended in 2013. Verhé has also helped develop student and staff exchange programs



FIG. 1. Between classes and lab work, students find time to visit cultural sites in their host countries, such as Vieux Lyon, a renaissance district of Lyon, France. Credit: Julian Silverman

in other regions of the world, including China, Mongolia, the Middle East, and Africa.

According to Verhé, a successful international exchange program requires a strong network of partner institutions with clearly defined expectations. "I don't want students to waste their time," he says. "If we send out a student to a partner in the network, we know exactly how many credits he is earning and how to translate grades into the home university of the student." Before the student leaves to study abroad, a contract should be made and signed by the student, the home university, and the host university. "Then there is never a discussion when something goes wrong," says Verhé. "We know exactly what a student has to do, and if he's not doing his examinations and things like this, he is not able to obtain grades."

TYPES OF EXCHANGE

For science students who wish to study abroad, there are three types of exchange programs, says Verhé. The first involves only coursework. The student attends lectures at the host university and obtains credits and grades that transfer to his or her home university. In the second type of exchange, the student completes a research internship in a lab at the host university. The research may contribute to the student's undergraduate, master's, or doctoral thesis at their home university. In the third type of program, students complete both coursework and research at the host university. "I am in favor of this last option because if a student is only coming for a research internship, they stay in the same laboratory doing experiments, and the majority of the student population is attend-

ing lectures," says Verhé. "The social experience is better if a student is able to attend lectures and be part of a group. Otherwise, students, especially shy students, sometimes isolate themselves."

Because Mayfield was concerned about falling behind in her coursework at the University of Arkansas, she chose a 3-month research internship at the University of Ghent that took place during her summer break. "It wasn't a formal exchange program in the sense that you go there and do classes, and you're with a bunch of other students, but there was an agreement between our two universities to send students back and forth," says Mayfield. She did not take any classes at the University of Ghent, but instead focused on her research analyzing the physical and chemical properties of shortening and chocolate made from CLA-rich soybean oil. The work not only comprised her undergraduate honors thesis, but also resulted in two peer-reviewed publications.

Julian Silverman, now a postdoctoral research and teaching fellow in chemical and petroleum engineering at the University of Kansas, in Lawrence, USA, participated in a student exchange program as an undergraduate at McGill University, in Quebec, Canada. His 1-month summer program at École Supérieure de Chimie Physique Électronique de Lyon (CPE Lyon), in France, included both coursework and a lab component, as well as opportunities for sightseeing (Fig. 1). "Every morning from 9 to 10 a.m., there was a French class," says Silverman "Every day there were also lab tours, a literature project, or a few experiments. I didn't get my hands too dirty, but it was a good bridge between my sophomore year in a university chemistry program and research."



FIG. 2. A visit to an edible oil mill during the Technical University of Graz's short course on food science. Credit: Andrew Proctor

Although exchange opportunities exist for graduate students, they are not as common as those for undergraduates. "The nature of the business is that a graduate student is usually on a professor's grant, and the professor wants results," says Proctor. "So what we're trying to do at the University of Arkansas is to embed the international experience into the work itself. For example, if we have two professors working together on a project, then they can exchange students and continue their project at the other institution." Proctor notes that one of the University of Arkansas' exchange partners, the Technical University of Graz, in Austria, has one of the best mass spectrometry facilities in the world, and student and staff exchanges have fostered research collaborations between the two universities.

As a master's degree student at Istanbul Technical University in Turkey, Derya Kahveci participated in an Erasmus exchange program at the Technical University of Denmark, in Copenhagen. "I was familiar with Professor Xuebing Xu's work, and I wanted to have the opportunity to work in his lab," says Kahveci. "I did not know much about Denmark when I applied for an Erasmus grant. I chose the lab rather than the country, and it turned out to be a wonderful choice in both aspects."

In Xu's lab, Kahveci studied the lipase-catalyzed synthesis of diacylglycerols in ionic liquids as solvents. "I can honestly say that this exchange of eight months actually changed my life afterwards," says Kahveci. "After two months of my stay, Professor Xu offered me a PhD position in his lab, which he later moved to Aarhus University [also in Denmark]." So after defending her master's thesis in Istanbul, Kahveci moved back to Denmark for 3 years to complete her PhD in Xu's lab. Then she returned home to Istanbul, where she is now an assistant professor at Yeditepe University.

For undergraduate or graduate students who hesitate to commit to months-long exchange programs, short courses are

an opportunity to obtain a quick snapshot of the science and culture of another country. For example, in July 2017, a group of undergraduate and graduate students from the University of Arkansas attended a 2-week short course on food science and culinary arts at the Technical University of Graz. The course included lectures, laboratory and culinary exercises, and group activities. The students also went on several field trips, including visits to a major chocolate company, a winery, and an oilseed press (Fig. 2). In 2018, a similar short course for Austrian and other EU students will take place at the University of Arkansas.

Mayfield, now a PhD student in the food science department at the University of Arkansas, participated in the recent short course at the Technical University of Graz. "I particularly enjoyed learning about the relationship between analytical and sensory analysis of food flavors. I also went to Graz a week early and stayed a week later to do some work in their analytical flavor chemistry lab," says Mayfield. "As a graduate student, it's not very feasible to go abroad for a whole semester solely for the cultural experience, but if it's for research and fits into your overall thesis, then it can be very beneficial."

NEW HORIZONS

Whereas some undergraduate students choose an international destination based on the strengths of a specific university or laboratory, others make their selection based on a particular region, language, or culture. As an undergraduate at Neubrandenburg University of Applied Science, in Germany, Fabien Schultz studied for a semester in Uganda. "I wanted to improve my English and experience a culture outside of Europe, so I had three options: Australia; the US; and some English-speaking countries in Africa, such as Uganda," says Schultz. "The study fees were quite high in Australia and the US, and visiting Africa has always been a dream of mine, so

that's why I went to Uganda." Under his own initiative, Schultz contacted Kampala University in Uganda, and inquired about the possibility of an exchange. Officials at Kampala University approved, so the two universities put together a collaboration contract, and Schultz's study fees were waived.

Schultz's girlfriend, Inken Dworak, a journalism student, arranged a similar exchange between her university and Kampala University. "We just booked a flight and went there, and we didn't even know what was going to happen," says Schultz. "We didn't even have accommodations." An Ebola outbreak coincided with their arrival in Uganda, which made their first few weeks in the country challenging. However, they remained in Uganda for 7 months, taking courses and teaching. "I'm a food biotechnologist focusing on lipid chemistry, and when I went to Uganda I took some chemistry courses, as well as some other courses that were interesting to me, like Swahili and the study of insects," says Schultz.

Because the courses in Uganda were structured differently than those in Germany, Schultz's credits did not transfer to his home university. Also, laboratory research was difficult to perform due to a lack of equipment and facilities, but Schultz was able to conduct research in the field. He has no regrets about his time spent abroad. "From then on, every year I returned to Uganda for at least a month," he says. "Now my PhD thesis at the Technical University of Berlin focuses on medicinal plants and lipids from the African rainforest."

Schultz was so affected by his experience in Uganda that, together with Dworak and a Ugandan friend named Kyewalyanga Moses, he founded a charity organization called ARUDEVO (African Rural Development Volunteers; www.arudevo.com) to facilitate the transfer of knowledge in the region. Through ARUDEVO, Schultz and other visiting students and professors give workshops on topics such as food safety, soap production with local raw materials, and microbiology (Fig. 3). The organization also sponsors projects to help women, vulnerable children, and people with disabilities, and to provide mobile telephones to people in impoverished rural communities of Uganda.

CULTURE SHOCK

Some students are reluctant to participate in exchange programs because of a perceived language barrier, or they worry that the culture of the country will be too different, and they will not be able to fit in. However, such fears are often unfounded, particularly for students who can speak English. "All the young people in Belgium, and everybody working in a university setting, speaks English," says Mayfield. Therefore, she had no difficulties in communicating with her research mentor or the people in her lab at the University of Ghent. "The only tricky thing was just being in the town walking around, and all the signs were in Dutch. Also, not many of the restaurants there have English menus," says Mayfield. "But it was definitely manageable."

"In Europe more and more, the working language is English," says Verhé. However, students who wish to learn the local language often have opportunities to take classes at their home or host universities, and ample chances to practice with



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native speakers. Proctor notes that language is almost never an issue for European students who come to the United States. “They’re all fluent in English,” he says.

Although cultures can vary significantly, international exchanges can help improve a student’s flexibility and ability to adapt to new environments, says Verhé. Schultz says that it took some time for him to adjust to the more relaxed attitude of many people in Uganda. “If I wanted to do something on a particular day, and I put out a schedule, I would have a hard time achieving it because people are often very late for appointments.” He recalls a university lecture that was scheduled to begin at 8 a.m. The students all waited until 2 p.m., when the lecturer finally arrived. “Although this big of a delay was uncommon, in the Ugandan culture it was a normal thing to do,” says Schultz. “Therefore, we had to always schedule alternative tasks and learn to be more flexible.”

As blonde-haired, blue-eyed Germans, Schultz and his girlfriend felt rather conspicuous in Uganda. “We were the first Europeans at that university, and also the only ones who were light-skinned, so we were quite a highlight at the university,” he says. At first, this made them feel insecure. “But in the end, everybody we met, especially the less-privileged people in the countryside, were all so welcoming and warm-hearted,” says Schultz. “When we came to their community, they would slaughter their only chicken just for us.”

Schultz did not realize the full cultural impact of his exchange until he and his girlfriend returned to Germany. “We noticed that German people complain quite a lot about minor things like the weather,” he says. “We realized that most people in Uganda lack material possessions, basic infrastructure, and some human rights, but they seem to be happier than we are in Germany. That’s when we said, okay, we have to give them something back, and that inspired us to found ARUDEVO.”

Lab cultures can also vary significantly among countries. Mayfield notes that the lab she worked in at the University of Ghent was much more formal and hierarchical than her lab at the University of Arkansas. “In our department at Arkansas, undergraduates can have conversations with graduate students and professors, and generally it’s not an intimidating thing,” she says. “But in Belgium, you don’t just walk into a professor’s office whenever you want to talk to them and say, ‘Oh, are you busy? I have to ask you a question.’ You have to make an appointment.” She also notes that PhD students, master’s students, and undergraduates were all on different hierarchical levels and rarely interacted. “It was kind of like, oh, you’re above me, so I feel weird talking to you,” she says. “And it wasn’t just that university. I talked to other European students, and they said it’s a pretty common thing throughout Europe.”

PERSONAL GROWTH

In addition to lessons learned in the classroom and experience gained in the lab, international exchange programs offer many opportunities for personal growth. “I’ve talked to many parents of exchange students, and they say, ‘My daughter or son was abroad for 6 months, and they returned a different person,’” says Verhé. “They have a much stronger personality, are



FIG. 3. Fabien Schultz guides a soap-making workshop in rural Uganda. Credit: Inken Dworak

more responsible, and are much more developed in their social behavior.”

“Until moving to Copenhagen, I lived with my family, so being alone in a foreign country away from everyone was tough at times,” says Kahveci. “However, looking back, I’m glad I had to do it—it forced me to grow up a bit.”

Mayfield says that her exchange experience helped her to become more independent in the lab. “When I started making the chocolate products, I had to figure out where to get the ingredients. I just assumed I would go over to the Cacaolab, and someone would have all the ingredients sitting out for me, but they didn’t.” Mayfield had to find and contact the appropriate people at the university for ingredients such as cocoa butter, locate their offices, and pick up the ingredients. “At the time that was very intimidating to me, but after doing it for a few weeks, I became a lot more comfortable with it,” she says. “I learned a lot of communications skills and teamwork, so it was very beneficial.”

Silverman also had to step outside his comfort zone as an exchange student in France. “I had to give this horrible presentation in French—my French has never been great—but the professors in the room were so kind,” he says. “It was a foreign place where you could try things and maybe fail a little bit, but you also had a supportive group of people who said, ‘Keep going,’ which is all you really need at that stage.”

Schultz says that his exchange trip to Uganda improved his responsibility and leadership skills. “When you go to rural Uganda, people ask you to give a speech or say a prayer, and speaking in front of that many people changed me quite a lot,” he says. “It definitely made giving a talk at the annual AOCS meeting seem rather effortless.” Schultz is also applying his leadership skills as co-leader of the AOCS Student Common Interest Group.

In addition, an international exchange looks good on a resumé, and may help students advance their career after graduation, says Verhé. “Students who have done 6 months abroad have a big advantage because they know about social and cultural differences of other countries,” he says. “For people in positions with an international character, they are not afraid if the boss says, ‘You have to go to China.’ They are used to doing this.”

MAKING AN EXCHANGE

For students who wish to participate in an international exchange, a first step is to contact their university's international programs office. Administrators can help students choose a host university, select appropriate courses or a research internship, and secure funding. How much of his or her own money a student will have to pay varies. Typically, an international student does not pay tuition fees above what they are already paying at their home university. Scholarships and grants can be obtained to help cover travel and living expenses. Students with research internships often obtain a stipend from either the home or host university.

Forming connections with professors overseas is important for students seeking research internships. "The AOCS Annual Meeting is a perfect place to make that happen because we have so many professors from all over the world there," says Proctor. However, he notes that often professors and their students at the Annual Meeting do not interact with their cohorts in other countries as much as they could. "International professors often keep their group of students with them," he says. "The students could interact more with the student division, and the education division would benefit from more faculty involvement. Students and professors tend to stay in their cultural comfort zone." Instead, Proctor recommends that professors and students interact more with their international colleagues at the meeting to develop collaborations that could result in student exchange programs. "The relationship comes before the program, and building mutual trust is essential," says Proctor.

Verhé agrees that most research internships arise from a personal relationship between two professors. "I have never sent students to a university or to a professor if I don't know the quality of that university or the strength of the laboratory," he says. "It is very important that the professors know and trust each other." He adds that an international exchange student must have both administrative support from the university's international programs office, and scientific support from her advisor or professors.

Kahveci is currently the departmental coordinator of exchange programs at Yeditepe University. "My students hesitate to apply sometimes, worrying that they will have to spend more semesters to graduate due to the time spent abroad," she says. "I always encourage them to look at it as a 'once in a lifetime experience,' and not only an academic achievement."

Schultz also encourages students to take the step of participating in an international exchange, during a time of life when they have the career and familial flexibility to live for several months in a foreign land. "Of course, there's some risk, but in the end it pays off, and this type of travel to a totally different culture becomes more complicated when you have a family or children," he says. "It broadens your horizon, and it's so much fun making new friends from other continents."

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

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Honorable Mention

Felicia Brewster
Jerry Buttell
Wade Chase
Lori Johnson
Travis Patterson
Ag Processing, Inc.
Hastings, NE 68901 USA

FEED MICROSCOPY

First Place

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Second Place

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Third Place

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FISH MEAL

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Samutprakaeng 10130 Thailand

Honorable Mention

Pedro Aguirre Larrain
Nsf Inassa SAC
Lima 32 Peru

GAS CHROMATOGRAPHY

First Place

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Paul Thionville
Kristopher Williams
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Honorable Mention

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Jamie Ayton
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Industries
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Australia

Shirley Elliott
Darling International
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Edwin de Klerk
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Rotterdam 3024AC Netherlands

Nicole Silva
Caloy
Denair, CA 95316 USA

GOED NUTRACEUTICAL OILS

First Place

Lita Kelly
James Hutton Limited
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Honorable Mention

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Ernst Renner
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Katrine Mork
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Kvaløya 9107 Norway

MARINE OIL

First Place

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Honorable Mention

Nancy D. Roman
Melissa V. Thrift
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Reedville, VA 22539 USA

MARINE OIL FATTY ACID PROFILE

First Place

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Honorable Mention

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Kristopher Williams
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Zineb Ballouch
Olvea Atlantic
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John Reuther
Derek Yeadon
Eurofins Central Analytical Labs
New Orleans, LA 70122 USA

NIOP FATS AND OILS

First Place

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Mumtaz Haider
Amspec
Pasadena, TX 77503 USA

AOCS Accredited Panels

The following panels received an SPN score of 5 or less in the 2016–2017 AOCS proficiency testing program. To achieve this recognition, panelists must be AOCS member in good standing and sign an agreement stating that all of the requirements for testing were met.

