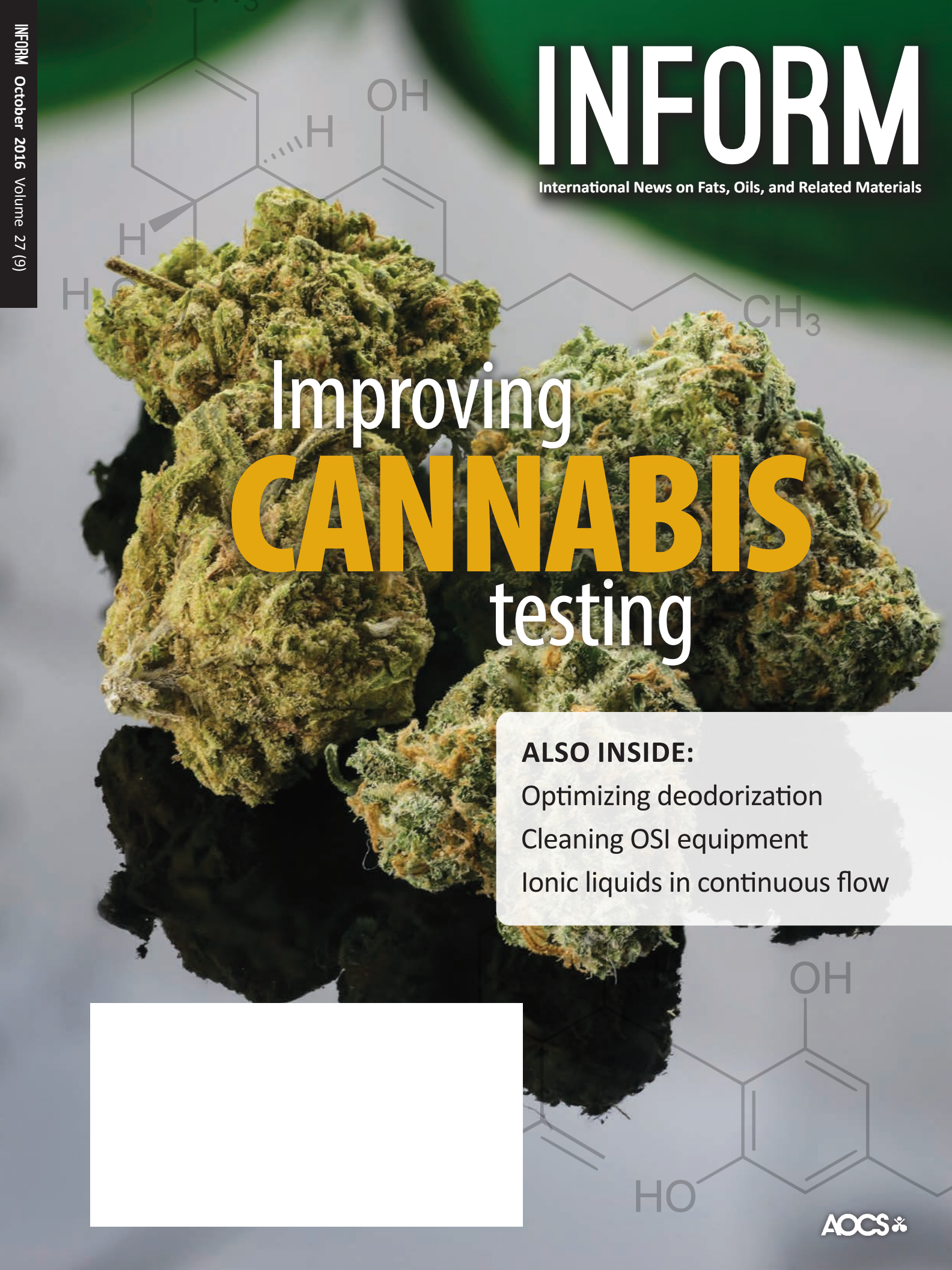
INFORM October 2016 Volume 27 (9)

# INFORM

International News on Fats, Oils, and Related Materials



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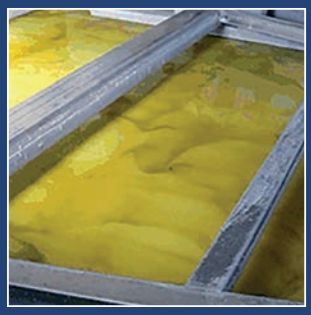

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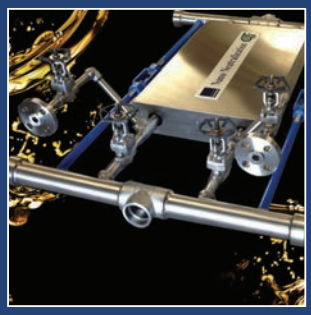

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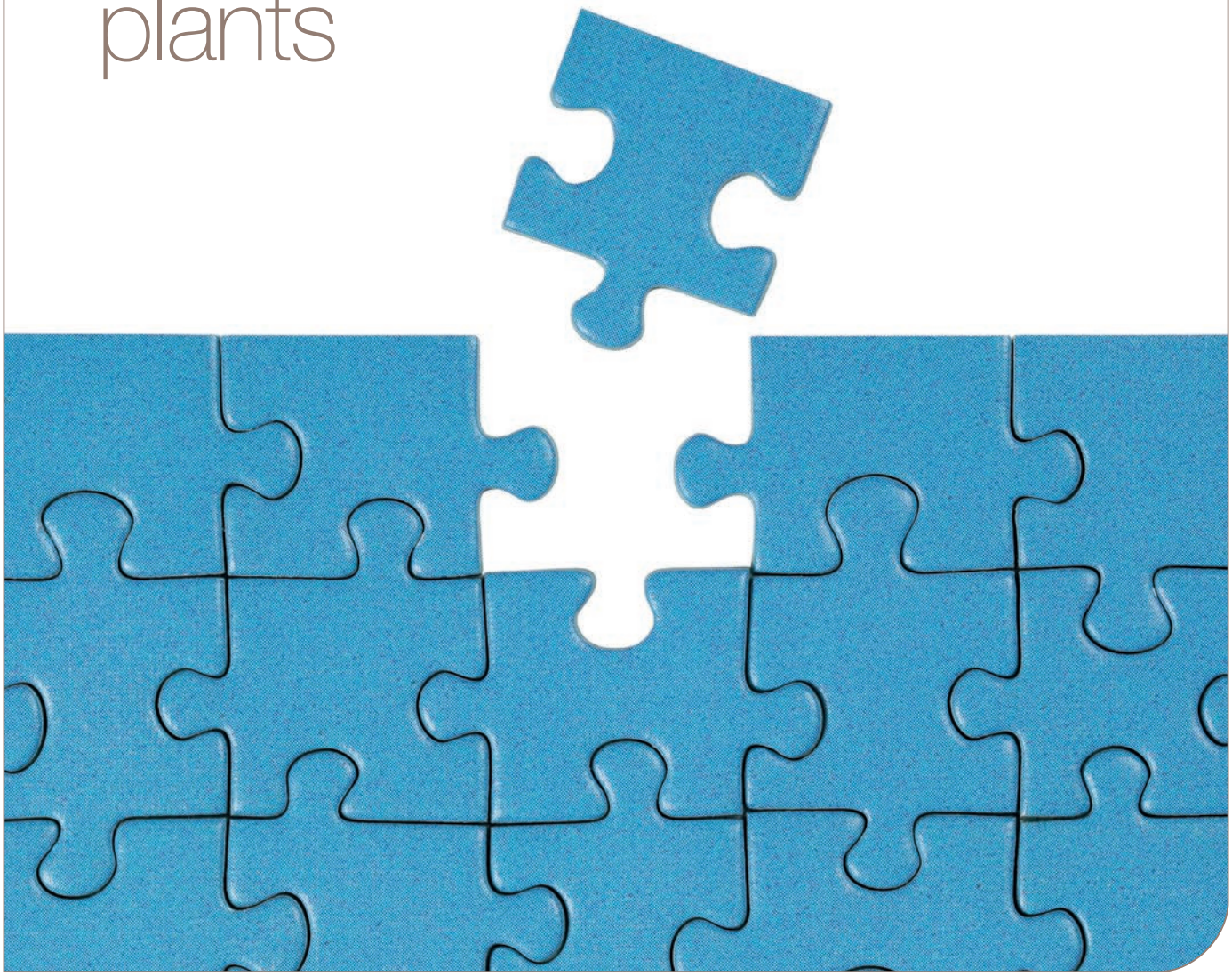

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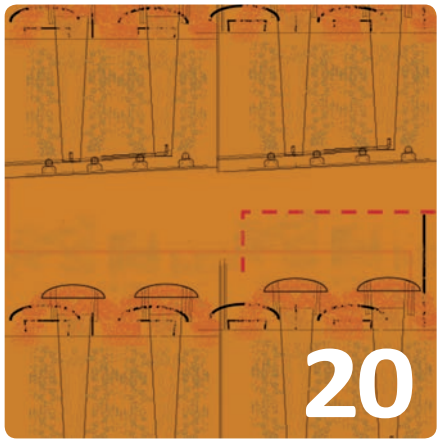





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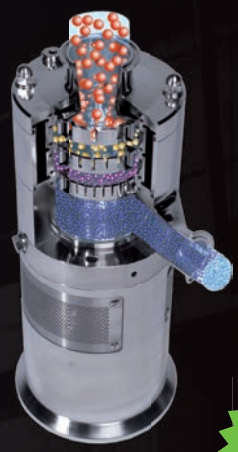

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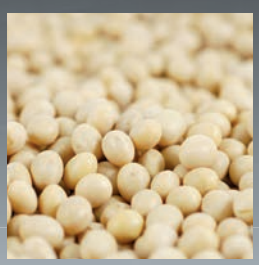

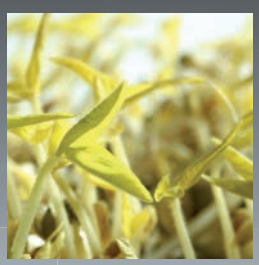

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# The Highs and Lows of Cannabis Testing

Laura Cassiday

Cannabis, also known as marijuana, is a flowering plant indigenous to Central and South Asia. The plant has been valued since ancient times for its psychoactive, medicinal, and fibrous properties; however, because of the potential for abuse, coupled with social and political factors, cannabis has been banned in most countries since the early 1900s. In the twenty-first century, this situation appears to be changing. Many countries, including Australia, Canada, North Korea, Colombia, Italy, Spain, and the Netherlands, have decriminalized cannabis possession and cultivation in one form or the other (adult recreational and/or medical use). Although cannabis remains illegal at the US federal level, 25 states plus the District of Columbia allow cannabis use for medical purposes, while four states (Colorado, Washington, Oregon, and Alaska) have legalized cannabis for both medical and adult recreational use.

- **With increasing legalization of both adult recreational and medical cannabis, there is a need for robust and reliable analytical testing to ensure consumer safety.**
- **Analytes of interest include cannabinoids, terpenes, residual solvents, pesticides, heavy metals, and microorganisms.**
- **As lipids, cannabinoids fall within the purview of AOCS. Therefore, AOCS is partnering with industry experts to help develop and validate methods for cannabis analysis and to increase the value of lab proficiency reports.**

Complete agreement has not been reached on cannabis' medical value or the ramifications of legal adult recreational use, but most would concur that cannabis products should be subjected to the same quality and safety tests as any other food or drug on the market. Therefore, testing labs have sprung up to help meet the quality, safety, and labeling requirements for legalized cannabis products in different jurisdictions. But with the legality and acceptance of cannabis use still murky in many locales, such labs have often operated on the fringes of lawfulness, without the benefit of widespread collaboration or guidance from established agencies such as the US Environmental Protection Agency (EPA), Food & Drug Administration (FDA), or Department of Agriculture (USDA) on how to develop and validate analytical methods specific to cannabis products. Despite a rocky start, the cannabis testing industry has matured rapidly in a relatively short period of time, and many competent, certified testing labs are now providing reliable quantitative data to producers and consumers. However, because most cannabis testing labs have developed their own proprietary methods, with little cross-validation among labs, many experts believe that there is a need for standardized analytical methods.

## CANNABIS COMPONENTS

Cannabis is a genus of flowering plant with compound serrated leaves. The most common species are *Cannabis sativa* and *Cannabis indica*. Through selective breeding, growers have developed strains with different sensory, psychoactive, and medicinal properties.

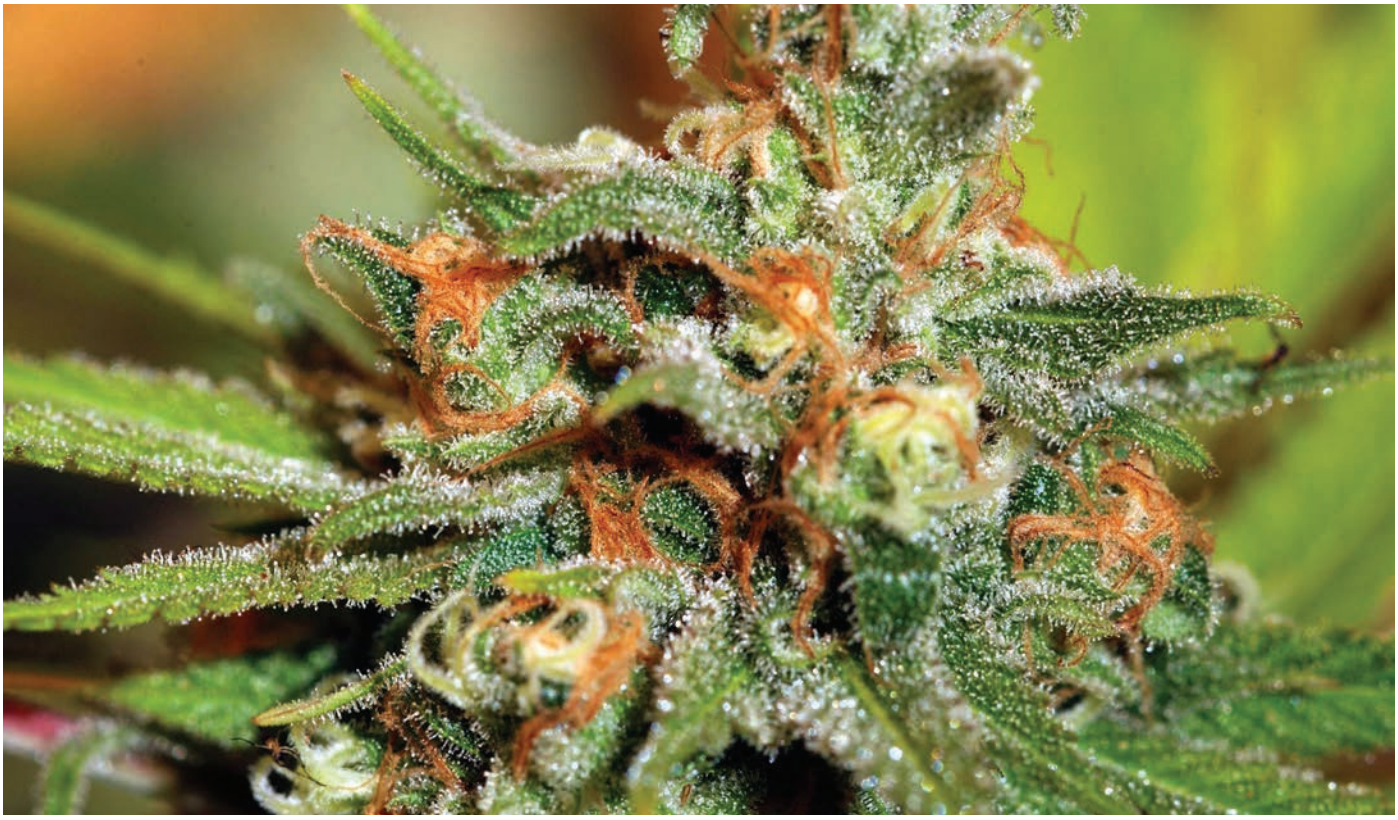

**FIG. 1. Glands called trichomes (tiny hairs) on the cannabis flower bud excrete a complex mixture of cannabinoids (including THCa and CBDa), terpenes, and other molecules.** Credit: GW Pharmaceuticals

Glands on the cannabis flower buds called trichomes excrete an oily substance containing cannabinoids, terpenes, triglycerides, and other compounds (Fig. 1). More than 480 compounds have been identified that are unique to cannabis, including over 70 cannabinoids (ElSohly, M. A., and Slade, D., http://dx.doi.org/10.1016/j.lfs.2005.09.011, 2005). Cannabis is smoked, cooked, or otherwise heated to produce the two most prevalent cannabinoids, tetrahydrocannabinol (THC) and cannabidiol (CBD), Fig. 2. In the plant, THC and CBD exist in their acid forms, THCa and CBDa. Heat decarboxylates the acid forms to produce THC and CBD. Other cannabinoids such as cannabinol (CBN), cannabigerol (CBG), cannabichromene (CBC), tetrahydrocannabivarion (THCV), and cannabidivarin (CBDv) are also being isolated and studied.

THC is the main psychoactive component of cannabis, whereas THCa (the native form in the plant) lacks psychoactive effects. CBD, which is non-psychoactive, is valued primarily for its medical effects, but CBD may also influence the psychoactive properties of THC. Cannabinoids produce their physiological effects by acting in distinct ways upon cannabinoid receptors, primarily in the brain and immune system. At this time, most evidence of cannabis' medical efficacy is anecdotal because limited clinical trials have been conducted, but proponents of medical cannabis claim that it can reduce nausea, seizures, inflammation, and pain, and can help treat ailments such as multiple sclerosis, epilepsy, glaucoma, Crohn's disease, and cancer.