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Chemiservice
Monopoli, Italy

Maria Garzon, Pompeian, Inc.
Baltimore, MD, USA

Jamie Ayton, NSW Dept. of
Primary Industries
Wagga Wagga, Australia

Natalia Ruiz, Modern Olives
Laboratory Services
Woodland, CA, USA

Claudia Guillaume, Modern
Olives Laboratory Services
Lara, VIC, Australia

Honorable Mention

Melanie Greer
Dallas Group
Jeffersonville, IN 47130 USA

NUTRITIONAL LABELING

First Place

Mathieu D'Amours
Nutreco Canada, Inc.
St. Hyacinthe QB J2R 1S5 Canada

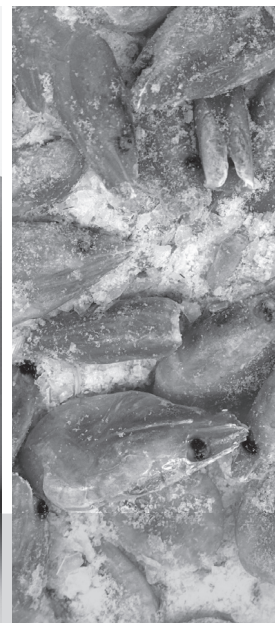
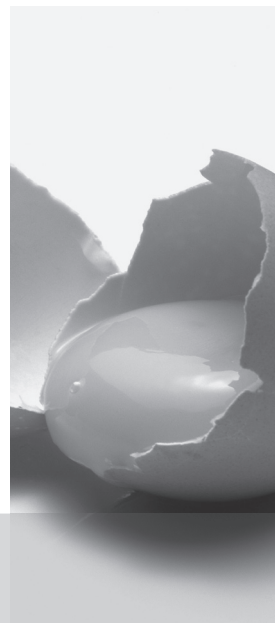
OILSEED MEAL

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Solbar QA Laboratory
Solbar Protein Food Ltd.
Ashdod 77121 Israel

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Joshua Bogan
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Memphis, TN 38116 USA

OLIVE OIL PART A*First Place*

Brenda Vest
Pompeian, Inc.
Baltimore, MD 21224

Honorable Mention

Maria Garzon
Alex Vargo
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Baltimore, MD 21224 USA

OLIVE OIL PART B*First Place*

Alex Vargo
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Baltimore, MD 21224 USA

Honorable Mention

Maria Garzon
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Wei Wang
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OLIVE OIL PART C*First Place*

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Honorable Mention

Alex Vargo
Brenda Vest
Pompeian, Inc.
Baltimore, MD 21224 USA

PALM OIL*First Place*

Magdy Rashwan
IFFCO Egypt
Suez 204 Egypt

Honorable Mention

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Gaik Ming Khoo
Intercontinental Specialty Fats
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Jalan Banting, Dengkil Malaysia

PEANUT SEED*First Place*

USDA AMS S&T Science Specialty
Lab
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PHOSPHORUS IN OIL*First Place*

Laboratory Portugal SA
Sovena Oilseeds
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Honorable Mention

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SOLID FAT CONTENT BY NMR*First Place*

Carrie Abrath
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Honorable Mention

Hiroshi Hirai
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SOYBEAN OIL*First Place*

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Honorable Mention

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SOYBEANS*First Place (tie)*

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Kester Emefina
Mumtaz Haider
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The Laboratory Certification Program is sponsored by AOCS and The National Oilseed Processors Association (NOPA). Certification entitles a laboratory to serve as a NOPA referee for soybean meal analyses, and publication in the annual edition of the *NOPA Rule Book*.

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Honorable Mention

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SPECIALTY OILS*First Place*

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Kent Karsjens
Keith Persons
Anders Thomsen
Eurofins Scientific
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TALLOW AND GREASE*First Place*

Adalberto Coronado
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Honorable Mention

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Laboratory Team
Sanimax Aci, Inc.
Charny PQ G6X 3R4 Canada

TRACE METALS IN OIL*First Place*

QA Team
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Honorable Mention

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TRANS FATTY ACID CONTENT

First Place

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Honorable Mention

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2016–2017 Olive Oil Sensory Panel test

AOCS thanks all of the panels that participated in the 2016–2017 Olive Oil Sensory Panel test.

AOCS Recognized Panels

The following panels attained a low SPN score in the 2016–2017 AOCS proficiency testing program. There was a four-way tie for first place. Congratulations to all of the winners.

First Place

**Agricultural Products
Distribution Division**
Kagawa, Japan

Modern Olives
California, USA

Multichrome Lab
Athens, Greece

Pompeian, Inc.
Maryland, USA

Honorable Mentions

Sunset Olive Oil, LLC
California, USA

Fabrica Torrejana SA
Riachos, Portugal

**NSW Department of Primary
Industries**
Wagga Wagga, Australia

Applied Sensory, LLC
California, USA

Chemiservice SRL
Bari, Italy

California Olive Oil Council
California, USA

UNGROUND SOYBEAN MEAL

First Place

QA/QC Laboratory
Vietnam Agribusiness Ltd.
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Honorable Mention

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Brad Beavers
Jennie Stewart
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Edgar Tenent
K-Testing Lab, Inc.
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Michael Hawkins
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Cannabis extraction

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Eric Miller

- Historically, the preferred delivery method for cannabinoids was smoking dried plant material.
- However, the mass appeal of edibles, capsules, tinctures, transdermal patches, topical lotions and creams, beverages, and other products infused with concentrated cannabis compounds is growing due to the health risks associated with smoking combustible material.
- This article reviews the variety of extraction and purification methods used to make cannabis concentrates, as well as safety concerns about the methods themselves and the resulting concentrated products.

The resinous oil of the cannabis plant consists of cannabinoids, terpenoids, and flavonoids. This oil is sequestered in hair-like glandular trichomes protruding from the surface of the leaves and female inflorescences (buds). The psychoactive cannabinoid tetrahydrocannabinol (THC) and non-psychoactive cannabidiol (CBD) have for centuries been revered (albeit ambiguously) for their recreational and medicinal properties. Historically, the preferred delivery method for cannabinoids was smoking the dried plant material. Since the early 1900s, extraction of cannabinoids from the plant by relatively non-polar solvents has gained popularity, skyrocketing since the turn of the millennium. In October 2016, *Inform* magazine's cover story, "The highs and lows of cannabis testing" reviewed the equipment, methods, and standard procedures analytical laboratories use to measure potency and identify contaminants in cannabis products. This follow-up article takes a look at the variety of extraction and purification methods used to make cannabis concentrates, as well as the safety concerns of the methods themselves and the resulting concentrated products.

The utility of extraction is easy to understand: Concentrates serve as the feedstock for myriad infused products with mass appeal—edibles, capsules, tinctures, transdermal patches, topical lotions and creams, and beverages—that arose from the attempt to mitigate health risks related to inhaling combusted marijuana. The vaporization of concentrates also offers a more healthful option than smoking, while the fast onset of THC and CBD vapor blurs the line between medical and recreational use in such a way that the vape cartridge has become the delivery method of choice for patients and tourists alike.



As the number of states that have legalized marijuana continues to grow, the manufacturing sector of the cannabis industry is maturing with respect to extraction, purification, formulation, instrumentation, and analytics. The driving force behind this evolution is safety: consumer safety, laboratory safety, and environmental safety. While microbial, heavy metal, and pesticide contaminants may primarily be a result of cultivation failures, residual solvent contamination of extracts is a consequence of poor manufacturing practices. State regulations are forcing manufacturers to comply with allowable solvent limits on inhalation and oral consumption bases. As a result, clandestine operations with unqualified personnel are giving way to licensed laboratories with trained professionals. Compliance with US Environmental Protection Agency (EPA) and US Occupational Safety and Health Administration (OSHA) regulations, and Good Manufacturing Practices (GMP) and Good Laboratory Practices (GLP) guidelines are increasingly being integrated into cannabis manufacturing business models. "Cannabis manufacturing facilities are well-served by adopting pharmaceutical GMPs and Quality Management Systems," says Andrew Samann, CEO of OrionGMP. "Today's entrepreneurs want to build quality, sustainable, and profitable businesses that protect consumer health and safety; engineering their manufacturing processes to pharmaceutical guidelines will ensure all those goals are met."

In an effort to broaden the ever-shrinking profit margins brought on by regulations and competition, manufacturers

are adapting scalable technologies from established industries and legitimizing nascent technologies from the black market. Supercritical fluid extraction (SFE) using CO₂ is an established scalable technology that has been adapted for cannabis extraction, while liquefied petroleum gas (LPG) extraction is an example of a former black market technology that, when done properly, is safe and economical. LPG (butane, propane, or a mixture of both) and supercritical CO₂ are very efficient solvents for nonpolar compounds such as cannabinoids and terpenoids. Their volatility at atmospheric pressure allow for easy removal of residual solvent. Both SF and LPG extraction must be performed with closed-loop systems which dramatically reduce atmospheric waste and—in the case of LPG—the risk of explosion. Ethanol extraction remains a popular extraction solvent due to safety and availability. Thanks to its relatively low volatility, there are a number of configurations for ethanol extraction.

The success of such solvents is underpinned by their effectiveness. However, co-extraction of undesired compounds is inevitable, considering the complex matrix of the cannabis plant. Sheathed by a cuticular layer of wax, trichomes are terpenoid and cannabinoid factories that protect the plant against herbivores and insects, and also attract pollinators to the female flowers. Fortunately, the trichome is a protruding structure and, thus, fairly exposed to the solvent. This results in cannabinoid contents exceeding 50% w/w in crude extract. The remaining content consists of terpenoids, waxes, and pigments.

More than 150 cannabinoids have been characterized to date, but just two cannabinoids garner most of the attention: Δ^9 -tetrahydrocannabinol (THC) and cannabidiol (CBD). THC, CBD, and the scores of other cannabinoids contain (with few exceptions) 21 carbon atoms and 2 oxygen atoms. The nonpolar nature of cannabinoids preclude aqueous extraction, as the partition coefficients (THC and CBD $pK_{ow} \approx 7$) prevent water solubility (1–2 $\mu\text{g/mL}$).^{1,2}

Terpenoids are the aromatic essential oils that give the cannabis plant its characteristic bouquet. Physiologically, the terpenoid profile modulates the psychoactive effect of THC at the receptor level of the central nervous system while producing an aromatherapeutic effect in the olfactory system of the brain. Chemically, terpenes are biosynthesized from isoprene (C_5) units to form monoterpenes (C_{10}), sesquiterpenes (C_{15}), diterpenes (C_{20}), and so on. Because oxygen is commonly incorporated into the molecule, this group of secondary metabolites is collectively referred to as terpenoids. The importance of terpenoids in vaporized concentrates is gaining recognition to such an extent that the chemical syntax has become commonplace to the Cannabis connoisseur. For example, myrcene, limonene, and β -caryophyllene are not only readily identifiable in a vapor, but their physiological effects are also understood by the discriminating consumer. Thus, co-extracted terpenoids are not considered to be contaminants but rather valuable components which can be further refined and isolated during post-processing.

Waxes originating from the cuticle and secretory vesicles within the trichome are generally considered a nuisance with little value as a byproduct. Precipitated by ethyl or isopropyl alcohol at low temperature, most of the wax content can be removed by vacuum-filtration. The remaining wax can be removed by distillation of the cannabinoids, whereby the waxes are left behind in the less volatile fraction. Under vacuum, thermal degradation of the cannabinoids can be avoided by exploiting their higher vapor pressures.

Chlorophyll and other pigments are solubilized by more polar solvents, such as ethanol and—in the case of incompletely dehydrated plant material—water. Chlorophyll can be removed downstream with activated carbon and bleaching earths, but circumventing pigment contamination can largely be achieved by thoroughly drying the plant prior to extraction and through proper chilling of ethanol during the extraction process.

In addition to dehydration, the cannabis plant material can be pre-processed to increase the efficiency of the extraction solvent and therefore improve product yield and potency. Decarboxylation of cannabinoids by judiciously heating the plant material will convert the *in vivo* carboxylic acid form of the cannabinoids (eg., THCA, CBDA) to the neutral form (THC, CBD). This decarboxylation step renders the cannabinoid less polar which enables it to cross the blood-brain barrier and subsequently activate neuronal receptors. The neutral cannabinoids are also more soluble in carbon dioxide and hydrocarbons, making the extraction process faster and more efficient. Even so, decarboxylation is not always optimal. For example, THCA crystallizes while THC does not, making the acid form a

prerequisite for high-purity isolates. Furthermore, recent studies have shown that the acid form of cannabinoids may have therapeutic applications which differ from those in the neutral forms. With such a variety of medical applications of cannabinoids, the extraction process can be customized according to the process or therapy.

Once the plant material has been prepared, the compounds of interest can be extracted. Carbon dioxide, LPG, and ethanol all have their advantages and disadvantages, both chemically and economically. A brief review of these cannabinoid solvents will explain their popularity in extraction facilities.

CARBON DIOXIDE

Carbon dioxide is in the supercritical fluid state of matter at temperatures and pressures above the critical point ($T = 31^\circ\text{C}$, $P = 1071$ psi for CO_2). In the supercritical state, CO_2 has both the density of a liquid and the diffusivity of a gas. This dichotomy allows CO_2 to quickly permeate through solid plant material while also having the capacity to solubilize considerable amounts of cannabinoids and terpenoids. By virtue of its relatively low supercritical temperature, CO_2 has the added benefit of preserving thermally labile compounds. Supercritical CO_2 is also highly tunable: specific components can be selectively extracted by slight variations in temperature and pressure. For these reasons, CO_2 has broad applications ranging from decaffeination of coffee to essential oil extraction, green synthesis reactions, and pesticide removal.

The solubilities of THC and CBD in supercritical CO_2 (1–2 g/kg CO_2) increase with temperature and pressure.^{3,4} However, the higher density of CO_2 at lower temperature offsets the faster mass transfer rate by offering greater solvent capacity.

References

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Therefore, many extraction facilities prefer low operating temperatures and longer run times to yield a product with minimal thermal degradation and essential oils rich in delicate monoterpene top notes. This is especially true when fractionating terpenoids and THCA at liquid (subcritical) CO₂ parameters. As extraction facilities scale to meet the needs of an expanding market, the demand for high-throughput extraction is being met with CO₂ pumps that can achieve flowrates up to 20 kg/min and pressures exceeding 5000 psi.

The cannabis extract market is continually creating new products and borrowing sophisticated technologies from other industries. Just as the cooking oil industry utilizes SFE for degumming, clarifying, and purification, the cannabis industry will eventually integrate fractionating columns, particle technology, co-solvents, and anti-solvents into the manufacturing process. Although the capital investment is very high for SFE, the transition from black to white market is stimulating financing.

Liquefied petroleum gas extraction, commonly known as butane hash oil (BHO), is a niche extraction method that has evolved from a dangerous, dirty, and highly illegal hobby to an accepted and regulated practice. Closed-loop extraction systems fabricated to ASTM standards have all but replaced the improvised contraptions in residential garages. High purity butane and propane are becoming status quo while the use of convenience store lighter refills are now considered a disgrace to the profession. Active recovery of purged butane and propane back into their LPG states through the use of a sparkless pump helps speed up the process; passive recovery uses hot and cold water baths to wheedle the solvents back to their storage tank. In states where LPG extraction is legal, the process must take place in a Class I Division I facility in accordance with the National Electric Code (NEC). In Colorado, fire codes require that LPG extraction facilities have detection systems that alert occupants when butane or propane vapors are present at levels above 10% the lower flammable limit (LFL). Fire suppression systems, exhaust systems, and electrical systems are mandatory safety features to prevent gas accumulation, sparks, explosions, and fire.

When done properly, LPG extraction is a safe and efficient technique. Butane and propane are highly selective for cannabinoids and terpenoids with yields rivaling those of hexane and supercritical CO₂ (>90%). Wax content is minimal, and when extracting high-quality plant material, post-processing can be avoided altogether. The speed with which LPG extracts THC underscores the high solubility of cannabinoids in butane and propane. The relatively low pressures needed to liquefy propane and butane reduce the risk of vessel or valve failures. Despite these safety measures, LPG extracts still suffers from bad publicity stemming from isolated incidents of explosions and solvent contamination of concentrates that were not adequately purged.

Ethanol is publicly accepted in many forms—as a beverage, antiseptic, and fuel—and is no less acceptable as an extraction solvent. Ethanol's low volatility and familiarity makes it an attractive option for cannabinoid extraction. Its main drawbacks are nonselective extraction of chlorophyll, an

energy-intensive evaporation process, and a high cost. Ethanol costs can be kept to a minimum with an efficient rotary evaporator, but considerable losses will occur, because the plant material acts as an alcohol sponge; squeezing requires a screw press. The polarity of ethanol results in chlorophyll contamination, which can be problematic to remove downstream. At high temperatures in a post-processing step such as distillation, the magnesium in chlorophyll can cause charring of complex and expensive glassware. Chlorophyll extraction can be minimized by chilling ethanol below -40°C. Closed-loop systems are effective for temperature control as well as increasing throughput via solvent flux.

Cannabis extraction technology will continue to progress at the pace of product development and consumer demand. Showing no signs of slowing down, the cannabis industry is poised to join the ranks of pharmaceutical, supplement, and food and beverage companies as a leader in natural products technology.

Eric Miller has a Ph.D in chemistry and is the founder of Phytophile Consulting in San Diego, CA, USA. He specializes in natural products extraction and analytics of Cannabis and marine algae. He can be contacted at Eric@phytophile.com.