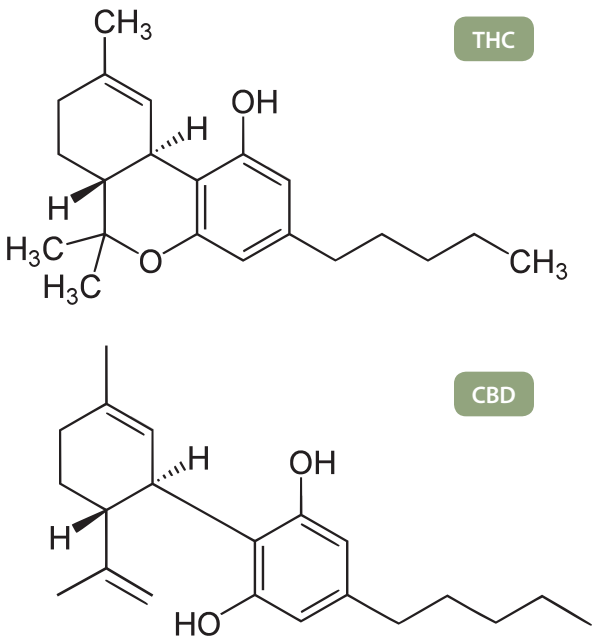


**FIG. 2. Structures of the cannabinoids THC and CBD.**
Credit: GW Pharmaceuticals

Growers of adult recreational cannabis often try to maximize THC content, as higher levels of THC demand higher prices. Today's THC levels, often 20% or more relative to the bulk plant material (w/w), are much higher than those in cannabis strains from the 1970s, which contained only 4–6% THC (Ruppel, T. D., Kuffel, N., http://tinyurl.com/PE-cannabis,

**TABLE 1. Recommended methods for cannabis analysis**

| Analyte | Examples | Significance | Recommended methods |
|---|---|---|---|
| Cannabinoids | THC*, THCa, CBD, CBDa, CBN | Potency testing; important for correct dosing of medical marijuana patients | HPLC-UV<br>GC-FID (cannot distinguish THC/THCa or CBD/CBDa without derivatization) |
| Terpenes | α -pinene, limonene, β -carophylene | Confer fragrances to cannabis and may influence medicinal properties | FET-HS-GC-FID<br>FET-HS-GC-MS<br>GC-FID<br>GC-MS |
| Residual solvents | butane, propane, isopropanol, acetone | Solvents left over from cannabinoid extraction; may be harmful | FET-HS-GC-FID<br>FET-HS-GC-MS |
| Pesticides | Organophosphates, pyrethroids, carbamates | Residual pesticides may be harmful, especially to young children or immunocompromised medical marijuana patients | HPLC-MS/MS<br>GC-MS/MS<br>GC-ECD (chlorinated) |
| Heavy metals | arsenic, mercury, lead, cadmium | Contamination from soil; high levels can be toxic | ICP-OES<br>ICP-MS |
| Microbial contamination | mold, mildew, bacteria, yeast, mycotoxins, aflatoxins | May be harmful, especially to immunocompromised medical marijuana patients | Plating assays<br>Films<br>qPCR |

*See text for abbreviations

2015). Levels of CBD are generally low in recreational strains (e.g., 2% w/w). In contrast, many medical cannabis strains contain higher levels of CBD (e.g., 14%) and lower levels of THC (e.g., 1%), and many strains target specific ratios of the compounds. For medical cannabis patients, the THC "high" may be unnecessary or undesirable, especially when treating children or chronic conditions that require medicating throughout the day.

Cannabis also contains approximately 140 terpenes (Ruppel, T. D., Kuffel, N., http://tinyurl.com/PE-cannabis, 2015). Terpenes, the basis of "essential oils," are molecules composed of multiple isoprene units and typically have pleasant fragrances. Examples include α-pinene (pine needles, rosemary), myrcene (clove-like, earthy, fruity), limonene (citrus), and linalool (floral). The particular terpene profile of a cannabis strain influences its flavor and fragrance. Different cannabis strains are named for their aromas, e.g., Super Lemon Haze, Grape Skunk, and Girl Scout Cookies. In addition to determining the sensory properties of cannabis, terpenes may enhance medical benefits through a process known as the "entourage effect."

## STATES' RIGHTS

Because the FDA still classifies cannabis as a Schedule 1 drug (having a high potential for abuse and no accepted medical use; this class also includes heroin, LSD, and ecstasy), the US federal government has mostly taken a "hands-off" approach to cannabis regulation, leaving these matters to the individual states that have legalized the substance. The exception is cannabis products that make medical claims, which are forbidden without prior FDA approval. Cannabis-based drugs that claim therapeutic effects must go through the same lengthy FDA approval process as other drugs, including clinical trials for safety and efficacy.

Although the FDA has not approved cannabis for any medical use, the agency has approved two drugs (Marinol and Syndros) that contain a synthetic form of THC (http://tinyurl.com/FDA-medical-marijuana). Both drugs were approved for the treatment of anorexia in AIDS patients and for nausea and vomiting associated with cancer chemotherapy in patients who did not respond to conventional treatments.

Cannabis testing requirements vary by state, but most states require testing and labeling for potency (THC and CBD) and various contaminants such as residual solvents, microbes, heavy metals, and pesticides. For example, in Colorado, all retail cannabis products must be tested for potency (THC, THCa, CBD, CBDa, and CBN), residual solvents (butane, hexane, heptane, and BTX), and microorganisms (*E. coli*, *Salmonella*, yeast, and mold) before hitting dispensary shelves. As yet, testing for heavy metals and pesticides is not mandatory, but the Colorado Marijuana Enforcement

Division has set limits for these contaminants that could be verified by random testing.

## POTENCY TESTS

The primary cannabinoids of interest for potency tests are THC, CBD, and CBN. A breakdown product of THC, CBN is an indicator of cannabis deterioration due to age or poor storage conditions. The two most common methods for potency analysis are high-performance liquid chromatography (HPLC) with UV detection and gas chromatography (GC) with flame ionization detection (FID) (Table 1). Although GC is more cost-effective and simpler than HPLC, this method requires sample derivatization to quantitate both the free and acid forms of THC and CBD. This is because the heat necessary for GC sample injection converts THCa into THC, and CBDa into CBD. Therefore, without deriviaization, the free and acid forms cannot be distinguished or quantified.

Derivatization methods are highly subject to error and difficult to validate, so many labs are choosing to invest in LC equipment. In a recent lab proficiency testing program, a survey of preferred potency testing methods found that 90% of the labs use LC (Emerald Test lab proficiency program, Emerald Scientific, San Luis Obispo, Calif., USA). However, GC without derivatization can provide a "quick and dirty" estimate of cannabinoid potency (THC + THCa, CBD + CBDa), which may be helpful for process monitoring.

In contrast, HPLC can separately quantify THC, THCa, CBD, and CBDa without derivatization, which is particularly useful for edible cannabis products because they will typically be consumed without additional heating. For edibles, GC might provide erroneously high potency values because the technique itself converts THCa into THC, and CBDa into CBD. Several companies, including Ceriliant (Round Rock, Tex., USA), Emerald Scientific, and Restek (Bellefonte, Penn., USA) offer cannabinoid standards to help identify and quantify peaks in GC and HPLC.

The desire to identify additional cannabinoids has led to wider use of two more sophisticated chromatographic techniques, Ultra Performance Chromatography (UPC) and Supercritical Fluid Chromatography (SFC). Compared to HPLC, UPC has the advantage of a higher separation efficiency, which results in better resolution, shorter analysis times, and reduced consumption of mobile phase (and therefore, less generation of hazardous waste). SFC has all of the advantages of UPC, combined with much easier sample preparation. SFC is very amenable to non-polar diluents, in which lipophilic cannabinoids are highly soluble. This attribute is particularly useful for isolating cannabinoids from the large variety of matrices available for cannabis-infused products.

Portable devices are also being used for potency tests. Fourier Transform infrared spectroscopy (FTIR) can provide quick and easy potency spot tests for THC, THCa, CBD, and CBDa in dried cannabis buds and processed oils. Although not as sensitive as chromatography, FTIR can analyze whole buds for potency, terpenes, and moisture content. Because the technique is not a primary method, standard samples are needed with known concentrations determined by other techniques, such as GC or HPLC.

## TERPENES

Terpenes present an analytical challenge because they are nonpolar and structurally similar, and many structural isomers exist. Mass spectrometry (MS) cannot distinguish terpenes that co-elute from a GC column because many have the same molecular weight and share fragment ions. While working at Restek, Amanda Rigdon (now chief technical officer at Emerald Scientific, San Luis Obispo, Calif., USA) and colleagues developed a method to analyze terpenes in cannabis using full evaporation technique (FET)-headspace (HS)-GC-FID (Rigdon, A, *et al.*, http://tinyurl.com/Restek-terpenes, 2014).

In FET-HS-GC-FID, a small amount of cannabis sample (20 mg or less) is placed in a 20-milliliter headspace vial and heated to volatilize terpenes in the sample into the gas phase, or headspace. The FET-HS technique is particularly useful for analyzing volatile components due to its ease of implementation and minimal sample processing. Once in the HS, the terpenes are injected onto the GC column where they are separated and then detected by FID. Less-volatile cannabinoids mostly remain in the sample, preventing them from overwhelming the less-abundant terpenes on the GC column. Using FET-HS-GC-FID, Rigdon and colleagues were able to profile 38 terpenes found in cannabis. However, the method is only semi-quantitative due to the relatively low volatility of some of the terpenes, as well as adsorption effects in solid matrices.

## RESIDUAL SOLVENTS

The extraction of cannabis to produce materials for use in oils, edibles, and other products often utilizes solvents such as butane, propane, isopropanol, or acetone. These solvents are harmful to health, so they should be absent from the final product. There is a trend in the industry to move away from these toxic solvents and employ supercritical carbon dioxide, ethanol, or water in extraction procedures. Because solvents are volatile, FET-HS-GC-FID can be used for both terpene and residual solvent analysis, says Rigdon. However, peak identification requires that the cannabis producer accurately reports which solvents were used in the extraction.

GC/MS can accurately identify peaks without prior knowledge of solvents; however, MS has a linear dynamic range that makes it difficult to analyze solvents that vary widely in concentration. “If you look at a lot of the state regulatory lists, the action level for butane is anywhere from 800 ppm to 5,000 ppm, whereas benzene is 1 to 2 ppm,” says Rigdon. “So if you were operating at a sensitivity high enough to see 1 ppm of benzene, you’re going to be overloading your detector with butane, because mass spectrometers overload much more quickly than FIDs.”

## PESTICIDES

It is illegal for cannabis growers to use pesticides and fungicides to control aphids, spider mites, and mold, which thrive in the warm, moist indoor conditions used to grow cannabis, unless such use is listed on the manufacturer’s label. Currently, there are no insecticides that list cannabis on the label, which puts some growers in a desperate situation. In trying to save their crop, they may choose to break the law and use compounds that are forbidden. State regulations on pesticides vary, but some agencies have defined pesticide-positive samples as those containing 0.1 ppm of any pesticide. There are thousands of known pesticides, so it is currently impossible to test for all. Oregon regulators have selected a 59-pesticide panel for the state’s testing requirements. “Even with 59 pesticides, it’s impossible for one chromatographic system to analyze them all effectively,” says Rigdon.

GC with an electron capture detector (ECD) can detect chlorinated pesticides at the parts-per-trillion (ppt) level, but the technique cannot detect non-chlorinated pesticides. GC in combination with tandem mass spectrometry (GC-MS/MS) can detect many pesticide classes. Although much more complex than GC-ECD, GC-MS/MS has an advantage for “dirty” samples such as edibles due to the selectivity of the triple quadrupole detector. HPLC-MS/MS can be used for many pesticide classes, as well, and is required for the analysis of heat-labile pesticides such as Abamectin.

“If I was going to buy one instrument for pesticides, it would be an HPLC-Triple Quad [MS/MS],” says Rigdon. “Ninety-five percent of the pesticides out there can be analyzed by HPLC-MS/MS, although there are some that you would need a GC-MS/MS for.”

For edibles, sample cleanup is essential prior to pesticide analysis by either type of MS/MS. A popular sample preparation method originally developed for analyzing pesticides in fruits and vegetables is QuEChERS (quick, ease, cheap, effective, rugged, and safe). QuEChERS can remove particulates, fats, and sugars in cannabis edibles that can foul chromatography columns or otherwise interfere with analyses. In QuEChERS, the edible sample is hydrated and homogenized with a tissue lyser or cryogenic grinder to produce very fine particles. Then, the sample is extracted with acetonitrile, and an extraction salt packet is added to cause partitioning. The resulting acetonitrile layer is then cleaned up using either dispersive solid phase extraction (dSPE) or cartridge SPE (cSPE). The cleaned-up sample can then be loaded onto an HPLC-MS/MS or GC-MS/MS instrument.

A couple of recent studies have exposed high levels of pesticides in cannabis products. As reported in a Spokane, Wash., USA, newspaper, *The Spokesman-Review*, Trace Analytics, a cannabis testing lab in Spokane, tested dozens of cannabis flowers and concentrates purchased from Washington dispensaries (http://tinyurl.com/TA-pesticides). The lab found many products with pesticides in the ppm range, well above proposed limits. (Like most states with legalized cannabis, Washington currently lacks official pesticide testing requirements.) OG Analytical, a cannabis testing lab in Eugene, Ore., USA, recently discovered that a “pesticide-free” plant wash used heavily in the cannabis industry actually contains an illegal pesticide not registered with the US Environmental Protection Agency (EPA). “It turned into a huge lawsuit,” says Rodger Voelker, lab director at OG Analytical. “That was sort of a wakeup call, and people started sending us lots of different products to test to make sure they don’t actually have pesticides in them.”

Voelker says that OG Analytical specializes in pesticide testing, but not every testing lab is set up to do the complex analyses. “Pesticides are by far the hardest analyses that are going to be done in the cannabis industry,” says Rigdon. “When it comes to screening, we’re getting pretty close, but actual quantitative testing is going to take a while. The food safety industry has had decades to develop their methodologies, and they’re still wrestling with pesticide testing in complex matrices.” She adds that a validated method for analyzing pesticides in a brownie is not going to work for pesticides in a gummy bear, soda, pasta or any of the other hundreds of matrices available for cannabis.

## HEAVY METALS

Heavy metals such as arsenic, mercury lead, cadmium, and chromium can enter cannabis plants from contaminated soil. These metals can be detected at trace amounts (ppt) by inductively coupled plasma (ICP)-MS or ICP-optical emission spectrometry (OES). Like techniques for pesticide analysis, methods for heavy metal analysis parallel those used by the food industry.

## MICROORGANISMS

During growth or storage, cannabis plants can become contaminated with microorganisms such as mold, mildew, bacteria, and yeast. Pathogenic bacteria such as *Escherichia coli* and *Salmonella*, as well as fungal toxins such as mycotoxins and aflatoxins, can cause severe illness, particularly in children or immunocompromised patients who are taking medical cannabis.

“Our micro testing is actually two different kinds of tests,” says Lucas Mason, co-founder and lead analyst at Aurum Labs, a cannabis testing lab in Durango, Colo., USA. “We test for pathogenic bacteria using qPCR [quantitative polymerase chain reaction], which is quick. Then we plate samples on standard media and get a total yeast and mold count after about three days.” Petri film techniques can also be used for microbiological analyses. “We started very old school, just standard plates, and that was really valuable for us,” says Mason. “We learned a lot about what grows on cannabis because there’s really not a lot out there in academia. We now have a library of about 30 common species that are growing across all of our clients, all of our regions.”

## CANNABIS FINGERPRINTING

As could be expected, pharmaceutical companies that market cannabis-based drugs typically conduct much more rigorous testing than producers of adult recreational cannabis products. GW Pharmaceuticals, a company based in Cambridge, UK, is developing a portfolio of cannabinoid-based medicines. One of these, Sativex, has been approved in the UK and 24 other countries (although not yet in the United States) for the treatment of multiple sclerosis-related muscle spasms. To

make Sativex, GW Pharmaceuticals combines extracts from two different cannabis strains, one high in THC and the other high in CBD, to yield a final THC:CBD ratio of 1. The company grows each cannabis strain under tightly controlled conditions (including no pesticides) in separate facilities (Fig. 3). Then, they make extracts from each strain and combine the extracts.

GW Pharmaceuticals wanted to perform quality control tests at multiple steps in the process, so they contracted a company called Infometrix to help with the analysis. Infometrix, based in Bothell, Wash., USA, develops chemometrics software tools and services for various industries. "Chemometrics is kind of like fingerprint analysis, only it's a fingerprint of a chemical system," says Brian Rohrback, president of Infometrix. "We've built custom quality control systems for a variety of applications, but they all have one thing in common: You've got complex data with a lot of correlations, and you have to remove those correlations to find out what's happening chemically."

GW Pharmaceutical's goal was to ensure batch-to-batch consistency for Sativex, a difficult feat for botanical extracts. So in collaboration with Infometrix, they divided the extracts from the two cannabis strains into four fractions each (cannabinoids, terpenes, sterols, and triglycerides), and analyzed the constituents in each fraction three times by HPLC or GC (24 analyses). Then, they combined the extracts and analyzed the cannabinoids and terpenes again three times (six analyses). "So the issue for us was, how do you combine these thirty analyses into red light/green light, pass/fail, do we sell this or not?" says Rohrback.