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NFPA 652: how to prepare for the new standard on combustible dusts

Matt Williamson

The original standards for combustible dusts were developed for specific industries, such as NFPA 61 for agricultural facilities and NFPA 664 for woodworking facilities; NFPA 654 was established to serve as a general standard for any facility not covered under one of the industry-specific standards. However, these standards often conflicted and left some industries with unclear direction on specific requirements. An overarching umbrella standard was needed to coordinate all of the industry specific standards and to provide guidance to assist all industries in the development of their own combustible dust control plans based on their individual needs. To do this, the National Fire Protection Association (NFPA) issued NFPA 652, *Standard on the Fundamentals of Combustible Dust*, on September 7, 2015.

- If your facility produces or handles combustible dusts, then you're well aware of its dangers and risks.
- With the implementation of NFPA 652, there is a new combustible dust safety standard.
- Here's what you need to do to get into compliance by the fast-approaching deadline of September 2018.

INTENT

The purpose of NFPA 652 is to establish the relationship and hierarchy between industry-specific standards and to ensure that fundamental requirements are consistently addressed across all industries, processes, and dust types. Existing standards that now fall under the umbrella of NFPA 652 include:

- NFPA 61, *Standard for the Prevention of Fires and Explosions in Agricultural and Food Processing Facilities*;
- NFPA 68, *Explosion Protection by Deflagration Venting*;
- NFPA 69, *Explosion Prevention Systems*;
- NFPA 484, *Standard for Combustible Metals*;
- NFPA 499, *Classification of Combustible Dusts and of Hazardous (Classified) Locations for Electrical Installations in Chemical Process Areas*;
- NFPA 654, *Standard for the Prevention of Fire and Dust Explosions from the Manufacturing, Processing, and Handling of Combustible Solids*;
- NFPA 655, *Standard for the Prevention of Sulfur Fires and Explosions*; and
- NFPA 664, *Standard for the Prevention of Fires and Explosions in Wood Processing and Woodworking Facilities*.

THE DUST HAZARD ANALYSIS (DHA)

NFPA 652 introduces one major requirement which was not previously included in the industry-specific standards: the **Dust Hazard Analysis (DHA)**. A DHA is a hazard identification and risk mitigation plan for managing combustible dust hazards, similar to a Process Hazard Analysis (PHA). Under NFPA 652, a DHA is now required for all operations that generate, process, handle, or store combustible dusts or particulate solids. This includes all oilseeds crush plants and grain elevators. For existing facilities, a DHA must be completed by September 7, 2018, three years after the implementation of NFPA 652. This requirement

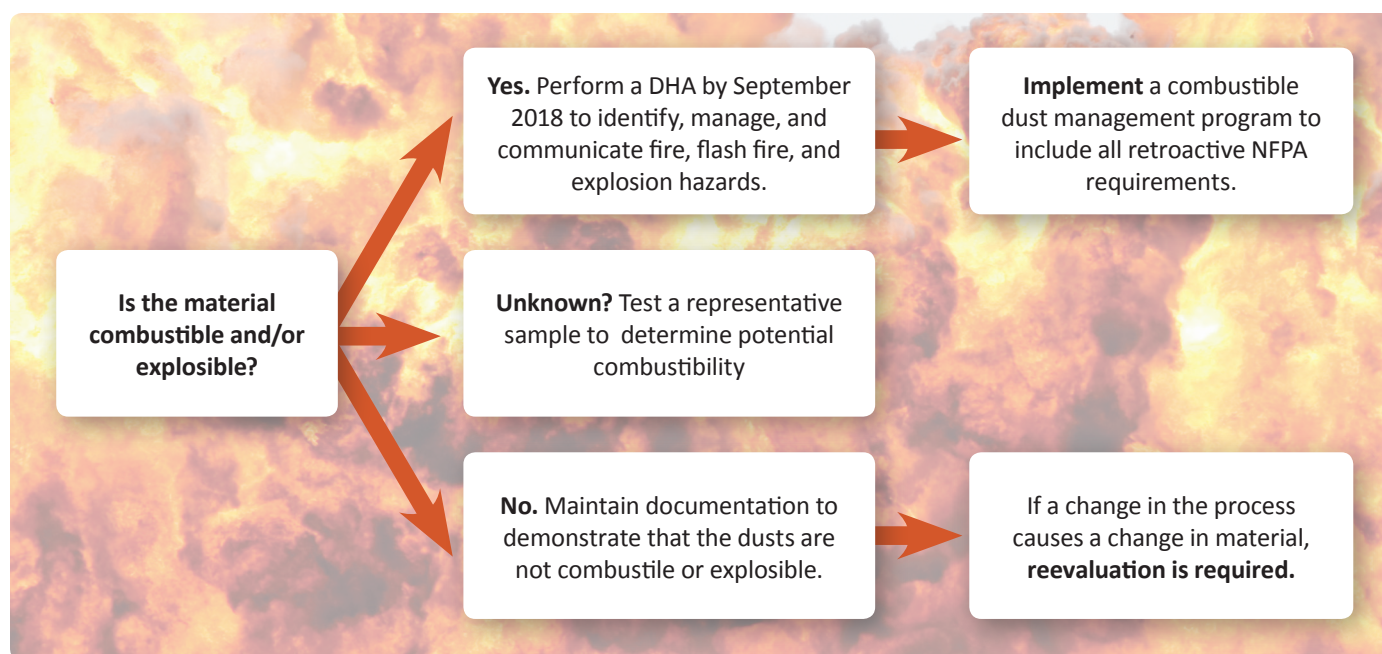


FIG. 1. The flow chart depicts whether or not a DHA will be required.

is retroactive, meaning all facilities that handle combustible dusts, regardless of age, must complete a DHA by the deadline.

NFPA 652 specifies that the facility owner or operator is responsible for determining if the handled materials are combustible or explosive. If so, they must also characterize material properties for the DHA. Figure 1 depicts whether or not a DHA will be required.

WHAT DOES A DHA REQUIRE?

The DHA document must include the following:

- combustible dust characteristics (Kst, Pmax, etc...) for all dusts, or evidence that it is non-combustible;
- identification of areas where the potential for a dust explosion exists;
- identification of the paths (ducts, vents, inlet air filters) where a dust explosion may travel with the potential for secondary explosions or personnel exposure;
- credible ignition sources and dust suspension mechanisms;
- safe operating ranges;
- existing protection methods;
- explosion propagation hazards; and
- additional protection recommendations and implementation plan.

The first and most critical requirement of the DHA is to determine and document the combustibility characteristics of all potentially combustible dusts on the site. Even if the dusts are known to be non-combustible, as with most inorganic minerals or salts, some evidence must be documented as part of the plant's safety records. For all carbonaceous, organic, or metal dusts, combustibility data must be documented. This data should include the following items:

1. Kst—defines the explosive potential of the material and is used to size explosion relief panels
2. Pmax—the maximum potential pressure developed by an explosion and is used in equipment pressure design if containment is an option, and for isolation system rating
3. dp/dt —the rate of pressure rise and is part of the explosion panel sizing calculation, as well as explosion isolation or suppression equipment rating
4. MIE—the minimum ignition energy defines the sensitivity of the material to sparking, such as from brush static electricity, and can determine PPE requirements for handling by personnel or for room humidification
5. MEC—the minimum explosible concentration defines the quantity of dust that is a risk for a fixed volume, such as the headspace of a bin
6. MIT—the minimum ignition temperature for a dust layer defines the electrical equipment and motor insulation specifications

Most facilities will have at least some of this data. Where data is insufficient or missing for common materials, some combustibility data from external sources, such as literature or online databases, may be used to fill gaps or establish a baseline. However, combustibility is sensitive to parameters that are likely specific to the site, including particle size, particle shape, moisture, and oil content. Dust from a flaking operation will behave differently than dust from the spent meal following extraction, for example. This also means that data from literature may not be sufficient to meet DHA requirements. Each site should have their own dusts tested and characterized to understand the potential impacts and explosion protection equipment sizing parameters specific to their operation.

Typical data for soybean dust (not to be used for design, field data will vary) are:

- Kst—50 - 80 bar m/s
- Pmax—8.5 bar/g
- dP/dt—55 bar/s
- MEC—60 g/m³
- MIE—100 mJ
- MIT—190°C

In general terms, soybean dust tends to be less combustible than canola, but more combustible than dry corn dust—although particle size and moisture content are overriding factors.

COMBUSTIBLE DUST MANAGEMENT PROGRAM

If combustible dust is present in your facility, several management systems should be in place to control the hazard, as is determined based on the DHA. Implementing a combustible dust management program will include all of the retroactive requirements of NFPA 652 depicted in Figure 2.

RELATION TO THE OSHA NATIONAL EMPHASIS PROGRAM

The US Occupational Safety and Health Administration (OSHA) implemented a National Emphasis Program (NEP) for combustible dusts in 2007 (CPL-03-00-008). The following year, 14 people were killed when the Imperial Sugar Refinery exploded in the state of Georgia. OSHA began work on a regulation governing combustible dusts in 2009, but has yet to issue its own proposed rule. Instead, under the NEP, OSHA has issued citations and fines each year based on the existing variety of industry-specific NFPA standards, under their General Duty clause.

With NFPA 652 now in place, OSHA will be able to follow its hierarchy to enforce a consistent set of requirements on all facilities, regardless of industry, including having DHA documentation and following its site specific implementation plan, similar to the way a Process Safety Management (PSM) program is expected to implement a PHA. Although OSHA has not formally adopted NFPA 652, it has never formally adopted any standard on combustible dusts and does not need to. The

OWNER/OPERATOR GENERAL REQUIREMENTS

- Determine combustibility and explosibility hazards of materials
- Identify and assess any fire, flash fire, and explosion hazards (perform DHA)
- Manage identified fire, flash fire, and explosion hazards
- Communicate hazards to affected personnel

HAZARD MANAGEMENT MITIGATION AND PREVENTION

- Building design
- Equipment design
- Housekeeping
- Ignition source control
- Personal Protective Equipment (PPE)
- Dust control
- Explosion prevention and protection
- Fire protection

MANAGEMENT SYSTEMS

- Operating procedures and practices
- Inspection, testing, and maintenance
- Training and hazard awareness
- Contractors
- Emergency planning and response
- Incident investigation
- Management of change
- Documentation retention
- Management systems review

FIG. 2. Retroactive requirements of NFPA 652

administration's consistent use of the General Duty Clause has taken care of that.

Matt Williamson has nearly 30 years of process engineering and project management experience and is a recognized expert on combustible dusts and related regulations. He is the Process Engineering Department Manager at ADF Engineering, Inc., based in Miamisburg, Ohio, USA. He can be contacted at mwilliamson@adfengineering.com.



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Foam optimization strategies in consumer formulations

Shui Ping Zhu, Ron Masters, Sarah Kovach, and Sangeeta Ganguly-Mink

- It is well established that each distinct cleaning application, formulation, or product needs a suitable foam profile.
- Some products, such as hand dishwashing detergents and shampoos, need high and sustainable foam. Other products, such as auto dishwasher detergents and swimming pool disinfectants, need low and fast-collapsing foam.
- This article reviews some basic foam-optimization principles, and examples of how to increase and decrease the foam of various surfactants and their formulations, based on application.

Foam consists of bubbles linked together. In many applications, such as in shampoo and hand dish detergents, foam is desirable; while in many other applications, such as in auto dishwashing gel and water treatments, low, no, or fast-collapsing foam is preferred. To control a product's foam profile, it is helpful to understand some principles related to foam and the factors that impact it.

PRINCIPLES GOVERNING FOAM GENERATION AND BREAKING

The nature of surfactants to adsorb onto water surfaces to reduce Gibbs free energy is the driving force for foam generation. The maximum adsorption happens at or above critical micelle concentration (CMC).

Once a bubble is formed—whether it floats under water or floats to the surface—there is either a single or double layer of film(s) of adsorbed surfactant molecules. Due to the bubble film curvature, there is usually a difference between the air pressure inside and outside the bubble. This pressure differential is the force that drives bubbles to break and, ultimately, foam to collapse.

To achieve a high initial foam height based on the Ross-Miles foam test method, the surfactant molecule needs to move quickly enough to reach new adsorption equilibrium on the newly increased area when a bubble floats to the top of the liquid and its size increases (Fig. 1).

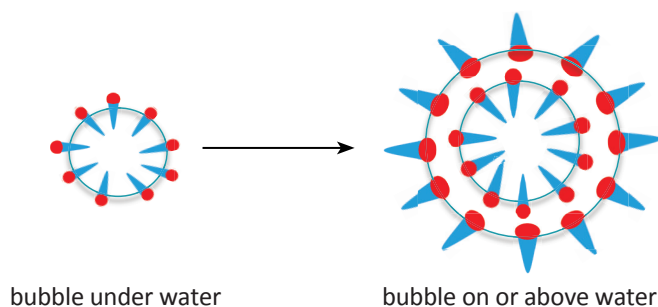


FIG. 1. Structure and size changes of a bubble in water vs. at the surface

When a bubble changes its film from a single layer to double layers and increases its size, the pressure difference also changes, as indicated by Young-Laplace equations (1) and (2).

$$\Delta P = \gamma \left(\frac{1}{r_1} + \frac{1}{r_2} \right) = \frac{2\gamma}{R_1} \dots \dots \dots (1)$$

$$\Delta P = 2\gamma \left(\frac{1}{r_1} + \frac{1}{r_2} \right) = \frac{4\gamma}{R_2} \dots \dots \dots (2)$$

Where:

γ is the surface tension.

ΔP is the air pressure difference.

r_1 and r_2 are the curvature radius, and R_1 is radius when a bubble is in a ball shape.

R_2 is the average radius of the double-layer films when a bubble is in a ball shape.

Equation (1) is used for the bubble under water with a single layer film

Equation (2) is used for the bubble on or above the water surface with a double layer films.

FACTORS IMPACTING FOAM

A bubble consists of both double-layer films and the liquid between the double-layer films (Fig. 2).

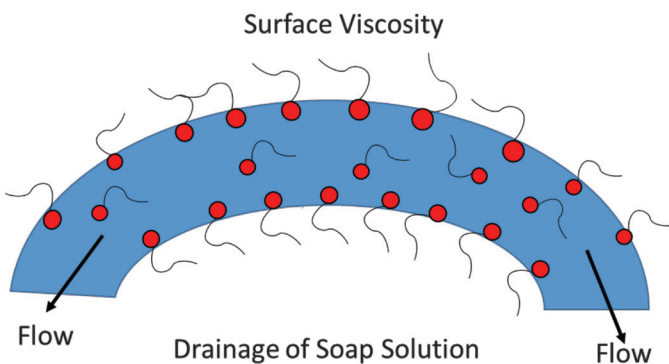


FIG. 2. Structure of a bubble

It is important to consider factors impacting both the films and the liquid. Among many factors impacting bubble (and ultimately foam) formation and breaking, there are three main factors impacting the films and four main factors impacting the liquid. For impact on film, presence of electrolyte, complex formation with electrolyte, and surfactant tail structure are crucial to consider. Viscosity, temperature, presence of polymer, and solvent are the factors impacting liquid.

FILMS

Electrolyte's impact on film: The impact of electrolytes on foam can be described by Gibbs adsorption isotherm equations (3) and (4).

$$\Gamma = -\frac{c}{RT} \frac{d\gamma}{dc} \dots \dots \dots (3)$$

$$\Gamma = -\frac{c}{2RT} \frac{d\gamma}{dc} \dots \dots \dots (4)$$

Where:

Γ is the adsorption amount of surfactant molecules per unit surface area. The bigger the number, the better packed the surfactant molecules are in the film, and hence more stable foam is obtained.

c is the surfactant concentration at or below CMC

R is the Avogadro constant

T is the system temperature

Equation (3) is applicable for ionic surfactant(s) with 1:1 type electrolyte (e.g., NaCl). Additionally, for equation (3) to be predictive, the electrolyte concentration must be much higher than the surfactant concentration. Equation (3) is applicable for nonionic surfactants, regardless of the presence of electrolytes.

Equation (4) is used for ionic surfactants when no electrolytes are present.

Equations (3) and (4) indicate that electrolytes help to achieve more stable foam generated by ionic surfactants, but the same is not achieved in the case of nonionic surfactants.

Electrolytes can be many different types of molecules which generate both positive and negative ions when dissolved in water, as shown in the following example.

Electrolytes and Counter ions: 1: n ($n \geq 1$)

NaCl, ionic surfactant like ROSO₃Na ($n=1$)

Na₂SO₄, CaCl₂ ($n=2$)

Na₄EDTA, TKPP ($n=4$)

H₃Na₇DTPMP ($n=10$)

Polyquaternium or Polidronium Chloride ($n>100$)

Na Polyacrylate ($n>100$)

One of the charged ions from an electrolyte can be the counter ion for ionic surfactants, such as an anionic, cationic, amphoteric/zwitterionic, and alkylamine oxide.

Complex's impact on film: An ionic surfactant molecule can form a complex with counter ion(s) from the electrolyte. Whether and how a complex impacts the film or foam depends on the water solubility of the complex formed.

Tail structure's impact on film: Generally, a straight carbon chain tail helps to achieve a relatively stable foam, while a branched tail helps to reduce foam. Multiple straight tails behave similar to a branched tail.

LIQUIDS

The amount of liquid is an important factor to provide stable foam. A higher amount of liquid between the double-layer films generates a rich, lathery foam. Dry foam with less liquid is less flexible and more susceptible to breakage. Four fac-

tors discussed below can impact liquid alone, or they can be interdependent.

Viscosity: Increased viscosity helps to retain water, thereby stabilizing the foam. Viscosity can be impacted by temperature change, addition of polymer, electrolytes, solvent, hydrotropes, or other factors.

Temperature: Higher temperature speeds water evaporation, drainage, and reduces the liquid viscosity, which destabilizes the foam. On the other hand, increased temperature helps nonionic surfactant molecules move faster to reach new adsorption equilibrium between the liquid and films in the newly increased surface area during foam generation, which generates a higher initial foam.

Polymer: A water-soluble polymer generally helps to achieve higher viscosity and to stabilize foam if there is no strong complex formed between the ionic surfactant and the polymer. It is pointed out that ionic surfactants of anionic, cationic, and amphoteric/zwitterionic—including alkylamine oxide—can form strong complexes with ionic polymers, depending on the concentrations.

Solvent: Volatile solvents, such as ethanol or isopropanol, typically used in consumer products for physical stability or clarity of products at different storage temperatures, normally evaporate faster than water. This reduces liquid viscosity and hence also destabilizes the foam.

FOAM STABILITY EXPERIMENTS

Our foam stability data were obtained by following the Ross-Miles Foam Test method (ASTM D1173-53), unless otherwise indicated.

Behaviors of surfactant foams in tap water and deionized water

Figures 3–5 depict the foam profiles of surfactants in tap water and deionized (DI) water.

Figure 3 shows that in DI water 4% Na_2SO_4 helps to stabilize the foam, but not sufficiently since after dilution the electrolyte level is only 40 ppm compared to 200 ppm of surfactant molecules. When tested in tap water of about 100 ppm water hardness, there is a reduction of initial foam due to weak complex between $\text{Ca}^{2+}/\text{Mg}^{2+}$ and LAS ion, but foam stability is achieved due to the overall higher level of electrolytes.

Figure 4 shows the foam profiles of seven surfactants in tap water. Foam from surfactants of SLES-3EO (sodium lauryl ethoxylated sulfate-3EO), cocamidopropyl betaine (CAPB), NaLAS, and C12AO (lauryl-N,N-dimethyl amine oxide) are not much impacted by the hard water; but the foam from ADBAC (alkyldimethylbenzylammonium chloride) is not stable due to the complex formed between the cationic surfactant ion and CO_3^{2-} from tap water. The foam from AEO11-9 is low and relatively stable due to no impact from electrolyte presence as predicted by equation (3), and the low foam is due to the much higher molecular weight versus that of ionic surfactant. The foam from SLS (sodium lauryl sulfate) is low and short-lived due to the strong complex formed between the surfactant's anionic charge and Ca^{2+} from tap water.

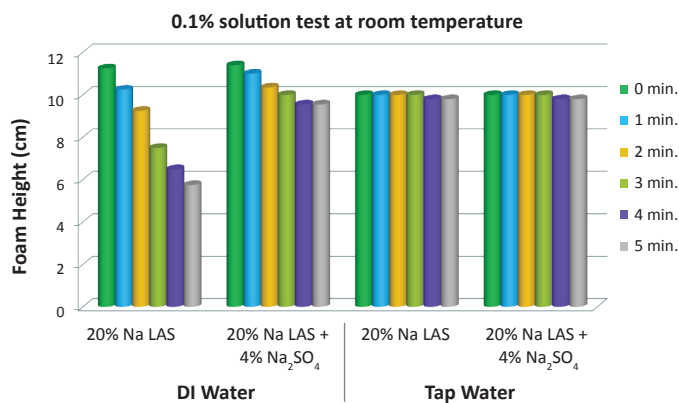


FIG. 3. Foam profile of Na LAS (linear alkylbenzenesulfonate) with and without 4% Na_2SO_4

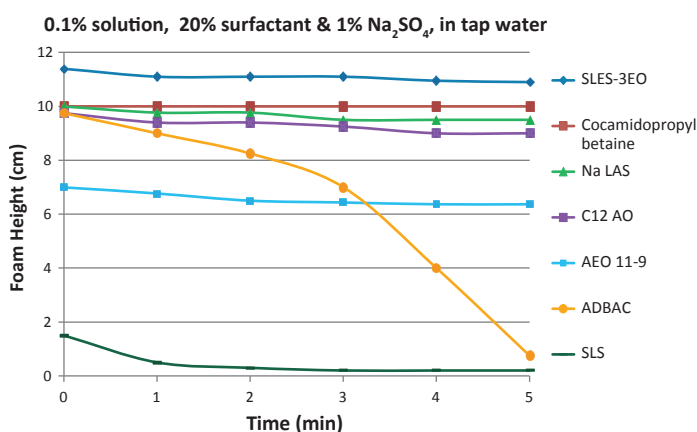


FIG. 4. Foam profile of 7 different surfactants in tap water

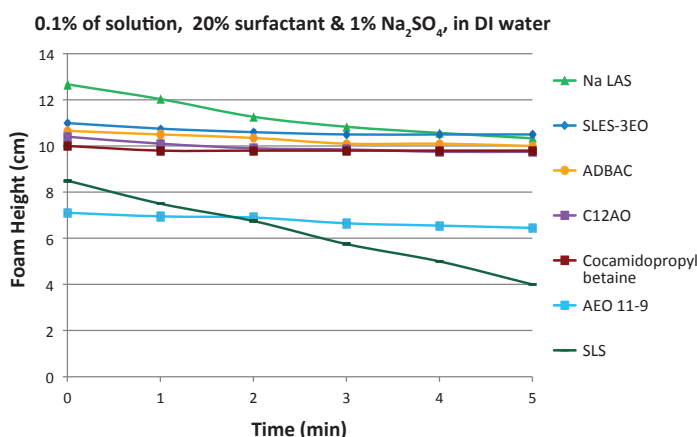


FIG. 5. Foam profile of 7 different surfactants in deionized water

Figure 5 shows foam profiles of surfactants in DI water. It should be noted that the foam profiles for surfactants of NaLAS, SLES-3EO, ADBAC, and C12AO are roughly the same as in tap water; but the foam profile for ADBAC is increased vs. its profile in tap water due to no or a much lower level of complex. Regular lab DI water still contains some water hardness and carbonates compared to super-pure water or ultra-pure water.

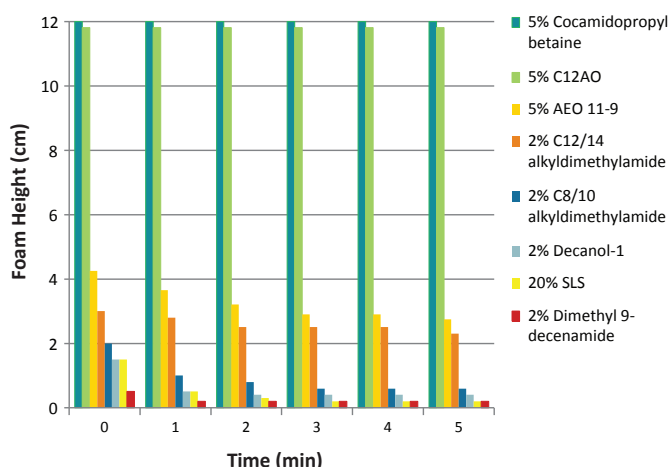
0.1% solution, 20% total (SLS + cosurfactant) & 1% Na₂SO₄ in tap water

FIG. 6. Foam enhancement for SLS anionic

Foam increase for different surfactants and formulations

The initial volume and stability of foam generated by anionic surfactants are not ideal, because their adsorption films are not tight enough. This is because repulsive forces from the surfactants' head group charge negatively impact film formation. However, the foam profile can be improved by adding amphoteric and nonionic surfactants, and co-surfactants, which results in a mixed and tighter film. Additionally, mixed surfactant films improve anionic surfactants' hard water tolerance to achieve better foam, as shown in Figures 6 and 7.

The foam profiles of mixed surfactant systems with anionic, amphoteric/nonionic, or with cationic, amphoteric/nonionic surfactant, could be improved by different strategies as shown in Figure 8.

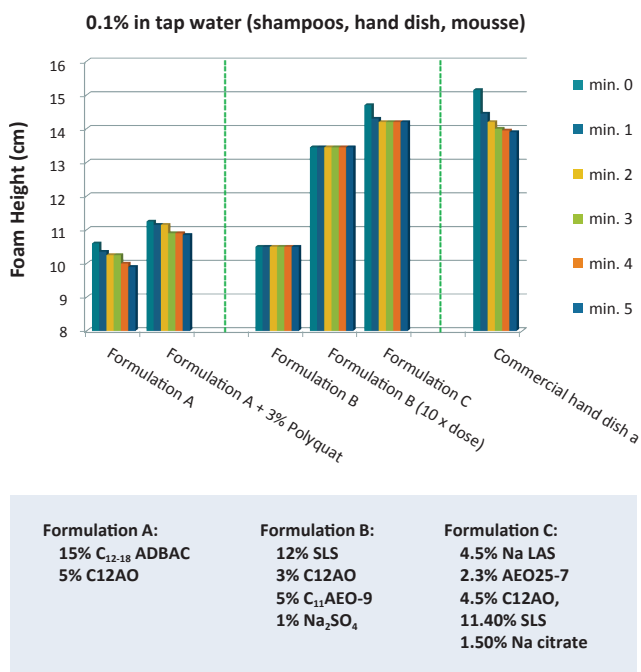


FIG. 8. Foam enhancement for a mixed surfactant system

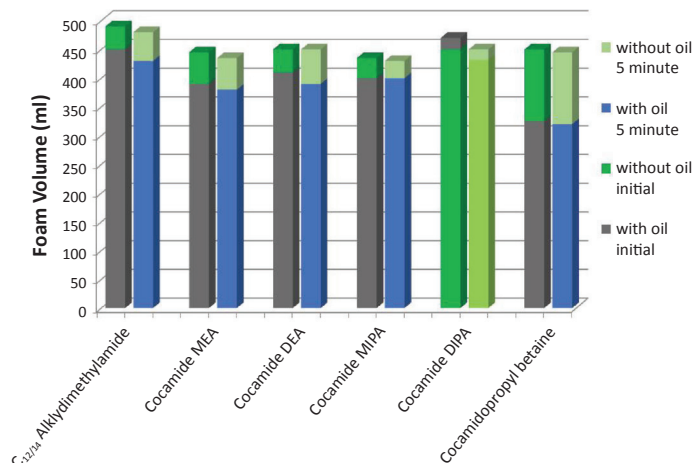
0.2% solution, tap water. Shaking Method.
100 ml solution/500 ml cylinder, 25°C

FIG. 7. Foam enhancement for SLES-2EO anionic + 1% other

Figure 8 shows that 3% polyquat (polidronium chloride with average molecular weight of 12,000 and with structure of $(\text{HOCH}_2\text{CH}_2)_3\text{N}^+\text{CH}_2\text{CH}=\text{CHCH}_2[\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}=\text{CHCH}_2]_n\text{N}^+(\text{CH}_3)_2\text{CH}_2\text{CH}=\text{CHCH}_2\text{N}^+(\text{CH}_2\text{CH}_2\text{OH})_3 \cdot (n+3)\text{Cl}^-$) can increase the foaming of Formulation A based on cationic and lauryl amine oxide, and the foaming of Formulation B based on anionic, lauryl amine oxide. The foaming of C₁₁AEO-9 can be increased by increasing the dosage and by surfactant-rebalanced Formulation C, which is more practical and cost effective.

Foam reduction for different surfactants and formulations

Foam reduction for different surfactants and formulations can be achieved in different ways. Figure 9 shows that solvent isopropanol and chelator EDTA can reduce the foam generated by a mixture of cationic surfactant ADBAC (C₁₂₋₁₈ alkyldimethylbenzylammonium chloride) and C12AO (lauryldimethylamine oxide), since IPA evaporates much faster than water, and the complex between EDTA and cationic surfactant is much less water-soluble, converting a single straight tail into multiple tails.

Trigger spray antimicrobial products: 0.2% sample in DI water

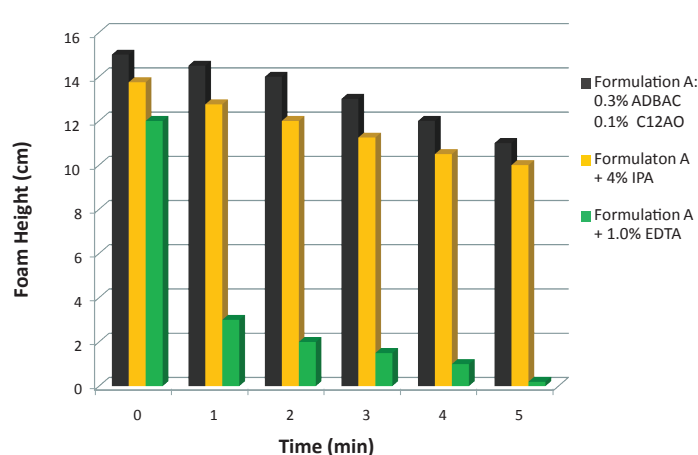


FIG. 9. Foam reduction for ADBAC

Figure 10 shows that double tails of cationic DDAC (didecylmethyl ammonium chloride) generate much lower foam than single-tail cationic ADBAC, since double-tail single-head group surfactants do not form tight adsorption films.

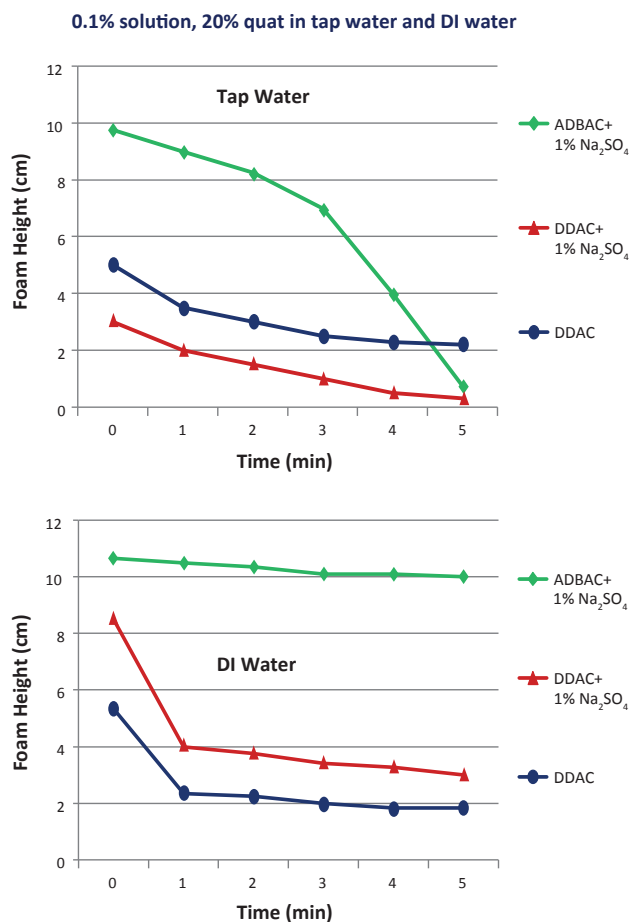


FIG. 10. Foam reduction from double tails of cationic surfactant

Figure 11 shows that different counter ions from organic propylenediamine sulfate, polyquaternium chloride, inorganic MgCl₂ and AlK(SO₄)₂ can reduce the foam generated by a mixed surfactant system, including 12% SLS (sodium laurylsulfate).

Figure 12 shows that coconut fatty acid sodium salt can reduce the foam for a commercial laundry detergent (regular, for top load), since its sodium salt can be turned into calcium/magnesium salt from the tap water, which is much less water-soluble and bears two tails.