To develop their chemometrics system, Rohrback and his colleagues examined six years' worth of GW Pharmaceutical's quality control data. "We built ten PCA [principal components analysis] models, one for each of the four fractions in the two cannabis strains and for the two fractions in the mixture," says Rohrback. "When we analyze a new batch, we can compare it against the models and say whether, statistically significantly, the batch falls within the 95% confidence interval" (Fig. 4, page 14).

## GROWING PAINS

As more US states legalize cannabis, the industry continues to grow, and continues to feel bumps along the way. Many growers of cannabis had no previous experience in farming, so they made mistakes like using pesticides illegally or growing bumper crops of fungi. On the analytical side, people with liberal arts degrees or no college education joined the ranks of Ph.D. analytical chemists, sometimes with less-than-satisfactory results. "What I've observed is a lot of people who've gotten their education on what to do for quality control from the internet," says Rohrback. "They will go off and buy an analytical instrument on eBay and try to set it up, when they know nothing about the instrumentation or how to repair it."

Another challenge is representative sampling. Because cannabis is a valuable commodity, producers are often

**FIG. 3. At GW Pharmaceuticals, cannabis plants are grown indoors under tightly controlled conditions to minimize chemical variability.**
Credit: GW Pharmaceuticals

reluctant to provide more than a gram or two of sample for testing. Sometimes they will send only a single cannabis bud, which is hardly representative of, say, a 250-pound crop. "When there's no third-party sampling, some people will cherry pick," says Mason. "They'll find the needle in the haystack and try to send me their best-looking bud. As a result, their potency numbers will bounce around, and they think it's my fault." Because products with a higher THC content can demand a higher price, lab shopping is a problem, says Mason.

Sample tampering is also an issue, says Cynthia Ludwig, director of technical services at AOCS. Some growers will roll buds selected for potency testing in a concentrated form of cannabis extract known as kief, which is 30–50% cannabinoid by weight, to boost their THC values. Or they will try to influence the microbiological tests. "In my discussions with labs doing microbial testing, I've heard stories of people who figured out how to cheat the microbial test by putting their samples in the microwave," says Ludwig. "Clients will send two samples to the lab, supposedly from the same batch—except the potency sample is green and beautiful, and the micro sample is brown, dry and crispy."

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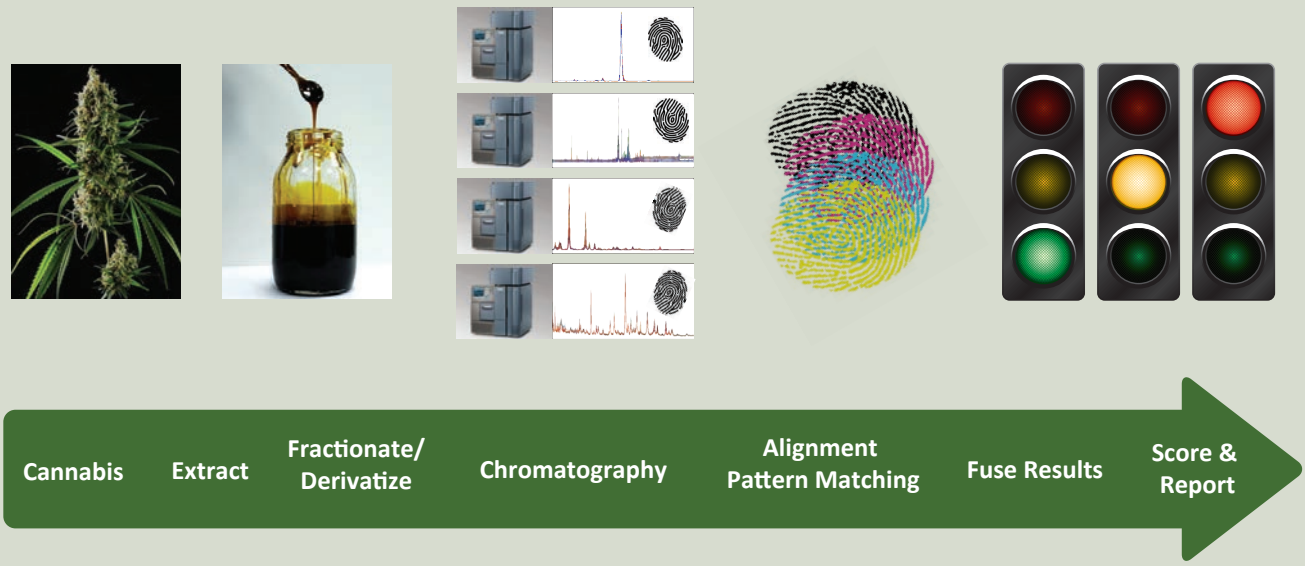
**FIG. 4. Quality control process for Sativex production. Cannabis plants are harvested and extracted with supercritical fluid carbon dioxide. The extract is divided into four fractions (cannabinoids, terpenes, sterols, and triglycerides), and then analyzed using HPLC or GC. The resulting chromatograms are aligned and compared to a database using principle components analysis. The results are fused into a single score indicating that the Sativex batch is appropriate for release (green light), failed (red light), or on the warning track (yellow light).** Credit: Brian Rohrback, Infometrix




## STANDARDIZED METHODS NEEDED

Currently, there are no standardized methods for cannabis analysis. As a result, each lab selects or develops its own methods to meet state testing requirements. According to Voelker, there is little collaboration among labs. "It's a complete crap shoot," he says. "Nobody shares anything, and everybody thinks they're the only ones doing a good job."

"A proprietary method is not a competitive advantage. It's a danger to medical cannabis patients," says Ludwig. "If everybody has their own proprietary method, then you don't know which one's correct, and it's hard to determine the correct THC dose for patients." To develop validated methods for cannabis analysis, AOCS has assembled a Cannabis Expert Panel of 75 top analytical scientists and cannabis industry professionals. The panel is helping to identify analytes of interest and the most accurate technologies for determining their levels.

"We currently have five methods that are in the midst of validation, and the hope is that they will be widely used by cannabis analytical labs," says Ludwig. These include two methods for cannabinoid analysis, a method for residual solvent analysis, one for heavy metals, and a sample preparation method. Ludwig expects validation data by the end of 2016, which will be followed by 3–6 months of collaborative studies. "We're hoping for the first methods to be available for use in Summer 2017," she says.

In addition, AOCS has partnered with Emerald Scientific, a supplier of cannabis testing products, to provide ISO 13528-compliant reports for the Emerald Test lab proficiency program. Interested labs receive samples that they test for potency and residual solvents, and they enter their data in an electronic portal. Then, AOCS analyzes the data using its established lab proficiency program. "The participants receive a report showing all of the participants' anonymous results, and they can see how they stack up against other analytical labs testing the same sample," says Ludwig. In addition to the raw data, the report includes the consensus mean and *z*-scores, as well as kernel density plots, so that participants can visualize where their lab falls within the group.

## THE MATURING OF AN INDUSTRY

In the past few years, the cannabis industry has matured from naïve exuberance to a more staid and reliable approach that craves legitimacy. "In the very beginning, there were a couple of labs where you paid one amount for the true potency value, and you paid another amount for something over 20%," says Mason. "So the precedent that the labs are shady and number factories was set pretty early on, and I think that sowed the seeds of distrust."

But this situation is changing, says Rigdon. "This industry is unique because everybody is so passionate and driven. They're trying their best," she says. "It's good to see them getting some wider recognition. I think that will bring them into the scientific community as a whole, which is where they need to be. They're not on the fringes anymore. They're a true analytical industry."

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

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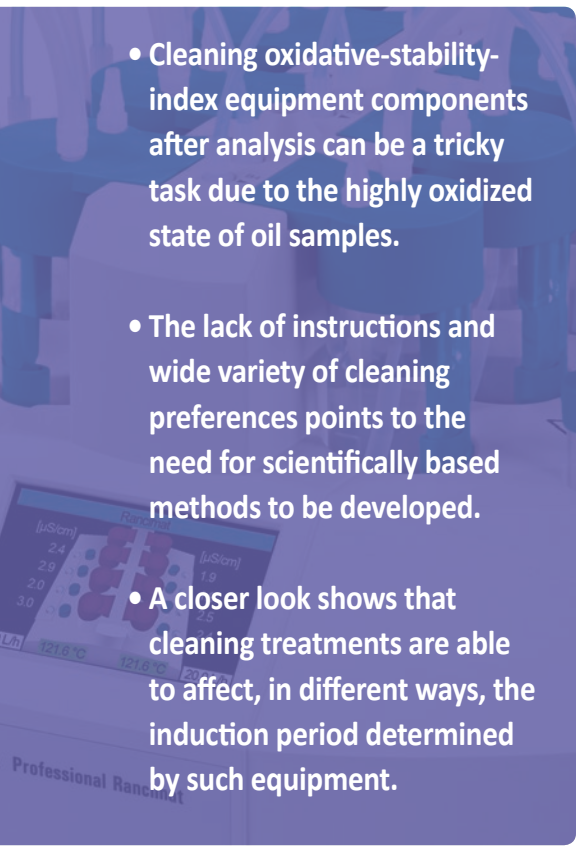
# Cleaning oxidative-stability-index equipment components

The oxidative stability of fats, oils, and biodiesel can be quickly determined under accelerated conditions by oxidative-stability-index equipment, such as Metrohm's Rancimat. Low-maintenance, rapid results, and ease of use are just a few of the elements that have contributed to establish Rancimat's widespread use. But, in contrast to the conveniences such equipment offers during analysis, cleaning the tubes and components can turn into a laborious task familiar to anyone who has worked with it. Rancimat can promote oil oxidation so effectively that it is difficult to know exactly how to ensure proper decontamination of its components. More importantly, this begs the question: Do glassware residues really affect oil stability results?

**Larissa Bueno-Borges and Marisa Regitano-d'Arce**



- **Cleaning oxidative-stability-index equipment components after analysis can be a tricky task due to the highly oxidized state of oil samples.**
- **The lack of instructions and wide variety of cleaning preferences points to the need for scientifically based methods to be developed.**
- **A closer look shows that cleaning treatments are able to affect, in different ways, the induction period determined by such equipment.**

Rancimat analysis has many applications, from the characterization of fat samples to the comparison of antioxidant action when two or more additives are tested. Rancimat can also be used as a preparative method to intentionally induce oxidation reactions and decomposition of certain thermally labile compounds. The principles involved in the Rancimat assay are based on the increase of conductivity in the water vessel caused by the collection of condensed, volatile, low-molecular-weight acids generated in oil-oxidation reactions [1]. This decomposition of the oil is provoked by the constant heating and presence of the air blown directly into the test sample. The analysis terminates when the equipment detects an exponential increase in the conductivity measurements, thus determining the induction period (IP) of the sample. A sample's IP can be interpreted as its resistance toward oxidation, expressed in units of time, under the conditions specified by the method. Consequently, the obtained IP of a given oil is not a fixed property. Its determination is sensitive to specific variations in oil composition (such as the presence of antioxidant compounds) and equipment parameters set at each analysis [2]. It is reasonable to assume that oxidation products generated during analysis could influence further assays if those oxidation products remain in the glassware.

Removing greasy residues is usually laborious, but the highly oxidized state of oil samples after Rancimat analysis aggravates this situation even more, along with such complicating factors as:

- increased viscosity and degree of polymerization of lipid samples;
- divergent characteristics of the many products generated by oxidation reactions; and
- particularities of the materials composing Rancimat's pieces, including irregular surfaces, different porosities, and areas with limited access (silicon hose, for example).

To ensure reliable results, some authors have proposed cleaning procedures involving painstaking steps for glassware treatment after analysis. These methods most commonly involve immersion of the components in strong alkaline solutions with ethanol and isopropanol [3,4]. The absence of instructions or data regarding the effects of such contamination on measurements calls for the development of scientifically based cleaning protocols designed specifically for Rancimat analysis—particularly as with any laboratory routine, a single piece of equipment is probably operated by several analysts, each employing his or her own cleaning preferences, sometimes arbitrarily.


Courtesy of Metrohm

## A STUDY OF THE EFFECTS OF CLEANING METHODS

As members of the Fats and Oils Laboratory at the University of São Paulo, Brazil, we use the same Rancimat equipment on a daily basis. This research started with the observation of discrepancies in cleaning methods and subsequent concern that these discrepancies would lead to erroneous results. This concern grew into experiments, which then became part of a still ongoing investigation.

To date, we have assessed the short-term effect of various post-assay procedures, applied to the components of Rancimat, on the measured IPs. A series of oxidative stability determinations was performed, assigning cleaning treatments (Fig. 1) to groups of analyzing units (the set of pieces that, once assembled, are capable of performing the analysis of one sample). The goal was to evaluate variations and trends in measured IPs compared to a control. The same vegetable oil was used for all determinations, and only new glassware was used. Since the subjects of research were the above-mentioned components, all efforts were focused on isolating these from external sources of contamination. These efforts included washing and storing each treatment group separately, and ensuring that all brushes and sponges used during scrubbing were exclusive for each treatment group.

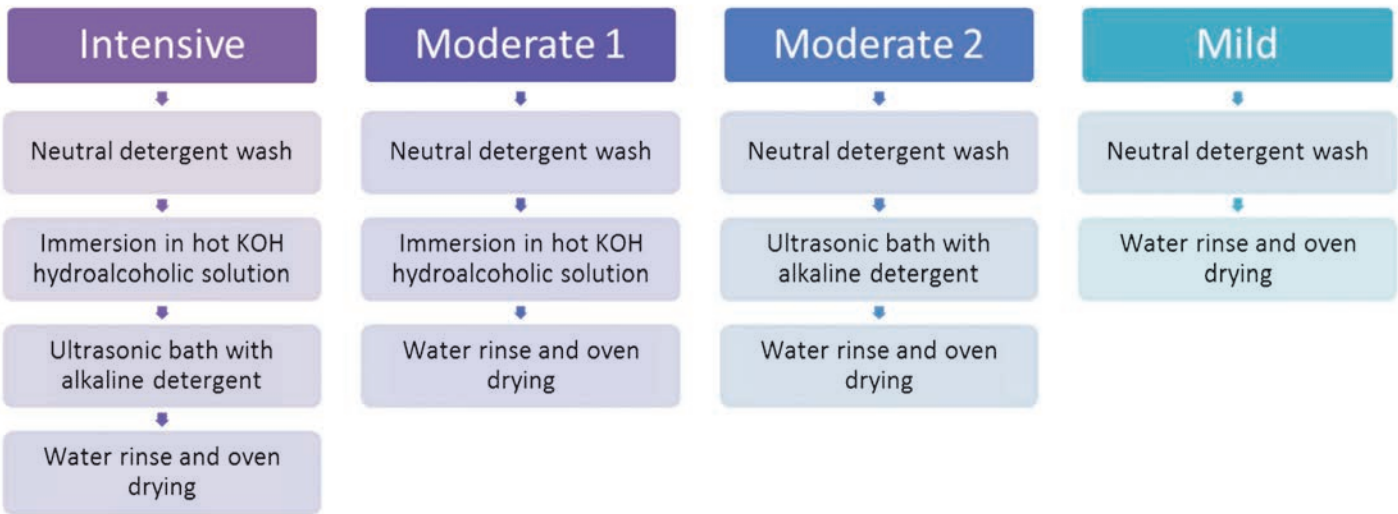



FIG. 1. Flowchart of treatment groups and proposed cleaning steps

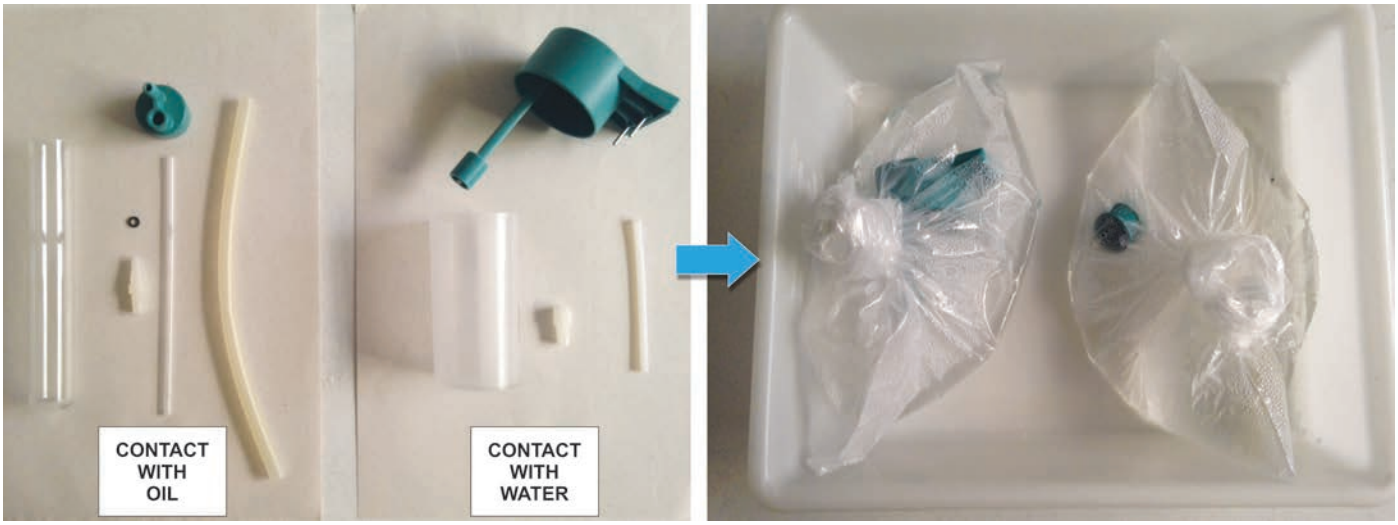
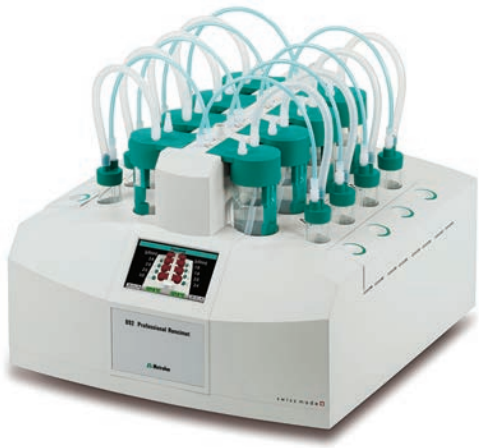



FIG. 2. Separate washing of components

After analysis but prior to the neutral detergent wash, all tubes and vessels were completely emptied. This stage consisted of thorough scrubbing of the parts with neutral detergent (common liquid dishwashing soap) and tap water, repeating when necessary until the greasy residue was completely removed. To prevent ethanol evaporation during heating of the KOH hydroalcoholic solution, water was preheated and added to a 6% KOH ethanolic solution at a proportion of 1:2. Pieces remained immersed for 4 hours and were mixed twice at every hour. For the ultrasonic bath, an alkaline detergent, specifically designated for laboratory glassware, was used to compose a 3% solution.