TABLE 1. Auto dish wash gel foam profile

0.25% sample in tap water

Sample	Surface Tension	Clarity		Foam height over time: cm at minutes				
	mN/m	Tap W.	DI W.	Temp.	0	1	2	5
Lab Sample*	40.44	Clear	Clear	70F	1.8	0	0	0
				120 F	0	0	0	0

* 2.5% EOPO copolymer, 1.5% crosslinked polyacrylate, 5% Na citrate, 30% Na sulfate, DI water to 100%

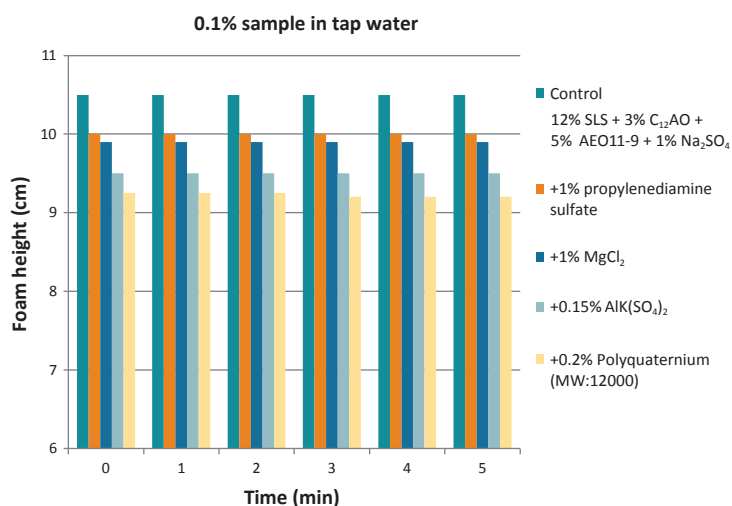


FIG. 11. Foam reduction from high valences of counter ions

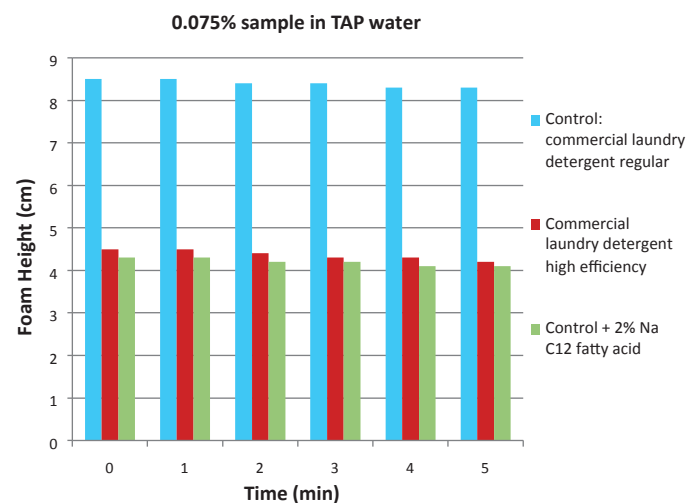


FIG. 12. Foam reduction by fatty acid sodium/calcium salt

Foam control from EOPO copolymer

EOPO copolymer is known to generate low or no foam, depending on its structure and molecular weight. It is worth pointing out that the foam of some EOPO copolymers and their impact on foam generated by other surfactants are related to the application temperature. Table 1 shows the relationship between foam and temperature for a lab-made auto dishwasher gel.

Figure 13 demonstrates in yet another application, spray wash, the foam reduction at high temperature by addition of EOPO copolymer. The EOPO copolymer effectively reduces the foam generated by other surfactants at 45°C, but doesn't at 25°C.

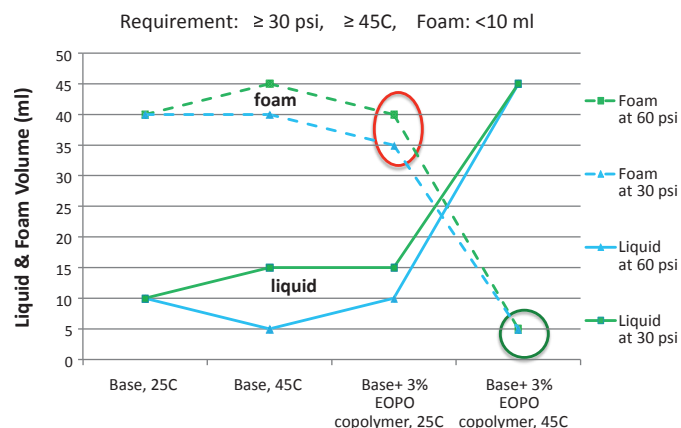


FIG. 13 EOPO copolymer foam reduction at different temperatures

Temperature's impact on foam

Temperature not only impacts the foaming of EOPO copolymer, but also of other surfactants or formulations as described in Table 2. Higher temperature increases the initial foam of AEO11-9 or formulations with AEO11-9 since it helps AEO11-9 move faster to reach new equilibrium for adsorption. But overall, increased temperature decreases foam stability, because it speeds up the evaporation and drainage of water between the double films.

Impact of counter ions and complexes on cationic surfactant foams

Counter ions and cationic surfactant can form complexes which may increase or decrease the foam, depending on the water solubility of the complexes and film strength as shown

TABLE 2. The impact of temperature on foams

0.1% sample (20% surfactants+1% Na_2SO_4) in tap water

Sample		t=0 min.	t=5 min.	IniMal foam	foam H change in 5 min	foam stability
Commercial Hand Dish b*	R.T.	15.4	13.9	base	9.7%	base
	120F	12.6	11.7	much lower	6.8%	better ?
SLS + C12AO +AEO11-9	R.T.	10.5	10.5	base	0.0%	base
	120F	11.4	11.2	higher	2.2%	worse
AEO11-9	R.T.	7.0	6.4	base	9.0%	base
	120F	8.3	6.3	higher	24.2%	worse
Na LAS	R.T.	10.0	10.0	base	0.0%	base
	120F	10.0	9.7	slightly lower	3.0%	worse
EO PO copolymer**	R.T.	1.8	0.0	base	100%	
	120F	0.0	0.0	much lower	N/A	worse

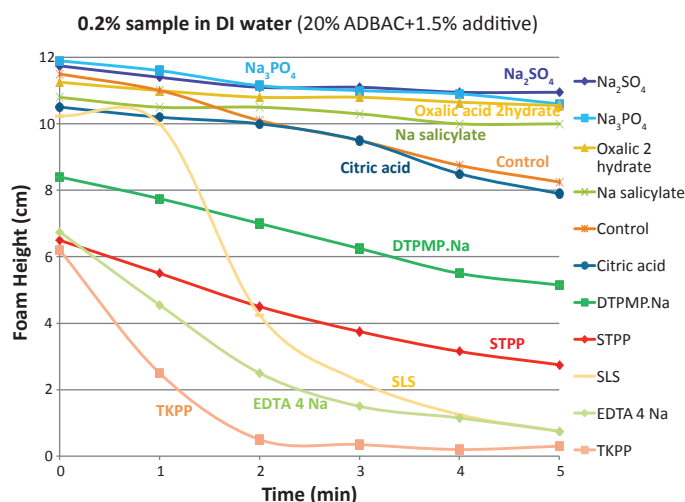
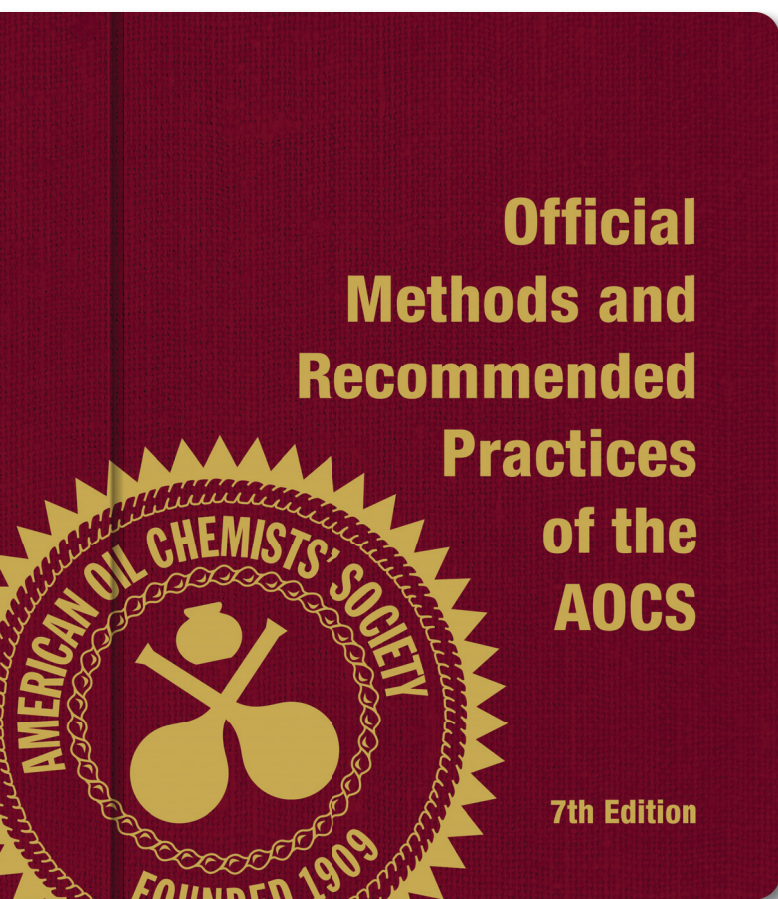


FIG. 14. Foam of cationic surfactant ADBAC impacted by different counter ions

in Figure 14. The additives studied impact ADBAC's foam very differently. It should be noted that anionic surfactant (SLS) and cationic surfactant (ADBAC) are mutual foam reducers.

Several conclusions can be drawn from these foam stability investigations. Water hardness normally reduces the foam of anionic surfactants. Foam from a single anionic surfactant can be improved by adding amphoteric/zwitterionic surfactants, nonionic surfactants and co-surfactants. Electrolytes form complexes with ionic surfactants which can result in increase or decrease of foam quantity and/or stability. Surfactant tail structures have impact on their foam profile. EOPO copolymer foam profile depends on the structure and application temperature. Raising temperature increases the initial foam of AEO nonionics, but renders the foam less stable.

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- ▶ Cd 30-15 (Official Method) Analysis of 2- and 3-MCPD Fatty Acid Esters and Glycidyl Fatty Acid Esters in Oil-Based Emulsions
- ▶ Ce 12-16 (Official Method) Sterols and Stanols in Foods and Dietary Supplements Containing Added Phytosterols
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Are edible oleocolloids the final frontier in food innovation?

Ashok R. Patel

- “Oleocolloids” represent a broad range of oil-continuous soft matter systems comprising colloids at an interface of complex fluids and phase-separated states of matter.
- Such oil-continuous systems have not received the same level of attention in food development as water continuous colloids have, but recent factors provide new incentives to develop innovations in formulating them.
- This article summarizes the microstructure and functionality of oleocolloids, their potential use in food formulation, the food-grade materials that can be used to create and stabilize them, the cost/benefits of using them, and the issues that need to be addressed in developing them.

“Oleocolloids” is used here as a term to represent a broad range of oil-continuous soft matter systems (Fig. 1) comprising colloids at an interface of complex fluids and phase-separated states of matter. Some examples include oleogels; biphasic colloids where water droplets or air bubbles are dispersed in an oil continuous medium (indirect emulsions and oleofoams, respectively); complex colloids such as foamed oleogels and oil-water-oil (O/W/O) emulsions; and layered structural matrixes embedding a large volume of liquid oil (oleofilms).

When it comes to innovation in food product development, oleocolloids have unfortunately not received the same level of attention as water-continuous colloids. The lack of interest in these systems is partly due to limited market needs (the range of food products based on them is narrow) and partly due to the relative ease of formulating these products with the help of partially hydrogenated oils (or saturated fats from natural sources) and synthetic emulsifiers, such as monoglycerides. However, a combination of recent factors, including regulations requiring the elimination of *trans*-fats from food products, rising concerns among consumers about ecological damage caused by palm oil production, and the industry’s drive toward more clean-labelled products have together provided new motivations for food material scientists to focus on innovations in formulating oleocolloids.

Although the drive for research in this area is stronger than ever, finding feasible solutions is an uphill task. The number of food-grade materials with the required properties needed to create and stabilize oil-continuous colloids is limited (Table 1), and identified materials must not only have the right structural features and molecular assembly properties (crystallization or supra molecular ordering), but also need to satisfy most (if not all) of the following conditions:

- approved for use as direct additive or formulation aid;
- effective at low concentration;

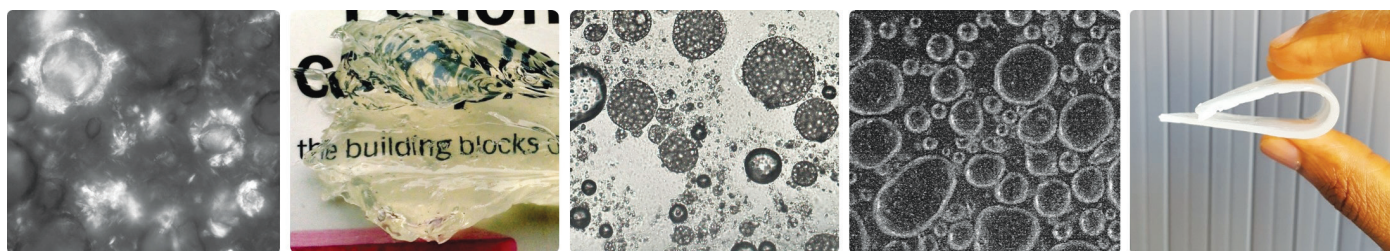


FIG. 1. Representative images of oleocolloids (from left to right): microstructure of surfactant-free water-oil (W/O) emulsion stabilized by interfacial accumulation of wax crystals; liquid oil structured into transparent oleogel; microstructure of surfactant-free O/W/O double emulsion; microstructure of oleofoam (air bubbles stabilized by interfacial accumulation of crystalline particles); and an oleofilm containing more than 98% wt liquid sunflower oil

- tolerant of the processing conditions and compatible with the product matrix;
- low-cost and easily available;
- the right functionality when used in intermediate products, such as cake shortening or baking margarine, and capable of mimicking a melt-in-the-mouth oral sensation (provided by fat crystals) when used in final products such as spreads, chocolate products, and so on.

In addition to meeting these conditions, the process used to create oleocolloids should be industrially feasible and not too energy intensive or harsh (to prevent deterioration of oil quality).

Academic research in the field has proliferated in the last few years. However, there is still a significant lack of knowledge in our understanding of solute-solvent interactions in hydrophobic mediums such as edible oils. Hence, there is an

urgent need to investigate the links between molecular features and parameters such as solubility, self-assembly (the process by which individual molecules form defined aggregates), and self-organization (the process by which the aggregates create higher-ordered structures) of different categories of solutes in liquid oil. This will help us unravel new materials (in processed or unprocessed form) that could be used to structure the oil phase. In addition, an in-depth study of co-assembly and self-sorting behavior of building blocks could unravel new synergistic combinations of gelling agents for bulk phase structuring of oleocolloids. Moreover, innovation in dispersion techniques could assist in the formulation of novel complex colloids, such as high internal phase emulsions, double emulsions (O/W/O), oleofoams, and oleofilms, which could be used to reformulate existing food products or to develop new product formats.

TABLE 1. List of ingredients that can be used for stabilization of oleocolloids

Ingredients	Functionalities/Properties	Type of oleocolloids stabilized
Natural waxes	<ul style="list-style-type: none"> • Crystalline network in the continuous phase • Pickering stabilization of interfaces 	Oleogels W/O emulsions
Fumed silica	<ul style="list-style-type: none"> • Network of agglomerated particles in the continuous phase 	Oleogels
Biopolymers (proteins and polysaccharides)	<ul style="list-style-type: none"> • Structuring of the water phase (providing texture) and stabilization of oil-water interface • Structuring of oil phase (through indirect routes) 	W/O emulsions (reduced fat) O/W/O emulsions Oleogels Oleofilms
Fatty acids/fatty alcohols	<ul style="list-style-type: none"> • Crystalline network in the continuous phase • Pickering stabilization of interfaces 	Oleogels Oleofoams
Sterols/sterol esters	<ul style="list-style-type: none"> • Structuring of oil phase through self-assembly 	Oleogels (mixed component)
Sucrose esters	<ul style="list-style-type: none"> • Stabilization of air-oil interfaces • Structuring of oil phase 	Oleofoams Oleogels (mixed component)
Lecithin	<ul style="list-style-type: none"> • Structuring of oil phase through self-assembly 	Oleogels (mixed component)
Cellulose derivatives	<ul style="list-style-type: none"> • Structuring of oil phase through direct dispersion (ethylcellulose) • Structuring of oil phase through indirect routes (hydroxyl propyl methylcellulose and methylcellulose) 	Oleogels

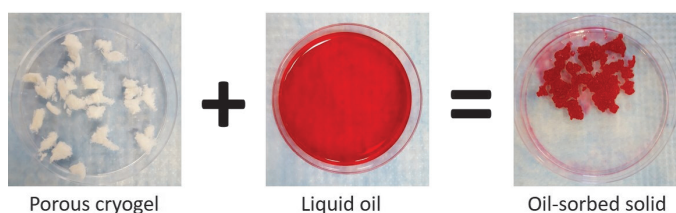


FIG. 2. Demonstration of excellent oil-absorbing property (≈ 100 fold) of hydrophilic food polymers pre-processed in form of porous cryogel through freeze drying. Sunflower oil with oil-soluble red dye was used for clear representation.