In order to limit the exposure of Rancimat's components to oxidized oil, pieces that remain in contact with the sample during analysis were washed separately (Fig. 2).

Our preliminary findings revealed unexpected outcomes. Instead of observing trends in IPs due to the accumulation of lipid-derived products, washing treatments greatly influenced results due to the action of cleaning agents. Such residues remained in the glassware even after thorough rinsing with tap water, followed by immersion with distilled water. Groups in which the KOH hydroalcoholic solution was used displayed greater IPs than the control (up to +3.5 hrs.), whereas the moderate 2 group cleaned with alkaline detergent showed considerably decreased IPs (up to –1.5 hrs. compared with the control). Results throughout 20 analysis/washing cycles were highly variable—except in the mild group, which is what would be expected from the influence of cleaning agents, since the removal of cleaning agents can easily be irregular. Although the mild treatment was believed to be ineffective, actual results did not vary significantly from those of the control, indicating that for the studied period a well-executed wash with neutral detergent was sufficient to ensure consistent results.

## FIRST CONTRIBUTIONS AND LONG-TERM INVESTIGATION

The presence of substances capable of under/overestimating results focuses attention on the importance of each wash after using the equipment. It is not uncommon for researchers to incorporate key analyses into a single batch, consequently sentencing all replicates to reproduce erroneous results. It became clear during our investigations that experimental errors in OSI determinations reside largely in the glassware cleaning stage.

Extensive experiments are being conducted to verify the overall effects of cleaning procedures during long-term use, with emphasis on both the IPs and the accumulation of oxidation products on glassware surfaces. At this point no treatment can be recommended with certainty, but a few conclusions were drawn based on our observations:

- Proper washing with a high-quality neutral detergent and scrubbing with brushes was essential to ensure visibly clean glassware. If this step was not executed well, further use of cleaning agents had little effect on grease removal.
- Every effort should be made to remove residues from cleaning agents, including thoroughly washing components with tap water and immersing them in distilled water.
- Washing components that are in direct contact with oil separately from those in direct contact with water is recommended.

Using standardized methods can generate great economy with respect to hydric resources, reagents, and working hours,

and this applies to glassware cleaning procedures as well. The elucidation of the effects of cleaning methods on Rancimat analysis may not only benefit researchers in the constant quest for reliable results, but may also increase the efficiency of laboratory routines by eliminating unnecessary decontamination steps.

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# Efficient optimization of deodorization through process simulation studies

Marcello Usseglio

- **Deodorization is an important economic factor in today's vegetable oil refinery.**
- **Small increases in yield, quality enhancements in outlet streams, and reductions in energy costs (steam) associated with deodorization can result in significant overall gains in productivity and revenues.**
- **In process optimization tasks, process modeling and simulation studies—in conjunction with lab results of key samples and actual process conditions—widen the scope of optimization activities, making it easier to select the best processing scenario while reducing the time, cost, and degree of uncertainty of industrial plant tests.**

The physical refining and deodorization of vegetable oils are stripping processes in which free fatty acids (FFAs) and other minor volatile compounds are stripped from vegetable oil by using stripping steam under suitable processing conditions—very low absolute pressure (vacuum) and temperature—to preserve the quality characteristics of the processed oil.

Usually, two types of deodorization columns are used: 1. columns with trays containing steam lift pumps, bubbling pipes, and other internals, in which stripping steam and vegetable oil come into contact in a cross-flow way and 2. columns with structured packing, in which stripping steam and vegetable oil come into contact at countercurrent. Both stripping column designs attempt to provide the best contact between the vapor phase and the liquid phase by creating a large contact surface, together with an optimal stripping steam distribution (Fig. 1).

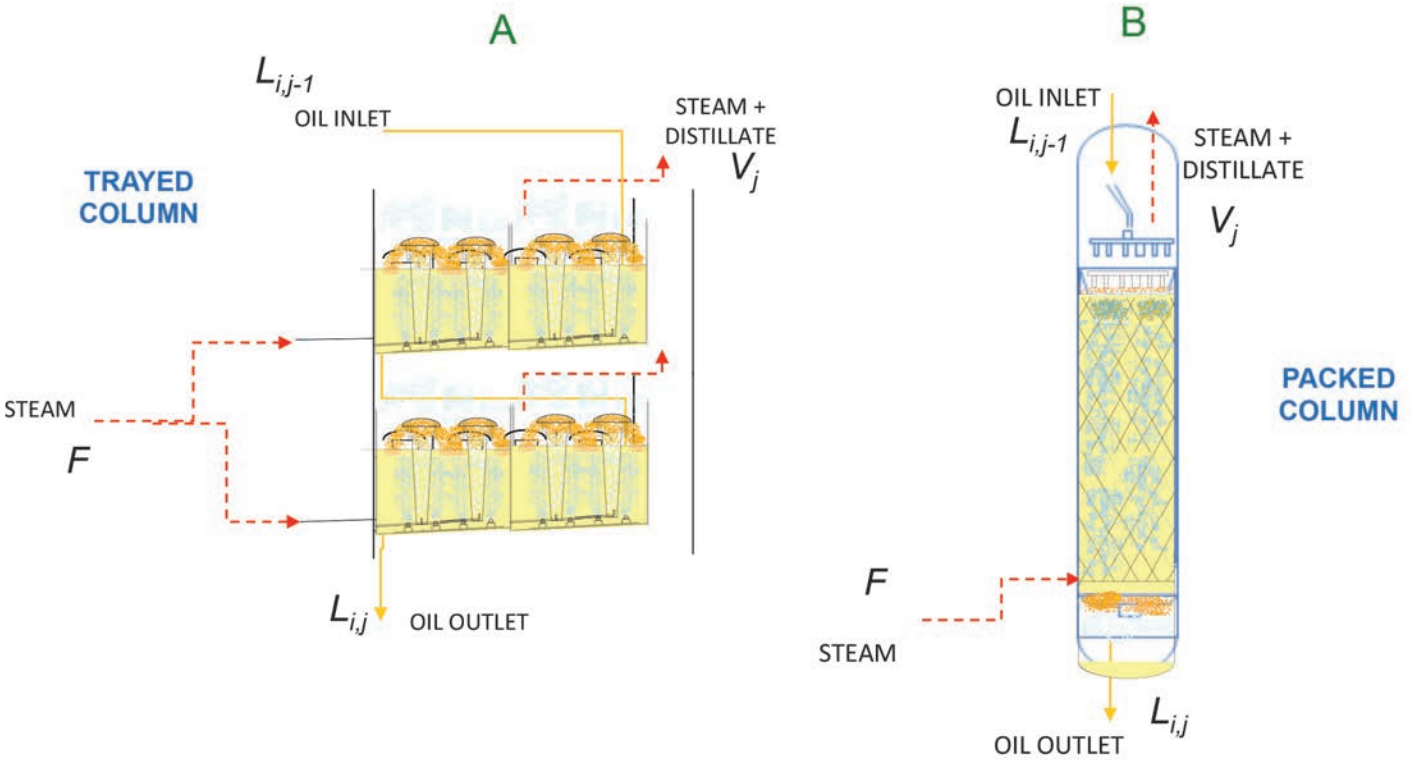
So the first question that immediately arises is: Do all deodorizers have the same performance? And, if not, how do we compare their performance, in terms of how much stripping steam we need, volatile compounds concentration in final oil, in terms of oil yield?

## THEORETICAL CONCEPTS INVOLVED IN DEODORIZATION/PHYSICAL REFINING

To answer these questions, it is important to understand all of the phenomena involved in the deodorization process. Let us talk briefly about some theoretical concepts.

As we know, vegetable oils are complex mixtures with respect to the compounds involved (triacylglycerols, monocylglycerols, diacylglycerols, free fatty acids, tocopherols, tocotrienols, sterols) and their physicochemical characteristics.

FIG. 1: A. Trayed column (cross flow), B. structured packed column (countercurrent flow)




In a stripping process—regardless of whether it is in a steam lift pump tray column, a structured packing column, or a combination of both—there are five key aspects to consider.

1. thermodynamic (liquid-vapor phase equilibrium)
2. mass transfer (compounds transport)
3. hydraulic behavior (vapor phase pressure drop)
4. side reactions (hydrolysis of acylglycerols, thermal degradation of tocopherols/tocotrienols, cis-trans isomerization of non-saturated carbon chains)
5. liquid/vapor entrainment (carryover of oil droplets to the vapor stream)

If we focus on mass-transfer phenomena, both types of equipment include liquid-vapor contact. This is the so-called interphase in which the mass transfer (traffic of compounds) and energy transfer take place.

In the case of columns with structured packing, this mass transfer occurs between the oil layer wetting the packing surface and the steam stream rising through the column. In columns with trays, the mass transfer occurs between the steam bubbles (created either from the bubbling at the bottom or from the steam injected inside the lift pump) and the oil circulating along each tray.

A compound mass balance applying to any type of column can be calculated for a given compound *i* (fatty acid, monoacylglycerol, tocopherol, and so on) by using the following equations.

Mass balance compound *i* in vapor phase

$$(1 + \emptyset_j^V)V_j y_{i,j} - F_j^V y_{i,f} + N_{i,j}^V = 0$$

Mass balance compound *i* in liquid phase

$$(1 + \emptyset_j^L)L_j x_{i,j} - L_{j-1} x_{i,j-1} - N_{i,j}^L = 0$$

Both diffusion and convection contribute to the mass-transfer rate:

Vapor phase

$$N_{i,j}^V = a_j^I c_t^V [k^V](\overline{y^V - y^I}) + y_{i,j} N_{t,j}$$

Liquid phase

$$N_{i,j}^L = a_j^I c_t^L [k^L](\overline{x^I - x^L}) + x_{i,j} N_{t,j}$$

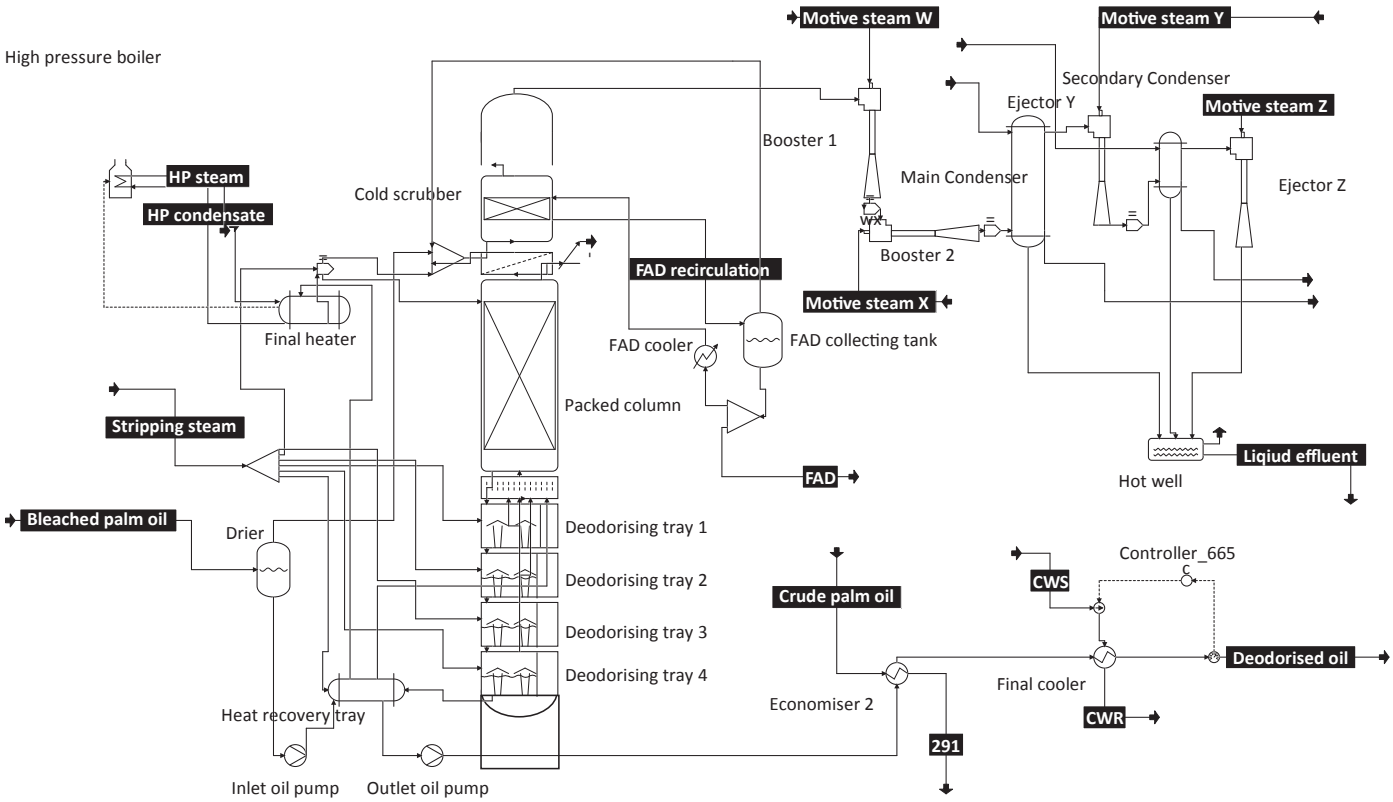
The traffic of a compound (mass transfer) through the interphase is controlled by the thermodynamic equilibrium of the phases and two additional variables:

1. *the interfacial area ($m^2$)*: a measure of how both liquid and vapor phases are contacted
2. *the mass transfer coefficient in each phase (vapor and liquid) ($m\ s^{-1}$)*: a measure of how effective and how fast the mass transfer of compounds is.

These two parameters are directly influenced by the characteristics of the internals in each type of column, and the differences in stripping performance are strongly influenced by these variables.

In the case of structured packing, the interfacial area is highly dependent on the specific packing surface, while in a steam-lift-pump tray, the interfacial area is related to the bub-

FIG. 2: Process simulation model of bleached palm oil deodorization




ble size, bubble diameter, how the bubbles are formed, and on the steam fraction in the mixture.

The interfacial area for a tray, $a_j$, is given by:

$$a_j^I = a'_B h_B A_B$$

in which $a'_B$ ($m^2\ m^{-3}$) is the interfacial area per volume unit (dependent on bubble size and population), $h_B$ (m) is the bubbling height, and $A_B$ ($m^2$) is the bubbling area of the tray.

The interfacial area for a structured pack, $a_j$, is given by:

$$a_j^I = a'_p h_P A_C$$

in which $a'_P$ ($m^2\ m^{-3}$) is the interfacial area per volume unit (dependent on the specific packing area, channel angle), $h_P$ (m) is the packing height, and $A_C$ ($m^2$) is the sectional area of the column.

Mass transfer coefficients [$k^x$] are dependent on the phase velocities and properties of the compounds (viscosity, density, diffusivity). These coefficients are also influenced by equipment characteristics: For a structured packing column, they are a function of the characteristics of the packing (channel angle, void fraction), while in a steam lift pump tray, they are related to the bubble characteristics.

## A PROCESS MODELING AND SIMULATION APPROACH TO PROCESS OPTIMIZATION

Process modeling and simulation studies allow for a systemic approach to optimization, by making it possible to consider all process variables involved in the different unit operations of the process, and to account for mass composition (compounds) of inlet streams.

These studies are not a substitute tool for experimentation or industrial plant tests, but when used in conjunction with them, modeling and simulation studies make it possible to explore unlimited processing scenarios for different qualities and types of vegetable oils. This allows us to: 1. identify the optimal processing scenario and 2. go back to the plant to test and implement the various process optimization strategies. Such an approach can speed up and significantly increase the effectiveness of empirical testing.

## CASE STUDY: DEODORIZATION AND PHYSICAL REFINING OF BLEACHED PALM OIL

Do we have to inject the same stripping steam mass flowrate in all deodorizing trays? Or, can we determine the minimum and accurate amount we need?

The implementation of a process simulation for the physical refining/deodorizing of a bleached palm oil is shown, considering a typical process flow diagram (Fig. 2) to remove

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fatty acids and other volatile compounds from an incoming bleached palm oil, using a stripping column (deodorizer) comprising a section with a structured packing and a section with steam lift pump trays.