As most of these studies are mainly exploratory in nature, extensive developmental work in collaborations with industry will be required to commercially exploit some of these potential systems for food product formulation (see “Anticipated industry actions”). In addition to conducting developmental work (reworking/optimizing formulations and understanding the behavior of oleocolloids in product matrixes), industry could also play a major role in taking the initiative to gain regulatory approval for efficient structuring agents such as natural waxes, fumed silica, and so on. Further, a three-way collaboration of industry-academia-ingredient suppliers could be explored to improve the functionality of prospective ingredients that require pre-processing, such as freeze-drying hydrophilic food polymers into porous cryogels capable of absorbing large quantities of liquid oil (Fig. 2), or creating fat crystalline units coated with thin layers of hydrophilic cellulose derivatives using an emulsion-in-capsulation approach (Patel, A.R., *Materials Chemistry and Physics*, 195: 268–274, 2017).

A complete replacement of saturated fats in food products is practically not feasible at this stage, but some of the progress made in the field of oil structuring can help us with the partial replacement of saturated fats by formulating the

Anticipated industry actions (in collaboration with academia)

- Initiate regulatory approval of additives, such as natural waxes, silicon dioxide, and other additives.
- Rework/optimize formulations to suit the processing conditions (shear, temperature modulation) encountered in large-scale production.
- Understand the behavior of oleocolloids in food product matrixes.
- Explore innovative approaches in liaison with ingredient suppliers to improve the functionality of commonly used ingredients.

so-called hybrid systems. (As the name suggests, these systems are formulated by combining conventional fat hard stock with gelled oils.) At the same time, using innovation to transform processing techniques can help us develop new product formats with the potential to replace synthetic emulsifiers (with biopolymers, for example), achieve new product textures, and reduce saturated fat and calories.

Given the evolving and often conflicting nature of nutritional evidence, replacing saturated fats with monounsaturated fats may no longer be appealing, but oleocolloids could be designed to structure and stabilize oils rich in polyunsaturated fatty acids (PUFAs) that have clear health benefits over saturated fatty acids.

It is also important to keep in mind that reformulated products may lose some functionality and not be able to match the characteristics of conventionally formulated food products. Such loss of functionality may include a decreased plasticity of intermediate-use products (such as bakery fats) and less-desirable organoleptic properties of final products (such as texture and mouthfeel of spreads).

To summarize, the estimated benefits of replacing saturated fats with heart-healthy unsaturated fats (especially with PUFAs), replacing palm oil, and developing clean-labelled products with unconventional textures far outweighs the costs associated with innovating and reworking oleocolloids for better scalability.

Given the progress made in this area and the lack of feasible options to fulfil the unmet industrial needs, I feel it is an ideal time to address the multidisciplinary challenge of innovating in the area of edible oleocolloids.

Ashok R. Patel is currently a Marie Curie Fellow (Established Researcher) at International Iberian Nanotechnology Laboratory in Braga, Portugal. In addition to being an active researcher, Patel is also a private consultant and works directly with food and related industries in an advisory role. He can be contacted at ashok2510@gmail.com.

This article is an extended version of a recently published scientific viewpoint (Patel, A.R., *J. Agric. Food Chem.* 65: 3432–3433, 2017).

TD NMR Sample Tubes

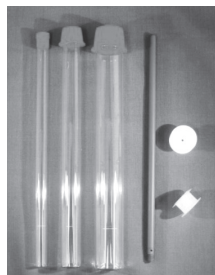
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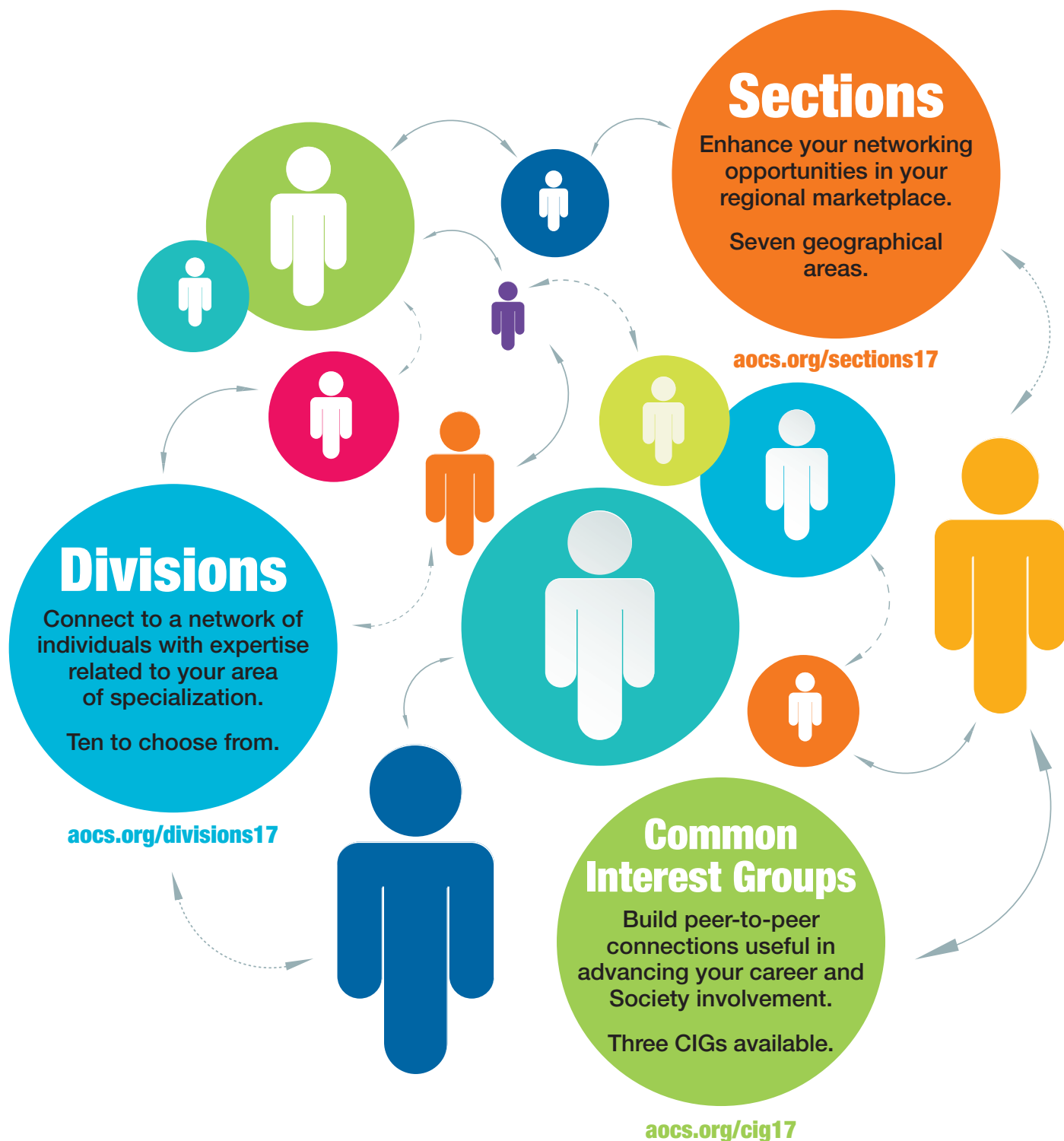
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Tailoring the digestion rate of omega-3's

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

Laura Cassidy

Incorporating omega-3 fatty acids into foods and beverages presents a challenge: The omega-3's must be protected from oxidative degradation during storage, but then released at the appropriate site within the human gastrointestinal tract (GIT) upon ingestion. Recently, researchers from Nanchang University, in China, and the University of Massachusetts, in Amherst, USA, encapsulated omega-3 fatty acids in nanoemulsions and microgels and studied their gastrointestinal fates using a simulated GIT (Chen, F., *et al.*, <http://dx.doi.org/10.1016/j.foodres.2017.07.039>, 2017).

Nanoemulsions, a simple and cost-effective way to control omega-3 stability and bioavailability, are tiny lipid droplets coated with an emulsifier and dispersed in an aqueous solution. However, in some applications, the encapsulated bioactive lipid may be degraded too rapidly during storage or released too early within the GIT. For tighter control of the bioactive lipid's stability and release, researchers have previously incorporated nanoemulsions into biopolymer microgels. To prepare microgels, researchers typically mix a nanoemulsion with a biopolymer solution (proteins and/or polysaccharides) to form a particle, and then alter the system conditions to promote polymer gelation. In previous studies, researchers have designed microgels that protect bioactive lipids during long-term storage and then release them in the mouth, stomach, small intestine, or colon. The site of release can be controlled by altering the composition and structure of the microgel to respond to differing conditions within the GIT, such as pH, ionic strength, temperature, or enzyme activity.

In a prior study led by Ze-Yuan Deng at Nanchang University and D. Julian McClements at the University of Massachusetts, researchers showed that the oxidative stability of flaxseed oil, a plant-based source of omega-3 fatty acids, could be greatly improved by encapsulating it in nanoemulsion-loaded alginate microgels (Chen, F., *et al.*, <https://doi.org/10.1016/j.foodhyd.2016.09.001>, 2017). They also found that the addition of caseinate, a protein from milk that acts as an antioxidant, to the microgels further improved their oxidative stability. In the new study, the researchers used a simu-

lated GIT to study the digestion and release of omega-3 fatty acids from these delivery systems.

The GIT model simulated the mouth, stomach, and small intestine of the human digestive tract. In this *in vitro* system, the mouth phase (pH 6.8) contained simulated saliva with 3% mucin, a protein in saliva that acts as a lubricant. The stomach phase (pH 2.5) contained simulated gastric fluid with 0.32% pepsin, a digestive enzyme produced in the stomach. The samples were swirled throughout incubation to mimic the churning action of the stomach. Finally, the small intestine phase (pH 7.0) contained simulated intestine fluid with bile salts (surfactants that aid digestion of lipids) and lipase (an enzyme that catalyzes lipid hydrolysis).

The team prepared four different delivery systems for the encapsulation of flaxseed oil: nanoemulsions (NE), nanoemulsions containing 2% caseinate (NE +C), hydrogel beads composed of alginate microgels (HB), and hydrogel beads containing 2% caseinate inside (HB + C). The nanoemulsions consisted of small lipid droplets (about 0.22 μm in diameter), whereas the hydrogel beads were visible with the naked eye (at least 100 μm in diameter). The researchers characterized the particle size, charge, and structure, before and during each stage of the simulated GIT.

Next, the researchers calculated the release of free fatty acids—a measure of lipid digestion—from the delivery systems during the small intestine phase of the GIT model. The small intestine is the site where the majority of omega-3 fatty acids are absorbed by the human body. The lipid droplets in

the nanemulsion systems were rapidly digested during the first 5 minutes of incubation. More than 76% of the free fatty acids were released from NE, whereas the addition of caseinate slightly slowed the release, with 65% of free fatty acids digested from the NE + C sample in 5 minutes. The researchers attributed the rapid release from the NE samples to the large surface area of lipids exposed to lipase in the simulated small intestine. The addition of caseinate may have slightly inhibited lipase activity, possibly by shielding the lipid molecules from the lipase. For both NE samples, about 96% of free fatty acids were released after 2 hours of digestion.

The initial release of free fatty acids was much slower when the nanoemulsions were loaded into microgels. For the HB system, only about 37% of free fatty acids were released during the first 5 minutes of digestion. Again, the caseinate appeared to inhibit lipid digestion, with 22% of free fatty acid released after 5 minutes from the HB + C system. In this case, the caseinate trapped within the microgel may have slowed the diffusion of lipase into the bead. The total amount of free fatty acids released after 2 hours was lower for the HB than the NE systems: 83% and 56% for the HB and HB + C systems, respectively.

“For nanoemulsions, the lipase molecules can readily adsorb to the lipid droplet surfaces and promote their digestion,” the researchers explain. “However, when the lipid droplets are trapped inside microgels, the lipase molecules have to move through the biopolymer network in the beads before

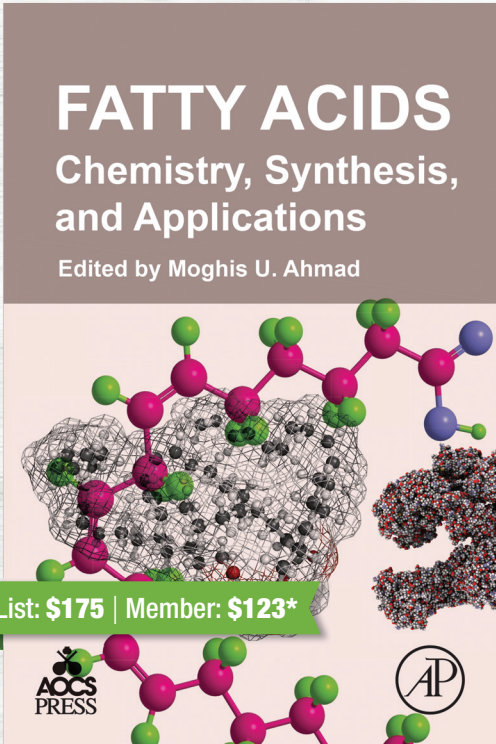
Information

Chen, F., *et al.* (2017) “Encapsulation of omega-3 fatty acids in nanoemulsions and microgels: impact of delivery system type and protein addition on gastrointestinal fate.” *Food. Res. Int.*, in press (available online July 17, 2017), <http://dx.doi.org/10.1016/j.foodres.2017.07.039>

Chen, F., *et al.* (2017) “Inhibition of lipid oxidation in nanoemulsions and filled microgels fortified with omega-3 fatty acids using casein as a natural antioxidant.” *Food Hydrocolloids* 63, 240–248. <https://doi.org/10.1016/j.foodhyd.2016.09.001>

they can reach the lipid droplet surfaces, which slows down digestion.” This research suggests that shelf-stable drug delivery systems for functional foods could be designed to control the digestion and release of omega-3 fatty acids within different regions of the GIT, with possible applications in controlling blood triglyceride levels, inducing satiety, or controlling the release rate of bioactive lipids, the researchers say.

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



Fatty Acids

Chemistry, Synthesis, and Applications

1st Edition

Edited by Moghis U. Ahmad

July 2017 | 600 pages

ISBN (softcover): 9780128095218 | ISBN (ebook): 9780128095447

Fatty Acids: Chemistry, Synthesis, and Applications is a comprehensive source of information about a wide range of industrially important fatty acids. Written by a team of experts, the 18-chapter book includes detailed descriptions of fatty acid crystallization, enzymatic synthesis, and microbial production. The book serves as a reference manual for the oleochemical industries and to a new generation of lipid scientists and researchers.

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TSCA “inventory reset” rule: reports due by February 7

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

Kelly Franklin

The US Environmental Protection Agency (EPA) published its final Toxic Substances Control Act (TSCA) “inventory reset” rule on August 11, starting the clock on the statutory 180-day reporting period.

The inventory notification rule will determine the substances active in commerce during the 10-year “lookback period” ending on June 21, 2016. Manufacturers and importers covered by the rule must report these by February 7, 2018. Processors—whose participation is voluntary— may report between now and October 5, 2018.

Legal experts agree that the final rule has been streamlined to be consistent with many of industry’s requests to adopt efficiencies in the process. Changes include broader reporting exemptions, decreased information to be included in notifications, and a longer reporting period for downstream users.

But despite these modifications—and although there is broad agreement that there are far fewer substances active in commerce than the 85,000 on the current TSCA inventory—the exercise promises to be a heavy lift.

REPORTING BASICS

The EPA will use this retrospective reporting to designate substances on the inventory as active or inactive. This, it says, will help inform the prioritization of chemicals for risk evaluation.

Once the list of designations is finalized, a user must submit a “forward-looking” notification prior to the manufacture, import, or process of any inactive substance.

The exercise also seeks to confirm the status of confidentiality claims by requiring manufacturers to indicate if they are seeking to maintain an existing chemical identity protection. The EPA is required to move to the public portion of the inventory any confidential substance for which it receives no requests to maintain the claim.

Reporters will have to provide chemical identity information and indicate if they seek to maintain an existing confidentiality claim. The agency removed the previously proposed



requirements to report commercial activity type and date range, as it determined these are “unnecessary to achieve the objective of designating substances as active or inactive.”

After the 180-day manufacturer reporting period, the EPA will publish a draft inventory “as soon as is practicable.” Processors may elect to report any substances that did not get notified by upstream suppliers prior to the agency finalizing the designations.

Processors may choose to forego reporting. But upon formal designation of substances, they must cease processing any inactive substance for nonexempt purposes until notifying the EPA.

Like other TSCA submissions, notifications will be made electronically through the EPA’s Central Data Exchange (CDX) and Chemical Information Submission System (CISS).