This process simulation model allows to represent accurately not only all the main and secondary streams of the different process fluids (vegetable oil, distillates) and utilities (stripping and motive steam, cooling water, high pressure steam) but also the relevant characteristics of the main equipment (deodorizer, drier, heat exchangers, vacuum system)—as well as the complete oil composition in terms of FFAs, triacylglycerols, diacylglycerols, monocylglycerols, tocopherol, sitosterol, moisture, and so on.

As explained before, the characteristics of the packing in the stripping and condensing sections are also considered. This gives us a high degree of certainty of results (Table 1), by taking the type of deodorizer into account.

A typical composition of a crude palm oil from Malaysia is considered. To make a realistic fatty acid composition, instead of considering fatty acid concentration obtained by titration (expressed as palmitic acid), it is preferable to consider the main fatty acids found in palm oil, which are palmitic acid, stearic acid, oleic acid, linoleic acid, and linolenic acid.

**TABLE 1. Main characteristics of structured pack**

| Characteristics | Unit | Stripping section | Scrubbing section |
|---|---|---|---|
| Packing | | Mellapack Plus 250.Y | Mellapack Plus 250.X |
| Diameter | mm | 3100 | 3100 |
| Total height | mm | 3888 | 1334 |
| Void fraction | $m^3\ m^{-3}$ | 0.988 | 0.988 |
| Specific area | $m^2\ m^{-3}$ | 252 | 250 |

Fig. 3 depicts the main results from the simulation run of the process modeling, considering all the main streams and describing for each stream the main process values and mass composition. These are the values you would expect to get when you implement the same process conditions during an industrial plant test.

Since the deodorizer was not modeled as a "black box," but as a stage-to-stage column, it is feasible to calculate and to analyze the traffic of compounds inside the column. To do so, we split the entire column into its main stages, aiming to see the compounds' compositions and streams information

**FIG. 3. Simplified flow diagram with main stream and process information**

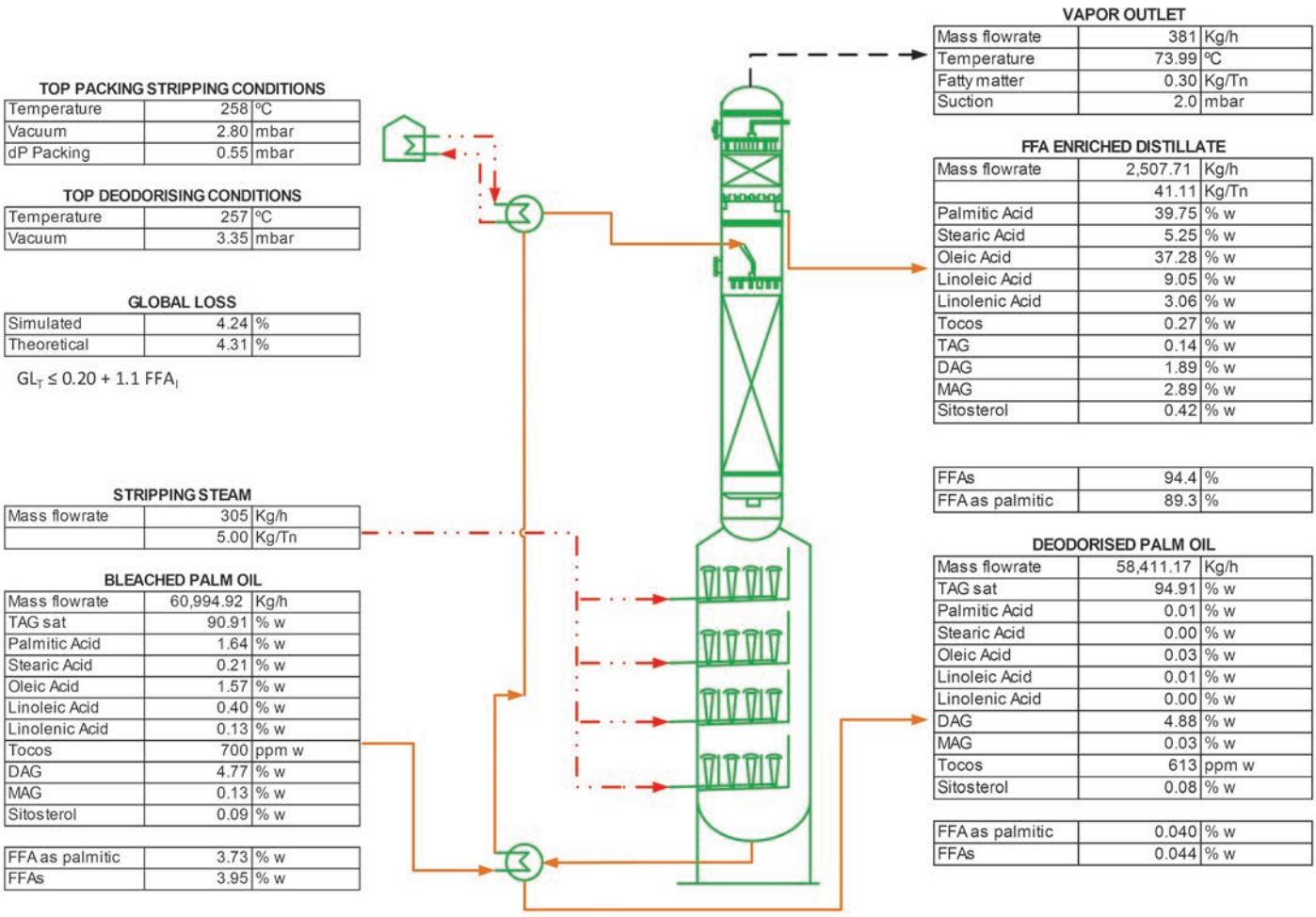


FIG. 4. Detailed study of streams inside the column; schematic

**FFAs removal in each stage**

| Liquid side | Composition | | | | | |
|---|---|---|---|---|---|---|
| | Palmitic Acid | Stearic Acid | Oleic Acid | Linoleic Acid | Linolenic Acid | Total FFAs |
| | (%) | (%) | (%) | (%) | (%) | (%) |
| 2 | 1.64 | 0.21 | 1.57 | 0.40 | 0.13 | 3.95 |
| 49 | 0.33 | 0.07 | 0.52 | 0.13 | 0.05 | 1.11 |
| 3 | 0.01 | 0.01 | 0.03 | 0.01 | 0.00 | 0.06 |
| 59 | 0.01 | 0.00 | 0.03 | 0.01 | 0.00 | 0.06 |
| 20 | 0.01 | 0.00 | 0.03 | 0.01 | 0.00 | 0.05 |
| 6 | 0.01 | 0.00 | 0.03 | 0.01 | 0.00 | 0.05 |
| 24 | 0.01 | 0.00 | 0.03 | 0.01 | 0.00 | 0.04 |

**Stripping steam injection in each stage**

| | | |
|---|---|---|
| Tray 1 | kg $h^{-1}$ | 73.49 |
| Tray 2 | kg $h^{-1}$ | 73.49 |
| Tray 3 | kg $h^{-1}$ | 73.49 |
| Tray 4 | kg $h^{-1}$ | 73.49 |

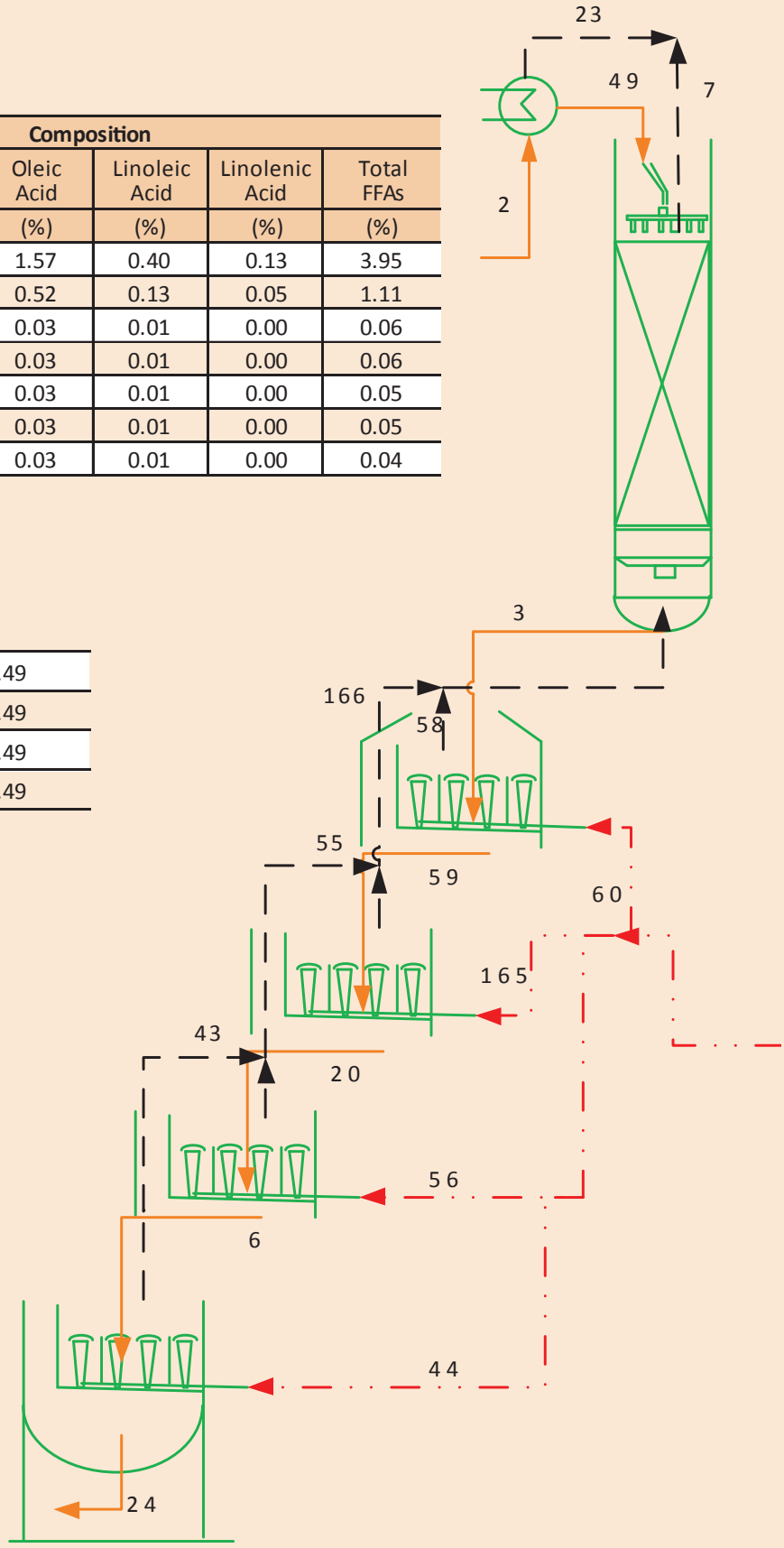
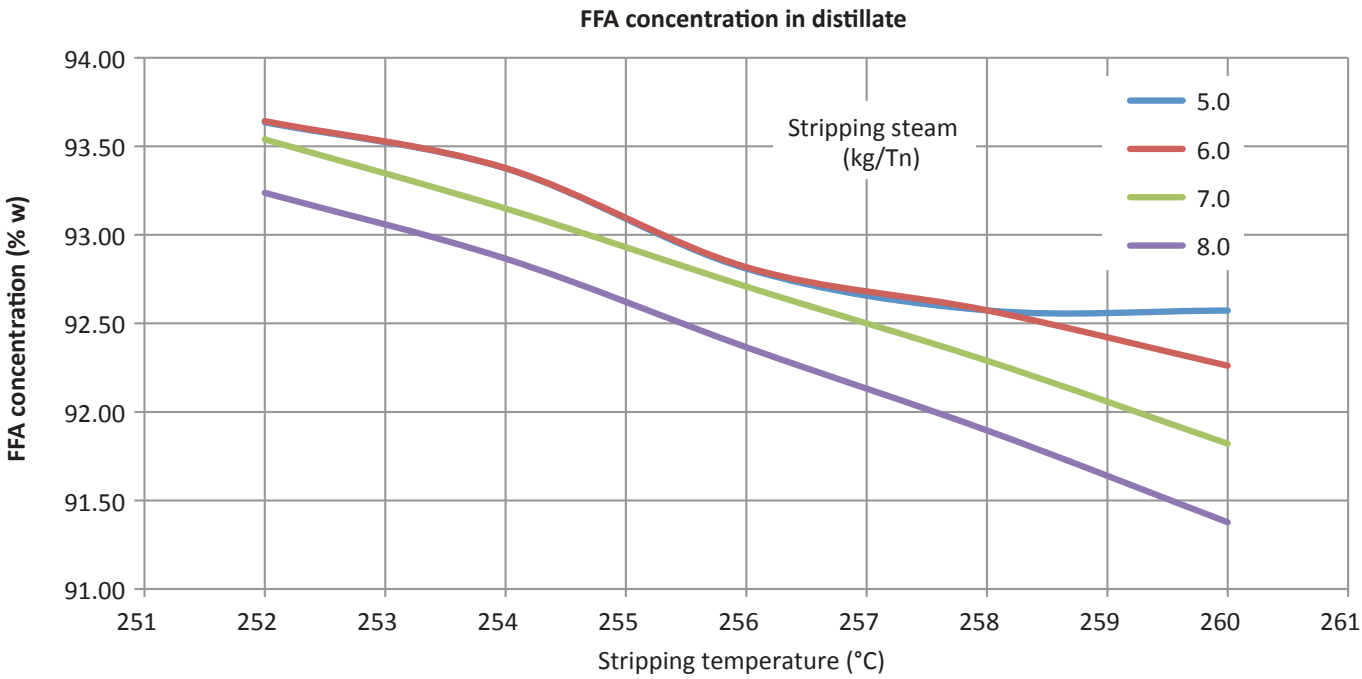


FIG. 5: Sensitivity study




between each stage (Fig. 4, page 25); normally all this information is not feasible to get from an actual deodorizer, since all these "streams" are internal liquid overflows or vapor streams.

As you can see, the process model allows us to get a deep knowledge about what is occurring at each stage inside the column, and then to make decisions about the process. In the case study just presented, if our target of final FFA mass concentration in deodorized palm oil is less than 0.05 % as palmitic, we might stop the injection of stripping steam in tray 4. A lower mass flowrate of stripping steam could then be injected by specifically reducing mass flow from a given tray, instead of reducing the entire injection such as is normally done.

A sensitivity analysis (Fig. 5) is also useful to jointly evaluate the impact of changing stripping temperature and stripping steam mass flowrate on total FFA mass concentration of distillate.

As the previous case study illustrated, process modeling and simulation studies are powerful and very valuable tools that make it possible to predict and analyze characteristics of outlet streams, and to investigate an almost unlimited number of processing scenarios. Such theoretical investigations, together with lab analysis and plant information, help us approach process optimization tasks in a systematic way. The collective information from a simulation run, sensitivity analysis, and detailed study gives us in-depth knowledge about our processes, so we can make appropriate decisions. Time, cost, and certainty of results are the driving factors when using process modeling and simulation.