EXEMPTIONS

There are several reporting exemptions. These include:

- “excluded chemical substances,” including those not listed on the inventory, those manufactured solely under a TSCA section 5(h) exemption, and naturally occurring substances;

- Substances manufactured or processed for an exempt commercial purpose, including byproducts or impurities with no commercial purpose, substances manufactured in small quantities solely for research and development, or chemicals manufactured only for export or test marketing purposes; and
- Substances for which the EPA “already has equivalent notice.”

For this last exemption, the agency says there are three scenarios:

1. Substances on the interim active list, comprising those reported under the 2012 or 2016 chemical data reporting (CDR) rule. The list—published in June 2017—includes nearly 10,000 substances;
2. Chemicals added to the inventory during the lookback period pursuant to a notice of commencement (NOC); and
3. Substances for which a manufacturer can produce a CDX receipt demonstrating that another company has already reported a substance.

However, the EPA notes that if a manufacturer wishes to maintain an existing confidential business information (CBI) claim it will need to submit a notice to that effect, even if the substance is considered exempt from reporting.

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AOCS MEETING WATCH

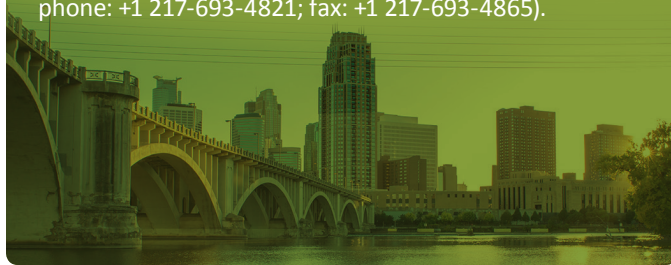
May 6–9, 2018. AOCS Annual Meeting & Expo, Minneapolis Convention Center, Minneapolis, Minnesota, USA.




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

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

May 5–8, 2019. AOCS Annual Meeting & Expo, Cervantes Convention Center at America's Center, St. Louis, Missouri, USA.


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Colombia: 2016 largest edible oil consumption in LATAM

Leslie Kleiner


Colombian consumers are changing their preferences from commodity oils to more innovative, higher-quality products. To understand the motivation behind this change, as well as new oil product launches, I consulted a recent report from global market intelligence agency Mintel entitled, “Oil innovations shift from quantity to quality in Colombia,” written by David Turner, Global Food and Drink Analyst. The following Q&A is extracted from this report.

Q: How would you describe the consumption of cooking and edible oil in Colombia, and how does it compare to that of other countries?

For the year 2016, data from Colombia’s national statistics bureau, Departamento Administrativo Nacional de Estadística (DANE) and the Economist Intelligence Unit, point out that on a per capita basis, Colombians consumed more oil than consumers of all other recorded Latin American countries. Compared to global markets, Colombian consumption of oil (10.83 kg per capita) was behind that of Spain and Malaysia (11.67 and 27.72 kg per capita, respectively). The reason behind such high consumption is the cuisine that comprises Colombia’s national dishes (arepas, empanadas, and others), which require frying and/or large quantities of cooking oil. Traditionally, soybean and sunflower oil are the most common oils used for these applications.

Q: Are Colombian consumers diversifying their interest from commodity oil to more innovative concepts?

Mintel research indicates a shift in focus from quantity to quality of edible oils. This can be seen by looking at 2015 versus 2016 volume and sales of edible oils, respectively. For example, in 2015, the value of the edible oil market increased by 13%, but volumes fell by 4%. In 2016, a similar behavior was observed, and this trend is expected to continue until the year 2020. This reflects a mature market in which raw materials are more expensive, but also reflects more complex purchasing behaviors. In recent years, Colombian consumers are purchasing less edible oil, but of higher quality.



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

Q: Is Colombia's economic outlook a concern for this shift in edible oil preferences?

Economic uncertainty due to weak international trade and adverse economic developments reduces consumers' optimism. For example, the International Monetary Fund recently downgraded its 2017 Gross Domestic Product (GDP) growth forecast for Colombia from 3% to 2.3%. Furthermore, 2017 tax reforms intended to make up for lost revenues on crude oil led to the raising of a value-added tax of 16% to 19%. This tax is applicable to basic items, including foods. With these pressures in mind, consumers' spending habits and confidence become affected. The lowest ever ranking of consumer confidence occurred before May 2017, forcing consumers to reconsider commodity versus specialty oils, or higher-quality products.

Q: Does Colombia's economic outlook prevent premium oil product launches?

Not necessarily, as many manufacturers have already launched premium oils, such as extra virgin avocado oil, organic virgin coconut oil, extra virgin olive oil, and others. Although it is true that manufacturers should be concerned about how to make these products more affordable for the near future, there is a large long-term potential for the Colombian market. For example, by the year 2020, the population of young individuals with upward mobility is expected to increase from 48 million (2016) to 50 million. These largely urbanized consumers who are exposed to multicultural cuisines will drive the demand on domestic and foreign flavors, which will translate to consumption of commodity as well as novel edible oils.

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



DID YOU KNOW?

Q: Chocolate is available in dark, milk, white, and what other natural color?

A: Ruby. The pinkish-hued chocolate, developed by the world's largest cacao processor, Barry Callebaut AG, has a natural berry flavor that's sour yet sweet. The unusual color comes from the powder extracted during the processing of beans from the Ivory Coast, Ecuador, and Brazil. No berries or colors are added.



Q: Who was the first woman to hold active membership in AOCS?

A: Rosalind U. Norris, chief chemist at Richard Hellmann Inc., whose founder created Hellmann's Mayonnaise. Norris had nine years of practical experience in food chemistry when she joined AOCS in 1927.

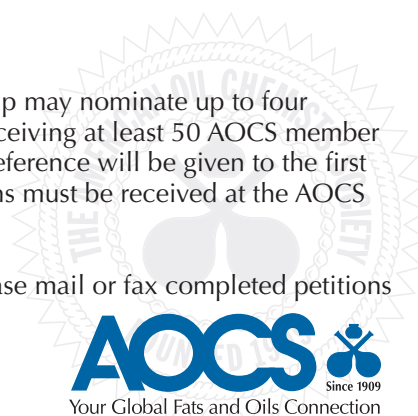


AOCS Board Petition to Nominate

For each annual election of AOCS Governing Board officers, the membership may nominate up to four additional member-at-large candidates by petition. Petitioned candidates receiving at least 50 AOCS member signatures will be added to the ballot approved by the Governing Board. Preference will be given to the first four petitioned candidates with at least 50 signatures. Petitioned nominations must be received at the AOCS Headquarters no later than **October 27, 2017**.

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



PATENTS

Additives for improving the resistance to wear and to lacquering of diesel or biodiesel fuels

Arondel, *et al.*, Total Marketing Services, US9587193, March 7, 2017

The present disclosure relates to novel anti-wear additives for diesel or biodiesel fuels having a sulfur content less than or equal to 500 ppm by mass. These novel additives will also improve the lacquering resistance of the higher-grade diesel or biodiesel fuels having a sulfur content less than or equal to 500 ppm by mass.

Compositions comprising omega-3 fatty acids and vitamin D for psoriasis, and methods and uses thereof

Fraser, *et al.*, Pronova Biopharma Norge AS, US9585896, March 7, 2017

The present disclosure relates to pharmaceutical compositions comprising omega-3 fatty acids and vitamin D for use in at least one of preventing and treating psoriasis, and to food supplement, dietary supplement, nutritional supplement, over-the-counter (OTC) supplement, medical food, or pharmaceutical grade supplement compositions comprising omega-3 fatty acids and vitamin D for use in improving at least one parameter associated with psoriasis.

Production of fatty acid alkyl esters by use of two lipolytic enzymes

Abo, *et al.*, Novozymes A/S, US9593352, March 14, 2017

A method for producing fatty acid alkyl esters, wherein a solution comprising triglyceride and alcohol is contacted with a first lipolytic enzyme having a relatively higher activity on free fatty acids than on triglyceride and a second lipolytic enzyme having a relatively higher activity on triglyceride than on free fatty acids.

Oil or fat composition

Homma, *et al.*, KOA Corp., US9591861, March 14, 2017

Provided is a fat or oil composition, comprising 50 mass% or more of diacylglycerols whose constituent fatty acids comprise 12 mass% or more of fatty acids having 20 or more carbon atoms and 5 mass% or less of a total content of eicosapentaenoic acid and docosahexaenoic acid, and having an iodine value of a fat or oil of 120 or less.

Process for the isolation of carotenoids

Deheri, *et al.*, ShayoNano Singapore Pte. Ltd., US9592499, March 14, 2017

Described herein is a material for reversibly binding to a carotenoid comprising a support coupled to silver ions in an amount to enable reversible binding with carotenoids, and wherein with the exception of silver ions, is substantially free of transition metals. Also described herein is a process for reversibly binding a carotenoid, the process comprising the steps of: providing a support coupled to silver ions in an amount to enable reversible binding with the carotenoid, wherein with the exception of silver, the support is substantially free of transition metals, contacting the support with the carotenoid under binding conditions to bind it thereto and dissociating the carotenoid from the support under dissociating conditions to release the carotenoid.

Process for producing a lubricant from an epoxy-triglyceride

Dalai, *et al.*, University of Saskatchewan, US9593287, March 14, 2017

A process for producing a lubricant from an epoxy-triglyceride comprising treating the epoxy-triglyceride with an esterifying agent in the presence of a heterogeneous catalyst under conditions to produce the lubricant is disclosed.

Cosmetic compositions comprising microalgal oil

Yaiser, TerraVia Holdings, Inc., US9597280, March 21, 2017

The invention provides cosmetic compositions comprising microalgal biomass, whole microalgal cells, and/or microalgal oil in combination with one or more other cosmetic ingredients, and methods of making such compositions. In preferred embodiments, the microalgal components of the cosmetic compositions are derived from microalgal cultures grown heterotrophically and which comprise at least 10% oil by dry weight.

Lubrication for drilling fluid

Massey, *et al.*, M J Research & Development, LP, US9598625, March 21, 2017

A drilling fluid lubricant includes a crude tall oil product and a derivative of ricinoleic acid that is at least partially transesterified product. A modified drilling fluid includes a water-based drilling fluid in a range of from about 95 vol.% to about 99 vol.% and a drilling fluid lubricant in a range of from about 0.1 vol.% to about 5 vol.% of the fluid. The drilling fluid lubricant includes a crude tall oil product selected from the group consisting of crude tall oil, distilled tall oil, acid tall oil, fractionated tall oil, and combinations thereof. The drilling fluid lubricant also includes at least partially transesterified derivative of ricinoleic acid product. The product comprises at least partially transesterified derivatives of ricinoleic acid that combined are in a range of from about 88 mole% to about 99 mole% of the product.

Method for the purification of lecithin

Jirjis, *et al.*, Cargill, Inc., US9605009, March 28, 2017

A method for the purification of lecithin, comprising the steps of: a. reducing the viscosity of lecithin to a viscosity of less than about 10 Pas; then b. mixing the lecithin with granulated active carbon; then c. separating the lecithin from the granulated active carbon and recover purified lecithin. Lecithin substantially free of poly-aromatic hydrocarbons, and a food or feed product comprising said lecithin.

Flexible solid-state conductors including polymer mixed with protein

Zhong, *et al.*, Empire Technology Development LLC, US9614250, April 4, 2017

Various embodiments of solid-state conductors containing solid polymer electrolytes, electronic devices incorporating the solid-state conductors, and associated methods of manufacturing are described herein. In one embodiment, a solid-state conductor includes poly(ethylene oxide) having molecules with a molecular weight of about 200 to about 8×10^6 gram/mol, and a soy protein product mixed with the poly(ethylene oxide), the soy protein product containing glycinin and β -conglycinin and having a fine-stranded network structure. Individual molecules of the poly(ethylene oxide) are entangled in the fine-stranded network structure of the soy protein product, and the poly(ethylene oxide) is at least 50% amorphous.

Omega-3 fatty acid nutraceutical composition and optimization method

Rosedale, US9610298, April 4, 2017

A novel omega-3 fatty acid/lipid based nutraceutical composition and a method of optimizing said omega-3 fatty acid/lipid based nutraceutical composition. The nutraceutical composition and method is based on the insight that different forms of high omega-3 fatty acid lipids (e.g., triglyceride form, ethyl ester form, free fatty acid form, phospholipid form) have different molecular modes and levels of action. Specifically, the phospholipid form is likely more effective at promoting membrane fluidity and permeability, while the free fatty acid form is likely more effective at regulating cell receptors, such as the PPAR α receptors, that are responsible for various metabolic effects including lipid metabolism. The desirability of producing omega-3 compositions that may act synergistically and thus more robustly to improve health and to some extent mimic markers of life extension such as shown by caloric restriction, along with specific optimization methods, markers, and compositions are taught.

Method to produce cake

Mastenbroek, *et al.*, DSM IP Assets B.V., US9615587, April 11, 2017

The invention relates to a novel use of a phospholipase A in the production of cake to improve at least one of the properties selected from the group consisting of: (i) batter viscosity, (ii) spe-

cific density, (iii) initial crumb softness, (iv) crumb pore homogeneity, (v) crumb pore diameter, (vi) crumb softness upon storage, (vii) shelf life, and/or (viii) cake volume. The invention also relates to a novel use of phospholipase A in the production of cake to enable reduction of the amount of eggs and/or fat used in the recipe.

Asphalt mixture, process for production of same, and paving method using same

Moriyasu, *et al.*, Maeda Road Construction Co., Ltd., US9617426, April 11, 2017

An asphalt mixture is provided which comprises an aggregate, an asphalt, a lubricative solidification material and an alkaline additive material that are mixed together. The lubricative solidification material contains a palmitic acid at a ratio of 1 to 15 wt%, a stearic acid at a ratio of 0.3 to 10 wt%, an oleic acid at a ratio of 39 to 59 wt%, a linoleic acid at a ratio of 20 to 48 wt%, and a linolenic acid at a ratio of 1 to 15 wt%.

Method of utilizing tall oil pitch

Saviainen, *et al.*, Forchem OYJ, US9624442, April 18, 2017

A method of treating tall oil pitch. In the present method, part of the fatty acids and the resin acids are released from their sterol esters and wood alcohol esters and converted into methanol esters or ethanol esters. The transesterified products are removed from the pitch by evaporation and then condensed, and by hydrogenating the generated condensate the acids are decarboxylated, and the double bonds are satisfied. It is possible to use the present method to produce automotive fuel from tall oil pitch.

Carotenoid oil suspension with high bioavailability and preparation method thereof

Xu, *et al.*, Zhejiang Medicine Co., Ltd. Xinchang Pharmaceutical Factory, US9622497, April 18, 2017

A carotenoid oil suspension and preparation method thereof are provided. The method includes the following steps: a) mixing carotenoid with organic solvent, heating the mixture to dissolve the carotenoid sufficiently to obtain carotenoid solution; b) introducing the carotenoid solution obtained in step a) into a vegetable oil solution stirred in high speed by spraying, meanwhile recovering the organic solvent generated during spraying under vacuum condition, then, simultaneously completing recycling and spraying, thereafter, obtaining carotenoid oil suspension; wherein, the carotenoid oil suspension comprises a carotenoid crystal with an average particle size of less than 5 mm. The method is applicable in industrial scale with continuous operation and increased efficiency without additional carotenoid crystal grinding processes, and decreases the degradation of carotenoid during the preparation process of carotenoid oil suspension.

Liquid-filled hard gel capsule pharmaceutical formulations

Chattaraj, *et al.*, Mylan Inc., US9622981, April 18, 2017

Embodiments of liquid-filled hard gel capsule pharmaceutical formulations comprise a non-emulsified mixture, wherein the non-emulsified mixture comprises about 0.1 to about 5% by weight of at least one active pharmaceutical ingredient, about 50 to about 95% by weight medium chain triglycerides, and about 5 to about 25% by weight medium chain mono/diglycerides, wherein the medium chain triglycerides and medium chain mono/diglycerides are present at a ratio by weight of from about 10:1 to about 5:1.

Convection recirculating fryer for cooking foods

Highnote, Pearl City Manufacturing, Inc., US9629502, April 25, 2017

A convection recirculating food product fryer with a fry tank with an inlet tube connected to a heat exchanger and an outlet connected to a magnetic pump with an outlet tube to the heat exchanger, the pump having a driving magnet assembly housing an impeller and a driven magnet, with a ceramic shaft extending through the impeller about which the impeller rotates when pumping oil; an electric motor magnetically coupled to the magnetic pump; a burner to heat the oil in the heat exchanger; a controller to control the ignition and running of the burner.

Production of soluble soy protein product ("S704")

Schweizer, *et al.*, Burcon Nutrascience (MB) Corp., US9629381, April 25, 2017

A soy protein product is obtained by extracting a soy protein source material with an aqueous calcium salt solution to form an aqueous soy protein solution and adjusting the pH of the mixture of aqueous soy protein solution and residual soy protein source to a pH of about 1.5 to about 4.4. The acidified soy protein solution then is separated from the residual soy protein source. The acidified soy protein solution may be dried, following optional concentration and diafiltration, to provide the soy protein product.