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## DEFINITION OF TERMS

**Symbol list**

L = liquid molar flow, mol $h^{-1}$
V = vapor molar flow, mol $h^{-1}$
F = feed steam molar flow, mol $h^{-1}$
c = mixture molar density, mol $m^{-3}$
N = molar flux, mol $s^{-1}$ $m^{-2}$
y = vapor molar fraction
x = liquid molar fraction
[k] = matrix of mass transfer coefficients, m $s^{-1}$
a = interfacial area, $m^2$
Ø = mechanical carry-over

**Subscripts, superscripts and acronyms**

*I* = interphase
*J* = stage j
*i* = compound *i*
V = vapor phase
L = liquid phase
t = total
TAG = triacylglycerol
DAG = diacylglycerol
MAG = monacylglycerol
FFA = free fatty acid

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# Ionic liquids in **continuous flow** processes

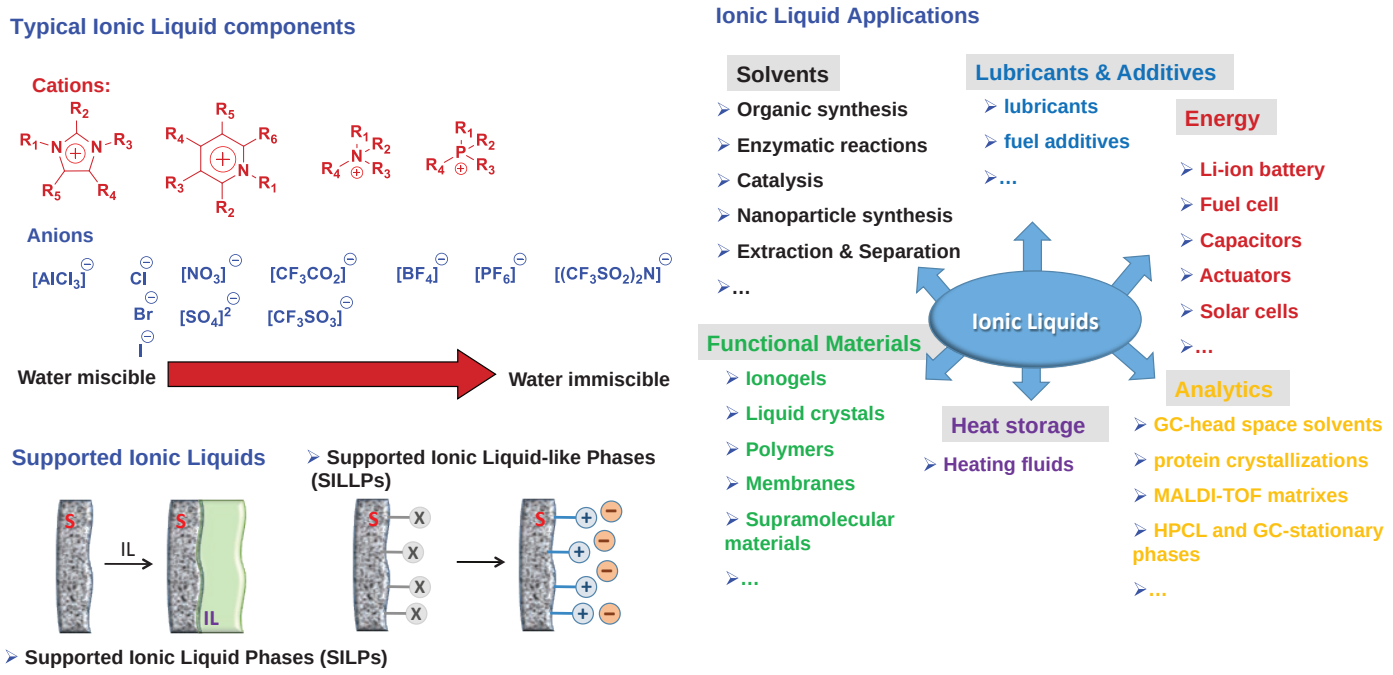
Eduardo García-Verdugo and Santiago V. Luis

- **The combination of ionic liquids (ILs) and continuous flow processes provides new opportunities for developing advanced processes in many fields.**
- **Supported ILs reduce the amount of IL needed, minimize potential leaching to the environment, and greatly simplify the development of flow applications.**
- **This article reviews several ILs, flow applications, and approaches to immobilization.**

Ionic liquids (ILs, Fig. 1) represent one of the most remarkable contributions to the field of green and sustainable chemistry. These salts with low melting points (< 100˚C) are composed of bulky organic cations (imidazolium, ammonium, phosphonium, among others) and a variety of delocalized inorganic or organic anions. Their essential lack of vapor pressure, low flammability, and tunable structures make ILs the solvents of choice for advanced applications in a variety of chemical and technological areas [1]. The use of bulk ILs, however, has some important drawbacks which may include undesirable physico-chemical properties, such as high viscosity; low biodegradability; negative (eco)-toxicological properties; and, finally, high costs that preclude wider application. Consequently, any practical use of ILs must guarantee intensive usage (to reduce the amount needed); full recovery; and prevention of their release to the environment.

With respect to chemical processes, flow conditions enable intensification. This reduces the amount of required energy, time, space, solvents, and materials, and allows some of the above-mentioned requirements to be fulfilled. Such requirements can alternatively be achieved by using supported ionic liquids. Two main approaches have been used to immobilize an IL on a solid support of organic or inorganic nature. The first involves the adsorption of a thin layer of the IL on the surface of the supporting material, which results in supported ionic liquids (SILs). The second involves attaching an IL fragment to a solid through a covalent linkage, which produces supported ionic liquid-like phases (SILLPs). Interestingly, it has been demonstrated that the modified surfaces in SILLPs maintain essentially the same properties ascribed to the related ILs [2]. The use of SILs, particularly SILLPs, makes it possible to significantly reduce the amount of IL needed for a given application, decrease or completely eliminate potential leaching to the environment, and greatly simplify the development of flow applications.

FIG. 1. Ionic liquids, applications, and approaches to immobilization



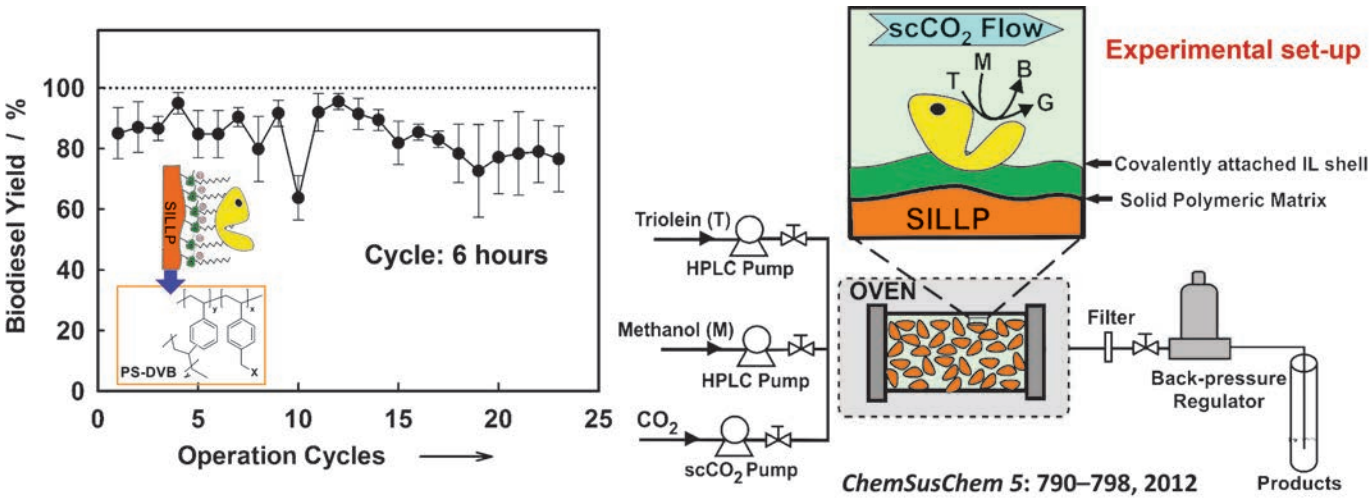

Many different flow applications have been described for ILs. [3] The use in flow of bulk ILs requires biphasic liquid-liquid or liquid-gas systems, while SILs and SILLPs provide solid-liquid or solid-gas systems that are easier to implement and develop for industrial applications. Perhaps the most simple and well-known flow application involves the separation of related products for purification or analysis (chromatography). Examples of applications in this area include the recent approach toward the separation of fatty acid methyl esters (FAMEs) from milk using a silica column modified with an l-phenyl alanine-derived IL. [4] In comparison to standard columns, the introduction of the IL phase improves the separation of many FAMEs in less time and with a different elution order. As a result, chromatographic columns based on ILs are becoming available to support such applications.

The potential of ILs, SILs, and SILLPs with respect to chemical transformations and processes can be illustrated with a few examples. All are based on the potential of IL-related structures to immobilize and stabilize a large variety of catalytic systems—from metal nanoparticles to biocatalysts. In the first example, the synthesis of biodiesel from a model oil, under fully environmentally benign conditions, is considered (Fig. 2, page 30). In this case, macroporous polystyrene-divinylbenzene resins with covalently attached 1-decyl-2-methylimidazolium subunits were able to efficiently immobilize and stabilize CALB lipase. This system was used for the continuous flow synthesis of methyl oleate by the methanolysis of triolein in $scCO_2$ at 18 MPa and 45˚C [5]. The system displayed an excellent stability under the optimized conditions, achieving an 85% mean yield for more than 45 operational cycles of 4–8 h. The exact structure of the IL was revealed to be a key parameter for the optimization of the process, highlighting the importance of the tunable character of ILs. Controlling the structure of the SILLPs in terms of IL and support nature makes it possible, then, to achieve continuous processes for the transformation of vegetable oils into biodiesel.

The same system could be applied for the preparation of chemicals of interest in the area of flavors and fragrances. Thus, SILLP-supported CALB was used for the continuous flow synthesis of citronellyl propionate, again in $scCO_2$ (Fig. 3, page 30). The presence of the IL-fragments provided an exceptional thermal stability to the enzyme. This allowed carrying out the process at 80˚C for more than 10 h of continuous operation without any observable decrease in the efficiency of the process. Yields obtained were higher than 90% for the whole operational cycle. It could be demonstrated that the presence of the IL moiety was essential for this excellent performance. In this case the support was prepared in the laboratory as a monolithic column with the desired chemical and morphological properties according to the needs of the flow process. The possibility of preparing polymeric monoliths specifically designed (shape, porosity, chemical composition, and so on) for the specific requirements of a given application adds a further value to the versatility of these systems.

The third example involves the self-metathesis of oleochemicals, representing also a process of relevance for the conversion of low-value oleo feedstocks into valuable chemicals. In this case, a well-known homogenous Ru catalyst was immobilized into a SIL ([BMIM][$NTf_2$] over $SiO_2$) by including into the catalyst an IL-tag. Conversions of up to 64% for methyl oleate were obtained for the alkene metathesis, with the reactor being stable, at 50˚C, for at least 10 h. Under these conditions, 6 g of substrate could be transformed per hour using only a 9 mL reactor [6] when the reaction was carried out using liquid $CO_2$ as the eluent. As in the case of using $scCO_2$, this allows the isolation of the product(s) without any contamination from the solvent.

**FIG. 2. Continuous synthesis of biodiesel in $scCO_2$ using CALB supported on SILLPs**

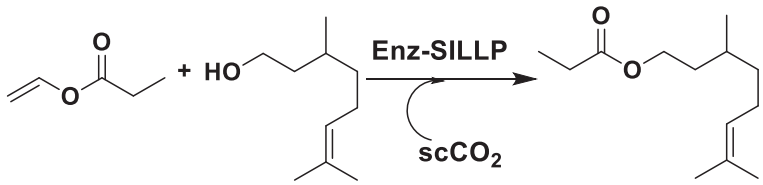


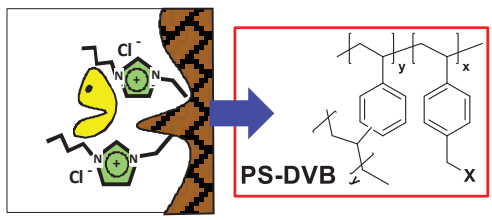

It is worth mentioning that the use of SILs and SILLPs has also allowed the development of flow processes containing several coupled or consecutive chemical transformations. The dynamic kinetic resolution of secondary alcohols to provide valuable chemicals from simple starting materials is an example [7] of this approach. SILLP-immobilized CALB can also be used for the kinetic resolution of secondary alcohols (Fig 4). In this process, only one of the enantiomers is transformed by the lipase into the corresponding ester. According to this scheme, the maximum attainable yield for the ester is 50%, which represents a much-reduced mass efficiency. To enhance the maximum yield that can be obtained, the kinetic resolution can be coupled with a second chemical transformation to racemize the unreacted alcohol, which is further esterified by the action of the enzyme. Coupling the two processes is not a simple matter. Often, this requires the use of very expensive and toxic metal catalysts and/or the use of conditions that are not compatible with the activity/stability window of the enzyme. Immobilization of the catalysts for the two steps in two different solid matrices, guarantees the full isolation of the catalytic sites, allowing this essential limitation to be solved. Accordingly, an efficient dynamic kinetic resolution was

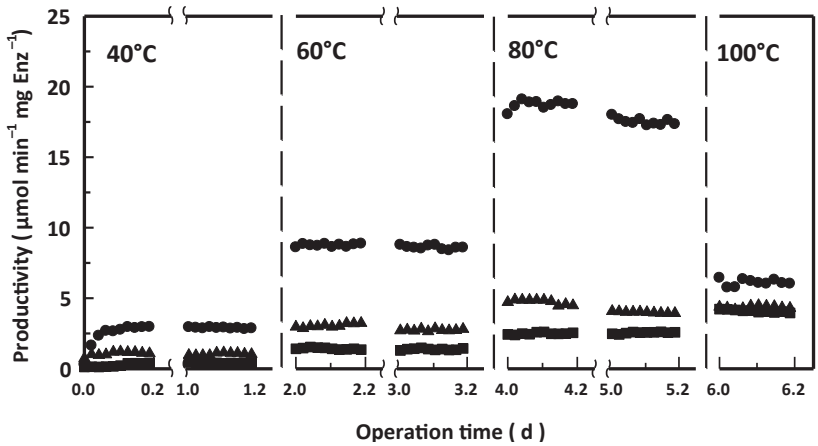
**FIG. 3. Continuous synthesis of a fragrance in scCO2 using CALB supported on SILLPs**

## ADVANTAGES

- reduce the amount of IL
- avoid any loss in the IL phase
- reduce mass-transfer limitations
- stabilize the enzyme activity for large operation times (including high T)
- favor partition effect of the substrate on the enzyme
- enable developing continuous flow processes




**Enzyme:** CALB adsorbed on SILLP

**Substrate:** VB (3.12 M) +citronellol (3.12 M)

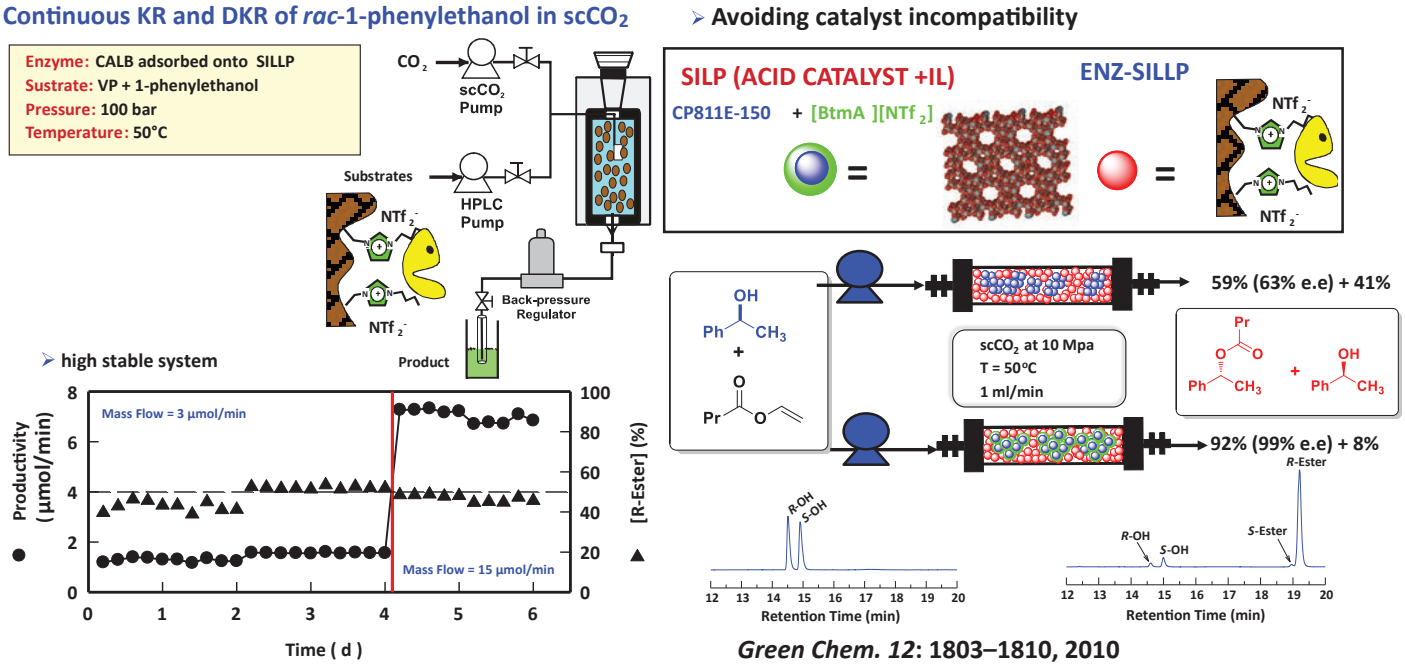
**Pressure:** 100 bar

**Temperature:** 40, 60, 80 or 100°C

● Enz-SILLP-1
▲ Enz-SILLP-2
■ Silica-Butyl

*Adv. Synth. Catal. 349*: 1077–1084, 2007

**FIG. 4. Continuous multicatalytic system based on SILLPs and SILs**




***Green Chem. 12*: 1803–1810, 2010**

achieved by combining an SILLP-supported CALB and an acidic zeolite. The application of this multicatalytic system for flow conditions requires some additional process parameters to be adjusted. Consequently, the use of three consecutive reactors (resolution-racemization-resolution) enhanced the efficiency but with the maximum attainable yield being 75%. Ideally, a full conversion (100%) could only be achieved through the combination of an infinite number of successive reactors. Thus, a different solution was sought. The preparation of a single reactor containing a solid cocktail with beads of SILLP-CALB and zeolite particles provided this infinite number of resolution-racemization-resolution cycles when properly parametrized. To guarantee that the zeolite did not affect the efficiency of CALB, its surface was covered by a layer of IL, turning it into a SIL. When this multicatalytic reactor was used, using $scCO_2$ again as the eluent, a 92% yield of the desired product with a 99.9% ee was obtained.

In summary, the combination of ILs and flow processes provides new opportunities for developing advanced processes in many fields. This is particularly true in the case of supported ILs (SILs and SILLPs), as this approach reduces the amount of IL needed, decreases (SILs) or eliminates (SILLPs) the potential leaching of ILs to the environment, and favors the recovering, reuse, and recycling of the IL-phase. Moreover, using SILs or SILLPs facilitates the elimination or reduction of the concomitant organic solvents used in the process (solventless processes, use of supercritical fluids). Though we have considered here just a few examples involving biocatalysts, zeolites, and organometallic catalysts, many other examples have been reported for flow processes involving ILs, and readers can obtain additional information through the accompanying literature.

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Marisel Corelli Lab 3
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Maike Timm-Heinrich
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Reedville, VA 22539 USA

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## Palm Oil

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## Peanut

*First Place*
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## Phosphorus in Oil

*First Place*
Eric Sweeney
LDC Yorkton Processing GP
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*Honorable Mention*
QA Team
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## Solid Fat Content by NMR

*First Place*
Joseph Maher
Barry Callebaut
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*Honorable Mention*
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Low Thing
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## Soybean Oil

*First Place*
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Andre Thionville
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## Soybeans

*First Place*
Lidieth Solera Carranza
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*Honorable Mention*
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Jesus Gomez Salgado
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## Specialty Oils

*First Place*
Anders Thomsen
Ardin Backous
Brian Gilchrist
Keith Person
Kent Karsjens
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## Tallow and Grease

*First Place*
Adalberto Coronado
National Beef Packing Company
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*Honorable Mention*
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LDC Yorkton Processing GP
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*Honorable Mention*
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*Honorable Mention*
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## trans by IR

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*Honorable Mention*
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Cianciolo, and Derek Gumm
Stratas Foods RDI Center
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## Unground Soybean Meal

*First Place*
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*Honorable Mention*
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Frank Hahn
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Ardin Backous
Brian Gilchrist
Keith Persons
Kent Karsjens
Eurofins Scientific
Des Moines, IA 50321 USA

Mike White
Brian Eskridge
ATC Scientific LLC
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## Vegetable Oil for Color Only

*First Place*
Bill Zubrinic
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Hamilton, ON L8N 3K Canada

## AOCS Board Petition to Nominate

For each annual election of AOCS Governing Board officers, the membership may nominate up to four additional member-at-large candidates by petition. Petitioned candidates receiving at least 50 AOCS member signatures will be added to the ballot approved by the Governing Board. Preference will be given to the first four petitioned candidates with at least 50 signatures. Petitioned nominations must be received at the AOCS Headquarters no later than **October 30, 2016**.

Petition forms can by obtained by visiting www.aocs.org/BoardPetition. Please mail or fax completed petitions with at least 50 AOCS signatures to:
AOCS Nominations and Elections Committee
P.O. Box 17190
Urbana, IL 61803-7190 USA

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

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# Bracing for **Brexit**

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

**Laura Cassiday**

On June 23, 2016, British citizens voted by a slim margin (52% to 48%) in favor of leaving the European Union (EU). The "Brexit" vote shocked many throughout the world who had assumed that the British would instead vote to continue their 43-year EU membership. The day after the vote, UK Prime Minister David Cameron, who had supported continued membership in the EU, resigned his post, to be replaced by Theresa May. The next step will be for the UK to invoke Article 50 of the 2007 Lisbon Treaty, which formally notifies the European Council of plans to exit the EU. Once this has been accomplished, the UK will have 2 years to negotiate a withdrawal agreement with the EU. During this 2-year period, the UK will remain an EU member.

Trade agreements with the EU could take one of three forms: 1) Like Norway, the UK could negotiate a European Economic Area-like agreement with the EU, with tariff-free access to the EU market but freedom to control overseas trade policy; 2) Like Switzerland, the UK could make multiple free trade agreements with EU member states; or 3) If no trade deal is reached, UK exports could be subject to the same tariffs the EU applies to other World Trade Organization members (i.e., 30% tariff on sugars and confectionary, 20% on tobacco and beverages, 10% on vegetables, etc.) (Kushner, G. J., *et al.*, http://tinyurl.com/IFT-Brexit-food, 2016).

In the aftermath of the historic vote, many in the food industry are wondering about the implications for businesses and consumers. The food industry is the UK's largest manufacturing sector, worth £80 billion per year and employing 400,000 people. At the present time, the only thing certain is uncertainty, but many analysts have made predictions about what is in store over the next few months and years:

## MARKET VOLATILITY

When the results of the Brexit vote were announced, the value of the British pound plunged to its lowest level since 1985. Some experts are predicting price inflation as a result of the devalued British pound. The fact that Brexit will not be complete for at least two years means that the market uncertainty could be prolonged.

## INCREASED FOOD PRICES

The UK produces about 40% of the food it consumes, relying on imports for the rest. In particular, the UK relies heavily on EU imports for fresh fruit and vegetables. Tim Lang, a professor in food policy at City University London, told Food Navigator, "People will pay more for food. The British people have voted to raise the food prices" (Michail, N., http://tinyurl.com/Navigator-Brexit-options, 2016). As a result, UK consumers may switch to less-expensive store brands or frequent discount supermarkets, and they may cut discretionary spending on luxury items such as confectionary and snack foods.

Toby Clark, director of research EMEA at the market research firm Mintel (London, UK), offered a more optimistic outlook, opining that grocery spending is unlikely to be substantially affected by Brexit. "For most people, life goes on. They still need to buy groceries … In the short-term at least, everyday spending is unlikely to change dramatically" (Morrison, N., http://tinyurl.com/Brexit-food-costs, 2016).

## DECREASED PROFITABILITY

According to the UK's Food and Drink Federation (FDF), 70% of its members favored continued EU membership (IFT, http://tinyurl.com/IFT-Brexit, 2016). Many industry experts are concerned that Brexit will decrease the profitability of British exports to the EU as they become subject to tariffs, quotas, and increased raw material costs. Unless the UK can negotiate to maintain the trade benefits of the EU single market, the prices of products from the UK will increase, likely affecting sales and profitability of these products in the EU.

Euromonitor International has predicted that confectionary, ready meals, and snack foods will be the hardest hit among packaged foods. From 2015 to 2020, the compound annual growth rate (CAGR) of UK confectionary products is expected to decline by –0.2%.

The agricultural sector in the UK will likely also be affected. In 2015, the EU's Common Agriculture Policy contributed €3.1 billion to UK farmers. Unless the UK government continues the subsidies, agricultural profitability will suffer.

## MORE TRADE WITH THE UNITED STATES

In 2015, more than 70% of UK food and drink exports went to the EU (Yu, D. http://tinyurl.com/Brexit-US-trade, 2016). With the Brexit vote, this situation may change, as the UK looks to boost trade with the US to avoid EU tariffs, quotas, and regulations. In particular, UK confectionary and snack businesses may be interested in increasing US exports. The increased value of the US dollar compared with the British pound means that products from the UK will be cheaper and more attractive to US consumers.

In addition, several countries, including Australia, New Zealand, and South Korea, have informally approached the British government in hopes of discussing free trade agreements (Rousseau, O., http://tinyurl.com/globalmeat-Brexit, 2016). Therefore, increased global exports of UK products may at least partially compensate for a drop in exports to the EU.

## LABOR SHORTAGES

Nearly one-fourth of the UK's food and drink manufacturing workforce of 400,000 people originates from EU countries outside of the UK (Stones, M., http://tinyurl.com/FDF-Brexit, 2016). Tightening borders with the EU could limit workforce availability.

## SHIFTING REGULATIONS

According to Food Navigator, 98% of UK food law is based on EU law (Michail, N., http://tinyurl.com/Navigator-Brexit-options, 2016). The UK could opt to retain this legislation, or establish its own labeling requirements and safety regulations. However, if the UK choose not to abide by EU food and safety regulations, then exports to the EU will be limited.

## FOOD COMPANY EXODUS

As a result of decreased profitability of UK exports, some food companies may decide to relocate away from the UK. To help quell these fears, some companies, such as General Mills (Minneapolis, Minn., USA) and Mondelez (Deerfield, Ill., USA), have released statements indicating their commitment to continue manufacturing in the UK.

Although Unilever (London, UK) publicly supported the "Bremain" option, chief executive Paul Polman tweeted, "The most important thing to have long-term prosperity is to accept the will of the people and respect democracy. Now we all need to unite."

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

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# TSCA reformed: what companies need to know

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

Lynn L. Bergeson

With significant revisions to the Toxic Substances Control Act (TSCA), companies producing or processing industrial chemicals in, or importing them into, the United States will need to become aware of what's changed, quickly. The amendments were immediately effective and important regulatory developments are underway.

If companies consider that these extensive TSCA revisions do not impact their business, they should think again. TSCA reform is far reaching, consequential, and will affect key manufacturing sectors, which includes manufacturers of chemicals and articles imported into the United States. These changes, and the TSCA implementation process now underway, need to be known, understood, and monitored by business people, lawyers, scientists, consultants, product stewards, and other chemical industry stakeholders.

## WHAT JUST HAPPENED?

TSCA is the federal law authorizing the US Environmental Protection Agency (EPA) to regulate the importing, manufacturing, and processing of industrial chemical substances, broadly defined to include just about every industrial chemical going into a manufacturing process for the production of a finished good. Importers of chemicals are defined as "manufacturers." Therefore, chemicals produced off-shore and imported into the United States as industrial chemicals are subject to TSCA, just as they would be if produced domestically.

TSCA was enacted in 1976 and has not been substantively amended since. A number of reasons were behind the reform: the passage of time, the enactment of the European Union's REACH and K-REACH in Korea, and elsewhere globally other progressive chemical management programs, and the rapid proliferation of US state-specific chemical regulatory measures. These developments put pressure on Congress to modernize TSCA and enact reforms urgently needed to assure a restless public that the EPA was capable of delivering on the promise of chemical safety.

## KEY PROVISIONS IN AMENDED TSCA

On June 7, 2016, Congress passed the Frank R Lautenberg Chemical Safety for the 21st Century Act; President Obama signed it into law on June 22. These TSCA amendments fundamentally revise the law, greatly expand the EPA's authority, and address many of the deficits that undermined the agency's ability to manage risks from existing chemicals. The EPA's implementation of these substantial revisions will improve public health protection and restore much of the public's confidence in chemical safety. The new law shifts the burden of demonstrating chemical safety of all chemicals, old and new, to chemical manufacturers, processors, and manufacturers of the finished goods that contain them, away from the EPA proving the opposite. There are many other changes to TSCA, too numerous to outline here. Key changes include the following.

### *Safety reviews are required for all chemicals in commerce.*

The EPA's implementation of the act had been hampered by the lack of a clear legislative mandate to prioritize, evaluate, and regulate the 62,000 plus chemicals originally "grandfathered" under TSCA, placing them beyond the EPA's jurisdictional scope to review before they entered the market. The agency is now required to prioritize and evaluate all existing chemicals in commerce according to enforceable deadlines. Chemicals that may present an unreasonable risk because of a potential hazard and exposure are deemed "high-priority" chemicals, while those not meeting this standard are "low-priority." Where information is insufficient to support a low-priority determination, the default is high-priority. The agency is required to prioritize the review of chemicals that are persistent and bioaccumulative, and that are known carcinogens and highly toxic.

### *Risk evaluation and risk management is required.*

Revised TSCA eliminates the challenging "least burdensome" requirement, previously required for the assessment of chemicals in commerce under the old law. It establishes a safety standard that excludes "consideration of costs or other non-risk factors," and authorizes looking at these in developing risk-abatement measures. Amended TSCA requires the protec-

tion of vulnerable populations, including children and pregnant women.

### *The EPA must make an affirmative determination for all new chemicals.*

Previously, TSCA allowed new chemicals—those not listed on the TSCA inventory—onto the market if the EPA reviewers of premanufacture notices (PMNs) or significant new use notices (SNUNs) did not notify a submitter within 90 days of submission. If the EPA flagged a concern within this timeframe, the review period could be suspended to allow the agency, and the submitter, to address the concern and allow the new substance onto the market, enter into a non-section 5(e) significant new use rule (Snur), negotiate a section 5(e) Snur or withdraw the PMN.

Under amended TSCA, the EPA must make one of three alternate affirmative determinations on all new chemicals and significant new uses of them. These are:

- the new chemical or significant new use presents an unreasonable risk;
- available information is insufficient or the new chemical or significant new use may present an unreasonable risk or it has substantial production and exposure; or
- the new chemical or significant new use is not likely to present an unreasonable risk.

The EPA must regulate under the first two scenarios. If the substance is determined not likely to present an unreasonable risk, the "submitter of the notice may commence" manufacturing (including importing) or processing, even in advance of the expiration of the 90-day review period. Importantly for importers of finished goods, the agency may require notification for the import or processing of a chemical as part of an article if the agency "makes an affirmative finding in a rule … that the reasonable potential for exposure to the chemical substance through the article or category of articles subject to the rule justifies notification." This will make it harder for the EPA to impose Snur requirements on chemicals in imported articles.

### *CBI claims must be substantiated.*

Amended TSCA limits entities' ability to claim information as confidential business information (CBI), requires substantiation of certain claims, including those for chemical identity for existing chemicals, and all expire after 10 years unless reasserted. For new claims, the EPA must affirmatively review all chemical identity CBI claims, and will screen a subset (25%) of non-chemical identity claims. It will review past claims to determine the adequacy of substantiation.

### *Preemption*

Amended TSCA grandfathers state actions taken before April 22, 2016, and those taken pursuant to a state law in effect on August 31, 2003 (California's Proposition 65, for example). After final EPA action, amended TSCA prohibits states from:

- establishing or continuing to enforce measures that duplicate certain TSCA provisions;
- prohibiting or restricting a chemical, after the agency has determined that it does not present an unreasonable risk, following the issue of a final rule under section 6(a); or
- subjecting a chemical to notifications already established under a section 5 Snur.

### *The EPA can order chemical testing and charge higher fees*

Under amended TSCA, the EPA is authorized to mandate chemical testing by order, adding to its preexisting authority to compel testing by rule or consent agreement, including for purposes of establishing chemical prioritization. Importantly, the new law expands the EPA's authority to collect fees from manufacturers and processors for chemical assessments. Fees are widely expected to increase, perhaps significantly.

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*Lynn L. Bergeson is managing partner at Bergeson & Campbell in Washington, DC, USA.*

# Flavor trends in the Latin American food and beverage market

Leslie Kleiner

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

Latin America is a vast and diverse region when it comes to flavor; to better understand what trends are predominant in this area, I interviewed Carlos Calderón, marketing head of Latin America, Symrise Flavor and Nutrition.

**Q: What are current flavor trends in Latin America (LATAM) for confectionery and bakery applications?**

Using a unique methodology for mapping flavor trends, along with quantitative and qualitative tools to analyze market changes and determine the way ahead in terms of flavors trends, we identified four main trends for the confectionery and bakery markets. However, since every organization has its unique branding, a taste fingerprint should be built with each particular brand in mind. For this purpose, Symrise's strategic platform "taste for life®" offers the food industry an innovative tool for faster, tailored, new product development aimed to suit specific needs while optimizing brand and product positioning.

### 1. NATURAL GOODNESS (ARGENTINA)

"Natural" is becoming more relevant for sweets and bakery products. Although organic and local sweets are far from being mainstream, the origin of the ingredients as well as their source plays a crucial role. Natural colors and flavors are usually associated with innovative products like snacks, which are also seeing the incorporation of healthy fruits and nuts. Products that are more traditional are being combined with exotic flavors in a creative way (eg., strawberry meets chia seeds). Vegan and vegetarian inspirations, as well as the use of vegetables (eg., beetroot, sweet potato, carrots, aloe vera) in sweet goods, are also creative tools of current use. However, depending on the market, these may or may not be the most preferable choices for consumers when it comes to price. For example, Argentineans, who are becoming more interested in organic and natural options, may think twice about paying for products with natural ingredients. Given the devaluation of the Peso and the increasing cost of natural ingredients, Argentineans may have to weigh the benefits versus cost when checking out from the supermarket.

### 2. PREMIUM INDULGENCE (BRAZIL)

When it comes to sweets, the premium aspect has always been strong, but the perception of premium has changed. Craft and artisanal approaches are gaining traction, and more and more catego-

ries are being redefined by exploring craft culture as a way to enhance quality. In Brazil, for example, artisan handmade chocolate treats are not new, but there are more people entering the business with small-batch production. Product quality tends to be strong, and product sophistication is achieved by adding adult flavor varieties in exciting versions. In Brazil, people love to prepare sweets and baked goods by themselves. Because of this, Brazil's modern street food in São Paulo is an innovation lab with vibrant scenes of hip food markets. In May 2015, the first market dedicated entirely to sweets ("Festival de Doces") was held in the city, with more than 60,000 participants over the course of a weekend.

Some opportunities and emerging flavors in this segment lie on upgrading common categories by using carefully sourced high-quality ingredients (eg., vanilla from Madagascar, forest berry, Swiss chocolate), and new flavors and ingredients derived from the dessert and breakfast world (eg., cheese cake, tiramisu, dulce de leche, waffle, brownie).

### 3. HEALTHFUL TREATS (MEXICO)

As consumers demand healthy and tasty products, healthy snacking becomes more popular, bringing health-fun and health-indulgence to the category. Since Mexico's Secretary of Health recently declared that Mexico leads the world in childhood obesity (~35% of children are obese or overweight), functional food and beverages are expected to be a top trend in 2016.

Super fruits, ginger, guarana, amaranth, and chia are additions that support current trends for this segment. Also, healthful treats can feature protein-rich ingredients with protein originating from plant-based sources.

### 4. EMOTIONAL DISCOVERIES (COLOMBIA)

Mix and match, as well as hybrid and fusion cuisine, have become a norm when providing new discoveries. For example, Colombian sweet makers are used to adapting foreign products to Colombian tastes, but are then willing to experiment more as the market matures. However, the nostalgia market is a strong counter-trend to creative hybrids, since many consumers look for products that provide an emotional connection to childhood or other periods in life.

In this category, Latin American consumers seek for more regional, bolder, and authentic flavors (eg., acai, guava, agave), as well as different flavor profiles (eg., flavor-changing candy), flavors that push boundaries (eg., sweet and savory combinations), and flavors known from childhood (eg., marshmallow, chocolate, cotton candy, popcorn, and birthday cake).