Guggulphospholipid methods and compositions

Ahmad, *et al.*, Jina Pharmaceuticals, Inc. US9637515, May 2, 2017

The present invention relates to the methods for preparing synthetic guggulphospholipids, their fatty acid analogues and other bioactive molecules. The present invention relates to E-guggulsterone and Z-guggulsterone or mixture of E- and Z-guggulsterones. The present invention also provides a novel method for the preparation of E-guggulsterol and Z-guggulsterol or mixture of E- and Z-guggulsterols from a mixture of E- and Z-guggulsterones. The present invention further relates to guggulphospholipids and other bioactive molecules incorporated

into complexes such as liposomes, complexes, emulsions, vesicles, micelles, and mixed micelles, which can include other active agents, such as hydrophobic or hydrophilic drugs for use, e.g., in treatment of human and animal diseases.

Eicosapentaenoic acid (EPA) formulations

Waibel, *et al.*, Qualitas Health, Ltd., US9629820, April 25, 2017

Provided herein are compositions comprising eicosapentaenoic acid (EPA) and polar lipids (e.g., glycolipids and phospholipids), and which do not contain any docosahexaenoic acid (DHA) or esterified fatty acids.

Pesticide and a method of controlling a wide variety of pests

Newberry, *et al.*, Gowan Comercio Internacional E Servicos Limitada, US9635858, May 2, 2017

A pesticidal composition including at least one substance derived from *Azadirachta indica* plant, at least one metal salt of a fatty acid and at least one excipient. The present disclosure also relates to a kit including the pesticidal composition. The present disclosure further relates to use of pesticidal composition/kit in controlling a wide variety of pests.

Refinement of oils using green tea extract antioxidants

Indrasena, DSM IP Assets B.V., US9637706, May 2, 2017

Disclosed are methods of improving the sensory and oxidative stability of oils (e.g., plant and animal oils) by combining an oil and an antioxidant composition comprising green tea extract and deodorizing the oil. Oils prepared by these methods are also disclosed.

Polymorphism and microstructure of certain triacylglycerols and fatty acid methyl esters

Narine, *et al.*, Trent University, US9637697, May 2, 2017

This application relates to the polymorphism and microstructure of certain triacylglycerols and fatty acid methyl esters, and how the properties of these individual components in a biodiesel fuel, as well as their combined mixtures, helps understand the fundamental mechanisms of their crystallization so as to design biodiesel fuels with improved low temperature characteristics.

Patent information was compiled by Scott Bloomer, a registered US patent agent and Director, Technical Services at AOCs. Contact him at scott.bloomer@aocs.org.





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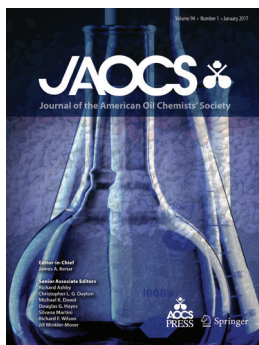
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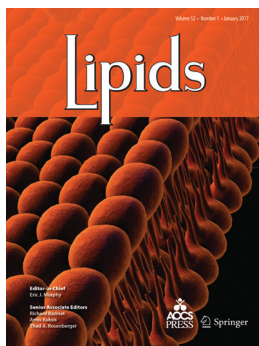
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Phytol promotes the formation of slow-twitch muscle fibers through PGC-1 α /miRNA but not mitochondria oxidation

Yang, K., *et al.*, *J. Agric. Food Chem.* 65: 5916–5925, 2017, <http://dx.doi.org/10.1021/acs.jafc.7b01048>.

Phytol is a side chain of chlorophyll belonging to the side-chain double terpenoid. When animals consume food rich in chlorophyll, phytol can be broken down to phytanic acid after digestion. It was reported that feeding animals with different varieties and levels of forage could significantly improve pH and marbling score of steer and lamb carcasses, but the internal mechanism for this is still not reported. The marbling score and pH of muscle was mainly determined by skeletal muscle fiber type, which is due to expression of different myosin heavy-chain (MHC) isoforms. Here, we provide evidence that phytol can indeed affect the diversity of muscle fiber types both *in vitro* and *in vivo* and demonstrate that phytol can increase the expression of MHC I ($p < 0.05$), likely by upgrading the expression of PPAR δ , PGC-1 α , and related miRNAs. This fiber-type transformation process may not be caused by activated mitochondrial metabolism but by the structural changes in muscle fiber types.

Lutein activates the transcription factor *Nrf2* in human retinal pigment epithelial cells

Frede, K., *et al.*, *J. Agric. Food Chem.* 65: 5944–5952, 2017, <http://dx.doi.org/10.1021/acs.jafc.7b01929>.

The degeneration of the retinal pigment epithelium caused by oxidative damage is a stage of development in age-related macular degeneration (AMD). The carotenoid lutein is a major macular pigment that may reduce the incidence and progression of AMD, but the underlying mechanism is currently not fully understood. Carotenoids are known to be direct antioxidants. However, carotenoids can also activate cellular pathways resulting in indirect antioxidant effects. Here, we investigate the influence of lutein on the activation of nuclear factor erythroid 2-related factor 2 (*Nrf2*) target genes in human retinal pigment epithelial cells (ARPE-19 cells) using lutein-loaded Tween40 micelles. The micelles were identified as a suitable delivery system since they were nontoxic in ARPE-19 cells up to 0.04% Tween40 and led to a cellular lutein accumulation of $62 \mu\text{M} \pm 14 \mu\text{M}$ after 24 h. Lutein significantly enhanced *Nrf2* translocation to the nucleus 1.5 ± 0.4 -fold compared to that

of unloaded micelles after 4 h. Furthermore, lutein treatment for 24 h significantly increased the transcripts of NAD(P)H:quinone oxidoreductase 1 (*NQO1*) by 1.7 ± 0.1 -fold, glutamate-cysteine ligase regulatory subunit (*GCLm*) by 1.4 ± 0.1 -fold, and heme oxygenase-1 (*HO-1*) by 1.8 ± 0.3 -fold. Moreover, we observed a significant enhancement of *NQO1* activity by 1.2 ± 0.1 -fold. Collectively, this study indicates that lutein not only serves as a direct antioxidant but also activates *Nrf2* in ARPE-19 cells.

Development of β -carotene-loaded, organogel-based nanoemulsion with improved *in vitro* and *in vivo* bioaccessibility

Fan, Y., *et al.*, *J. Agric. Food Chem.* 65: 6188–6194, 2017, <http://dx.doi.org/10.1021/acs.jafc.7b02125>.

β -Carotene (BC), a naturally occurring lipophilic carotenoid, is beneficial for human health. However, its water solubility and bioavailability are low. In this study, organogel-based nanoemulsion was successfully prepared to improve the loading amount, solubility, and bioavailability of BC. Corn oil was selected as the oil phase for the organogel as a result of the greatest release amount of BC. Tween 20 was optimized as the emulsifier based on the highest extent of lipolysis and BC bioaccessibility. The nanoemulsion was a better alternative than the organogel according to both the extent of lipolysis and BC bioaccessibility. Cellular uptake of BC was significantly improved through organogel-based nanoemulsion compared to BC suspension. Caveolae-/lipid-raft-mediated route was the main endocytosis pathway. Pharmacokinetic results confirmed that the *in vivo* bioavailability of BC in nanoemulsion was 11.5-fold higher than that of BC oil. The information obtained suggested that organogel-based nanoemulsion may be an effective encapsulation system for delivery of insoluble and indigestible bioactive compounds.

Synthesis, chemical characterization, and economical feasibility of *poly*-phenolic-branched-chain fatty acids

Ngo, H., *et al.*, *Eur. J. Lipid Sci. Technol.* 119: 1–12, 2017, <http://dx.doi.org/10.1002/ejlt.201600380>.

New *poly*-phenolic branched-chain fatty acid (*poly*-PBC-FA) products were synthesized from a combination of soybean fatty acids and phenolic materials through a highly efficient zeolite catalyzed arylation method. These *poly*-PBC-FAs are liquid at room temperature and do not have the unpleasant odor like the parent phenol reagent. They were found to be comprised of various numbers of phenol rings on the fatty acid chain, and this is important because compounds with a higher number of hydroxyl groups are expected to have better antimicrobial activities. Detailed characterization using gas chromatography and high performance liquid chromatography coupled to a quadrupole-time of flight mass spectrometer techniques were used to measure the number of phenol groups on the alkyl fatty acid chains. A cost process modeling technique was used to determine the economic feasibility of this arylation process to make these important *poly*-PBC-FA products.

The *E/Z* isomer ratio of lycopene in foods and effect of heating with edible oils and fats on isomerization of (all-*E*)-lycopene

Honda, M., *et al.*, *Eur. J. Lipid Sci. Technol.* 119: 1–9, 2017, <http://dx.doi.org/10.1002/ejlt.201600389>.

Since *Z*-isomers of lycopene are more bioavailable and show a higher antioxidant capacity than the (all-*E*)-isomer, it is important to investigate foods containing high amount of the *Z*-isomer and develop practically feasible method for *Z*-isomerization of (all-*E*)-lycopene. First, we investigated the *E/Z* isomer ratio of lycopene in raw and commercially available processed tomato products using an improved normal-phase HPLC method. The tomato products contained 4.6–33.4% of *Z*-isomers to the total lycopene, (*SZ*)-lycopene being the most abundant *Z*-isomer. The oil-containing products like tomato sauce and tomato soup suffered heat processing contained a higher percentage of *Z*-isomers of lycopene (27.4–33.4%). Subsequently, the impact of the amount and types of oils added on thermal *Z*-isomerization of (all-*E*)-lycopene contained in tomato puree was investigated. Increased addition of olive oil to tomato puree increased the production of lycopene *Z*-isomers upon heating at 120°C. (all-*E*)-Lycopene contained in tomato puree was converted to *Z*-isomers in the range of 39.2–50.7%, when 5% of vegetable oil (linseed, soybean, corn, sesame, rapeseed, rice bran, safflower seed, olive, sunflower seed, or coconut oils), or animal fat (beef tallow and pork lard) was added before heating at 120°C for 30 min. When sesame oil was employed, the total *Z*-isomerization ratio and (*SZ*)-lycopene content were significantly increased.

Lycopene inhibits metastasis of human liver adenocarcinoma SK-Hep-1 cells by downregulation of NADPH oxidase 4 protein expression

Jhou, B.-Y., *et al.*, *J. Agric. Food Chem.* 65: 6893–6903, 2017, <http://dx.doi.org/10.1021/acs.jafc.7b03036>.

NADPH oxidase 4 (NOX4), with the sole function to produce reactive oxygen species (ROS), can be a molecular target for disrupting cancer metastasis. Several studies have indicated that lycopene exhibited anti-metastatic actions *in vitro* and *in vivo*. However, the role of NOX4 in the anti-metastatic action of lycopene remains unknown. Herein, we first confirmed the anti-metastatic effect of lycopene (0.1–5 μ M) on human liver adenocarcinoma SK-Hep-1 cells. We showed that lycopene significantly inhibited NOX4 protein expression, with the strongest inhibition of $64.3 \pm 10.2\%$ ($P < 0.05$) at 2.5 μ M lycopene. Lycopene also significantly inhibited NOX4 mRNA expression, NOX activity, and intracellular ROS levels in SK-Hep-1 cells. We then determined the effects of lycopene on transforming growth factor β (TGF- β)-induced metastasis. We found that TGF- β (5 ng/mL) significantly increased migration, invasion, and adhesion activity, the intracellular ROS level, matrix metalloproteinase 9 (MMP-9) and MMP-2 activi-

ties, the level of NOX4 protein expression, and NOX activity. All these TGF- β -induced effects were antagonized by the incubation of SK-Hep-1 cells with lycopene (2.5 μ M). Using transient transfection of siRNA against NOX4, we found that the downregulation of NOX4 could mimic lycopene by inhibiting cell migration and the activities of MMP-9 and MMP-2 during the incubation with or without TGF- β on SK-Hep-1 cells. The results demonstrate that the downregulation of NOX4 plays a crucial role in the anti-metastatic action of lycopene in SK-Hep-1 cells.

Nonenzymatic β -carotene degradation in provitamin A-biofortified crop plants

Schaub, P., *et al.*, *J. Agric. Food Chem.* 65: 6588–6598, 2017, <http://dx.doi.org/10.1021/acs.jafc.7b01693>.

Provitamin A biofortification, the provision of provitamin A carotenoids through agriculture, is regarded as an effective and sustainable intervention to defeat vitamin A deficiency, representing a global health problem. This food-based intervention has been questioned in conjunction with negative outcomes for smokers and asbestos-exposed populations of the CARET and ATBC trials in which very high doses of β -carotene were supplemented. The current notion that β -carotene cleavage products (apocarotenoids) represented the harmful agents is the basis of the research presented here. We quantitatively analyzed numerous plant food items and concluded that neither the amounts of apocarotenoids nor β -carotene provided by plant tissues, be they conventional or provitamin A-biofortified, pose an increased risk. We also investigated β -carotene degradation pathways over time. This reveals a substantial nonenzymatic proportion of carotene decay and corroborates the quantitative relevance of highly oxidized β -carotene polymers that form in all plant tissues investigated.

Effects of medium- and long-chain triacylglycerols on lipid metabolism and gut microbiota composition in C57BL/6J mice

Zhou, S., *et al.*, *J. Agric. Food Chem.* 65: 6599–6607, 2017, <http://dx.doi.org/10.1021/acs.jafc.7b01803>.

Obesity is related to an increasing risk of chronic diseases. Medium- and long-chain triacylglycerols (MLCT) have been recognized as a promising choice to reduce body weight. In this study, three MLCT with different contents of medium-chain fatty acids (MCFA) (10–30%, w/w) were prepared, and their effects on lipid metabolism and fecal gut microbiota composition of C57BL/6J mice were systematically investigated. MLCT with 30% (w/w) MCFA showed the best performance in decreasing body weight gain as well as optimizing serum lipid parameters and liver triacylglycerol content. The expression levels of genes encoding enzymes for fatty acid degradation increased markedly and expression levels of genes encoding enzymes for *de novo* fatty acid biosynthesis decreased significantly in the liver of mice

treated with MLCT containing 30% (w/w) MCFA. Interestingly, the dietary intake of a high fat diet containing MLCT did significantly decrease the ratio of *Firmicutes* to *Bacteroidetes* and down-regulate the relative abundance of *Proteobacteria* that may attribute to weight loss. Furthermore, we found a notable increase in the total short-chain fatty acid (SCFA) content in feces of mice on a MLCT containing diet. All these results may be concomitantly responsible for the antiobesity effect of MLCT with relatively high contents of MCFA.

Industrial Applications

Dual-functionality ionic liquid mix for extraction and esterification of fatty acids as a step towards increasing the efficiency of conversion of waste cooking oils to biodiesel

Grimes, S.M. and P. Kewcharoenway, *J. Chem. Technol. Biotechnol.* 92: 2098–2105, 2017, <http://dx.doi.org/10.1002/jctb.5207>.

The increasing value of pure vegetable oils has made the use of waste cooking oils an attractive alternative feedstock for biodiesel production but the presence of free fatty acids in the waste significantly reduces efficiency of the conversion. A low-temperature mixed ionic liquid system with dual extraction and catalytic functions for the conversion of waste oils containing high levels of free fatty acids to biodiesel was successfully trialled. The solvent, a totally miscible system of HPyrBr and the Brønsted acid, SPyrHSO₄, allows the key properties of each ionic liquid, as extractant or catalyst, to work uninhibited by the presence of

the other. Extraction of free fatty acids from vegetable oils and their conversion to methyl esters in high yield is demonstrated for vegetable oils, containing single and mixed fatty acids, and for a model waste cooking oil. The oil separated from free fatty acids in the waste has a low acid value and can be converted efficiently to biodiesel; with the ionic liquid solvent recovered unchanged for reuse.

A mixed ionic liquid extraction-esterification solvent process has been developed to effect efficient conversion of free fatty acids in waste vegetable oils to biodiesel and their conversion to esters.

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

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

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

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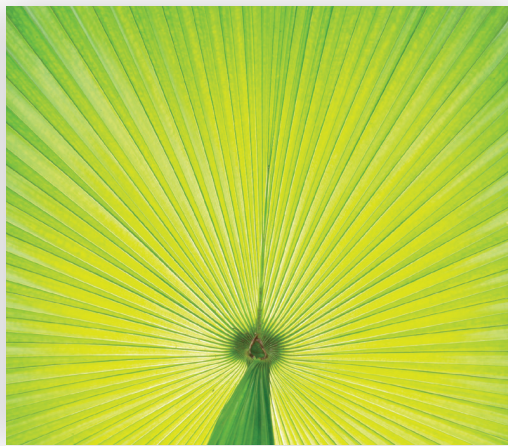
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