**Q: What are the trends in LATAM regarding natural and artificial flavors?**

A report by Allied Market Research, mentions that in 2014, the natural flavors segment was leading in terms of value. A strong demand for organic processed food and beverages remains the major growth driver for the natural flavors market segment. The growth outlook for natural flavors remains strong, while artificial flavors may witness negative growth. Over 2015–2020, the biggest challenge for manufacturers, both big and small, is to maintain their products at affordable prices for Latin consumers during an unfavorable economic scenario, while dealing with increasing cost pressures (eg., raw materials, packaging, and infrastructure).

**Q: How are flavors used in nutraceutical applications to reinforce the health benefits?**

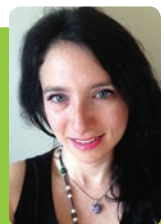
Symrise's value proposition offers strong R&D capabilities in flavors, consumer health, and life essentials, which will advance innovations in key business segments such as nutraceuticals. With the Acquisition of Diana Group in 2014, we now offer tailor-made solutions from natural raw materials for the food industry. In addition, Diana's nutritional concepts help to reinforce the health benefit of products by using functional actives. Through its Nutri'Health™ range, Diana Food offers numerous natural ingredients obtained from fruit and vegetables with intrinsic organoleptic and nutritional properties (Nutri'balance), and standardized actives ingredients with characterized health benefits to boost product formulation (Boost'health).

**Q: Are there flavors that we can soon expect to see in the United States?**

With the growth of the Hispanic population in the United States, Latin American cuisine became quite popular. In particular, Mexican flavors had a significant influence on American cuisine and eating habits. For sweet and baked products, we see various LATAM flavors that are becoming more mainstream (eg., dulce de leche, cilantro, orchard Mexican style, chili churritos, hot spicy, tamarind, and tropical established flavors like guava, mango, coconut, and acai).

As to future flavor influencers, I think that Peruvian cuisine will soon influence US tastes with novel ethnic products such as "Tiradito," "Lomo Saltado," Huancayo-style potatoes, Pisco sour, functional ingredients like quinoa, and the fruits "Lúcuma," "camu camu," and "chicha morada". Peruvian cuisine may seem like a smaller portion of the Latin palette, but it has already painted many New York restaurants. Peru has one of the greatest cuisines of the world, since it is an original fusion-cuisine, having absorbed influences from almost every continent over the last 500 years, and mixed them with ingredients and dishes that provide a direct link to the Incas.

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



# PATENTS

## Thermal methods for treating a metathesis feedstock

Uptain, K.D., *et al.*, Elevance Renewable Sciences, US9284515, March 15, 2016

Various methods are provided for metathesizing a feedstock. In one aspect, a method includes providing a feedstock comprising a natural oil, heating the feedstock to a temperature greater than 100˚C in the absence of oxygen, holding the feedstock at the temperature for a time sufficient to diminish catalyst poisons in the feedstock, and, following the heating and holding, combining a metathesis catalyst with the feedstock under conditions sufficient to metathesize the feedstock.

## Disinfectant detergent composition

Wakita, K. and M. Satoko, NOF Corp., US9288977, March 22, 2016

A disinfectant detergent composition containing a food or food additive as a main component is provided. The disinfectant detergent composition contains the following components (a), (b), and (c). The sum [a+b] of the component (a) content and the component (b) content of the composition ranges from 0.1% to 30% by mass, the mass ratio [a/b] of the component (a) to the component (b) ranges from 1/2 to 4/1, and the component (c) content of the composition ranges from 60% to 99.5% by mass. (a) polyoxyethylene sorbitan monolaurate (b) a fatty acid glyceride in which the number of carbon atoms of its acyl group ranges from 8 to 12, the monoglyceride content of the component (b) being 85% by mass or more, the mass fraction of 1-monoglyceride relative to the total amount of monoglycerides being in the range of 0.9 to 1.0 (c) water and/or ethanol.

## Human sebum mimetics derived from botanical sources and methods for making the same

Brown, J.S., *et al.*, International Flora Technologies, Ltd., US9289373, March 22, 2016

Human sebum mimetics and methods for producing human sebum mimetics are provided. In one exemplary embodiment, a human sebum mimetic comprises a wax ester derived from interesterification of refined botanical oil comprising palmitoleic acid and refined jojoba oil, a phytosterol, and phytosqualene. A method for producing a human sebum mimetic comprises mixing refined macadamia oil and refined jojoba oil, interesterifying the refined macadamia oil and the refined jojoba oil, adding a phytosterol after the interesterifying, and adding phytosqualene after the interesterifying.

## Curable and cured compositions

Campbell, C.J. *et al.*, 3M Innovative Properties Co., US9290683, March 22, 2016

Curable compositions, cured compositions, and articles that include the cured compositions are described. The curable composition contains a) an epoxy resin, b) a curing agent, c) a reactive liquid modifier, and d) a toughening agent. The reactive liquid modifier is an acetoacetate ester of a polyol that is a vegetable oil, that is prepared from a vegetable oil, or that is a mixture thereof. The cured compositions can be used as adhesives such as structural adhesives or as polymeric coatings.

## Substitute for fat within meat and the forming method thereof

Chang, H.W., *et al.*, Food Industry Research and Development Institute, US9295277, March 29, 2016

The invention provides a meat-fat substitute, the forming composition thereof including: at least one edible gum; at least one starch; and water, wherein the at least one edible gum is present in an amount of about 0.5–20 parts by weight, the at least one starch is present in an amount of about 0.5–10 parts by weight and the water is present in an amount of about 60–99 parts by weight.

## Method for supercritical diesel combustion

Stone, C.S., WSC Three S.A., US9297299, March 29, 2016

A method for superheated glycerin combustion (SGC) combines fumigation and SGC to effect greater fuel efficiency and reduce exhaust gas pollutants from a compression ignition engine such as a diesel engine. The invention utilizes the fumigant method by combining two gases (dimethyl ether and propane) which auto ignite prior to the injection of the liquid glycerin water solution (GWS) fuel. This pre-combustion of the fumigant gases combined with the engine's compression of the combustion chamber gases is managed to attain a supercritical combustion chamber environment into which the liquid GWS fuel is injected. This targeted supercritical combustion chamber environment causes the GWS fuel to first vaporize the water which leaves the glycerin, prior to combustion, as highly dispersed monomers within the combustion chamber which auto ignite similar to a "homogenous charge compression ignition" (HCCI) event resulting in significantly greater efficiency and negligible exhaust gas pollutants.

## Air flow cooking device

Payen, J. M., *et al.*, SEB S.A. US9301644, April 5, 2016

The invention relates to a household cooking appliance (1) comprising food-receiver (2) for receiving loose pieces of food (100), and heater means including generator means (3) for generating a flow of air (4), said household appliance being characterized in that the generator means (3) for generating the flow of air (4) include an air outlet (5) and an air inlet (6), the appliance (1) further comprising a screen (7) arranged relative to the air outlet (5) and to the air inlet (6) so that it forces the flow of air (4) to go

around the screen (7) in order to reach the air inlet (6) from the food-receiver (2). Cooking appliances of the deep fryer type.

## Liquid discharge recording apparatus and method for recovering liquid

Sugimoto, J., *et al.*, Brother Ind. Ltd., US9302489, April 5, 2016

A liquid discharge recording apparatus includes: a liquid; a liquid discharge head configured to discharge the liquid; an absorber configured to absorb the liquid discharged from the liquid discharge head; and unsaturated fatty acid of which specific gravity is smaller than that of the liquid and which is contained in the absorber.

## Immobilization of enzymes

Mazeaud, I., *et al.*, Novozymes A.S., US9303256, April 5, 2016

The present invention relates to the immobilization of enzymes by adsorbing enzymes, a polyfunctional amine and a cross-linking agent onto a particulate porous carrier in a mixer apparatus or in a fluid bed apparatus.

## Bioremediation of soil and groundwater

Archibald, J. and G.M. Birk, Tersus Environmental LLC, US9309136, April 12, 2016

A composition for the bioremediation of soil or groundwater includes 35 to 60% by weight of an emulsifiable C4-C22 vegetable oil such as soybean or corn oil, about 60–35% by weight of a non-ionic surfactant/co-surfactant blend such as Tween 80(R)/Labrafil(R) mixed with Kolliphor EL(R)/Waglinol(R), and about 0–12% by weight water.

## Biological effects of compositions comprising rosmarinic acid

Crespy, V., *et al.*, Nestec S.A.; L'Oreal, US9314490, April 19, 2016

The present invention relates to compositions of rosmarinic acid or its derivatives and to the use of a hydrolytic enzyme or of microorganism containing or producing hydrolytic enzymes in these compositions. The invention also pertains to methods for improving the biological effects of the rosemary extracts and for administering such compositions to a human or animal subject for improving the skin, coat, hair or health of the subject.

## Bio-renewable plasticizers derived from vegetable oil

Kodali, D., *et al.*, Regents of the University of Minnesota, US9315650, April 19, 2016

A composition includes estolide esters of vegetable oil fatty acid alkyl esters where the vegetable oil has an unsaturation greater than 90 IV. The fatty acid alkyl esters include unsaturated and saturated fatty acid alkyl esters, and each unsaturated fatty acid alkyl ester has greater than one estolide ester functionality.

## Dispersants having biobased compounds

Baseeth, S., *et al.*, Archer Daniels Midland Co., US9315652, April 19, 2016

The present disclosure is directed to compositions having lecithin and an organic acid and related methods. The disclosed compositions may also include one or more co-surfactants such as anionic surfactants and/or non-ionic surfactants, and may be used as a dispersant.

## Green approach in metal nanoparticle-embedded antimicrobial coatings from vegetable oils and oil-based materials

John, G., *et al.*, Research Foundation of the City University of New York, US9315676, April 19, 2016

The present invention generally relates to a method of making nanoparticles and uses thereof. In particular, the invention relates to methods of making metal nanoparticles (MNPs). The invention also relates to antimicrobial uses of the nanoparticles.

## Vegetable oil-based pressure sensitive adhesives

Li, K. and A. Li, Oregon State Univ., US9315704, April 19, 2016

A pressure sensitive adhesive construct comprising: (a) a backing substrate; and (b) a pressure sensitive adhesive composition disposed on the backing substrate, wherein the pressure sensitive adhesive includes a product made from at least one epoxidized vegetable oil and at least one dibasic acid or anhydride, or a combination of a dibasic acid or anhydride and a monobasic acid or anhydride.

## Method of processing phospholipid based lipid materials

Dasari, M.P.A. and A. Mahfuz, Kru Ltd., D/B/A Feed Energy Co., US9315764, April 19, 2016

The present invention provides methods of processing lipid materials such as soapstock, wet gums, and dry gums. Enzymes are utilized to catalyze hydrolysis of the lipids materials to recover fatty acids. Addition of organic acids and/or polyols improved yield of fatty acids and reduced formation of emulsion. Lipid materials can be formulated with other agricultural products as new value-added animal fee products.

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

# EXTRACTS & DISTILLATES

## Antioxidant behavior of olive phenolics in oil-in-water emulsions

Paradiso, V.M., *et al., J. Agric. Food Chem., 64*: 5877–5886, 2016, http://dx.doi.org/10.1021/acs.jafc.6b01963.

The effect of the surrounding molecular environment (β-lactoglobulin as an emulsion stabilizer and maltodextrin as a viscosity modifier) on the antioxidant activity of three olive oil phenolic compounds (PCs) in olive oil-in-water emulsions was investigated. Oxidation potential, phenolic partitioning, and radical quenching capacity were assessed in solution and in emulsion for oleuropein, hydroxytyrosol, and tyrosol; the influence of β-lactoglobulin and maltodextrin concentration was also evaluated. Finally, the observed properties were related to the oxidative stability of the emulsions containing the PCs to explain their behavior. The order hydroxytyrosol > oleuropein > tyrosol was observed among the antioxidants for both oxidation potential and radical quenching activity. Radical quenching capacity in emulsion and anodic potential were complementary indices of antioxidant effectiveness. As the intrinsic susceptibility of an antioxidant to oxidation expressed by its anodic potential decreased, the environmental conditions (molecular interactions and changes in continuous phase viscosity) played a major role in the antioxidant effectiveness in preventing hydroperoxide decomposition.



## Binding to bovine serum albumin protects β-carotene against oxidative degradation

Chang, H.-T., *et al., J. Agric. Food Chem. 64*: 5951–5957, 2016, http://dx.doi.org/10.1021/acs.jafc.6b02436.

Binding to bovine serum albumin (BSA) was found to protect β-carotene (β-Car) dissolved in air-saturated phosphate buffer solution/tetrahydrofuran (9:1, v/v) efficiently against photobleaching resulting from laser flash excitation at 532 nm. From dependence of the relative photobleaching yield upon the BSA concentration, an association constant of $K_a = 4.67 \times 105$ L mol$^{-1}$ for β-Car binding to BSA was determined at 25 °C. Transient absorption spectroscopy confirmed less bleaching of β-Car on the microsecond time scale in the presence of BSA, while kinetics of triplet-state β-Car was unaffected by the presence of oxygen. The protection of β-Car against this type of reaction seems accordingly to depend upon dissipation of excitation energy from an excited state into the protein matrix. Static quenching of BSA fluorescence by β-Car had a Stern–Volmer constant of $K_{sv} = 2.67 \times 104$ L mol$^{-1}$, with $\Delta H = 17$ kJ mol$^{-1}$ and $\Delta S = 142$ J mol$^{-1}$ K$^{-1}$ at 25 °C. Quenching of tryptophan (Trp) fluorescence by β-Car suggests involvement of Trp in binding of β-Car to BSA through hydrophobic interaction, while the lower value for the Stern–Volmer constant $K_{sv}$ compared to the binding constant, $K_a$, may indicate involvement of β-Car aggregates. Bound β-Car increased the random coil fraction of BSA at the expense of α-helix, as shown by circular dichroism, affecting the β-Car configuration, as shown by Raman spectroscopy.

## Technoeconomic analysis of small-scale farmer-owned camelina oil extraction as feedstock for biodiesel production: A case study in the Canadian prairies

Mupondwa, E., *et al., Ind. Crops Prod. 90*: 76–86, 2016, http://dx.doi.org/10.1016/j.indcrop.2016.05.042.

This study evaluated costs and profitability associated with small scale camelina oil extraction plant in the Canadian Prairies for the purpose of selling camelina oil for further biodiesel production. In this case, *Camelina sativa* is targeted for production on underutilized summerfallow land to avoid displacement of crop lands. Saskatchewan soil zone 7A has the capacity to provide camelina for oil extraction based on small scale capacities of 30,000–120,000 t annum$^{-1}$ and capital investment of $10–24 million. Oil production price is reduced with increased camelina oil content, field yield, plant scale, and camelina meal price. Oil production costs range from $0.39 to $1.88 L$^{-1}$ when camelina meal has a market value of $0.30 kg$^{-1}$. These results provide an informative basis for investment decisions by farmers and investors vis-à-vis the advancement of farm-adoption of camelina as a dedicated industrial crop, as well as the development of an integrated camelina-to-processing oilseed value-chain.

## Squalene in virgin olive oil: screening of variability in olive cultivars

Beltrán, G., *et al., Eur J. Lipid Sci. Technol. 118*: 1250–1253, 2016, http://dx.doi.org/10.1002/ejlt.201500295.

Twenty-eight olive cultivars (*Olea europaea L.*) from the World Olive Germplasm Collection of IFAPA in Cordoba were studied, analyzing the squalene concentration of their virgin olive oils (VOO). Squalene content ranged from 110 to 839 mg/100 g achieving a mean value for the set of olive cultivars of 502 mg/100 g. The high variability observed could be explained only by the genetic component. Five olive cultivar categories were established for the oil squalene concentration when a Hierachical discriminant analysis was performed. Virgin olive oil can be considered an important source of squalene, it may be used also for characterization and discrimination of monovarietal virgin olive oils.

## Formation of chlorinated lipids post-chlorine gas exposure

Ford, D.A., *et al., J. Lipid Res. 57*: 1529–1540, 2016, http://dx.doi.org/10.1194/jlr.M069005.

Exposure to chlorine ($Cl_2$) gas can occur during accidents and intentional release scenarios. However, biomarkers that specifically indicate $Cl_2$ exposure and $Cl_2$-derived products that mediate postexposure toxicity remain unclear. We hypothesized that chlorinated lipids (Cl-lipids) formed by direct reactions between $Cl_2$ gas and plasmalogens serve as both biomarkers and mediators of post-$Cl_2$ gas exposure toxicities. The 2-chloropalmitaldehyde (2-Cl-Pald), 2-chlorostearaldehyde (2-Cl-Sald), and their oxidized products, free- and esterified 2-chloropalmitic acid (2-Cl-PA) and 2-chlorostearic acid were detected in the lungs and plasma of mouse and rat models of $Cl_2$ gas exposure. Levels of Cl-lipids were highest immediately post-$Cl_2$ gas exposure, and then declined over 72 h with levels remaining 20- to 30-fold higher at 24 h compared with baseline. Glutathione adducts of 2-Cl-Pald and 2-Cl-Sald also increased with levels peaking at 4 h in plasma. Notably, 3-chlorotyrosine also increased after $Cl_2$ gas exposure, but returned to baseline within 24 h. Intranasal administration of 2-Cl-PA or 2-Cl-Pald at doses similar to those formed in the lung after $Cl_2$ gas exposure led to increased distal lung permeability and inflammation and systemic endothelial dysfunction characterized by loss of eNOS-dependent vasodilation. These data suggest that Cl-lipids could serve as biomarkers and mediators for $Cl_2$ gas exposure and toxicity.

## Association between serum long-chain omega-3 polyunsaturated fatty acids and cognitive performance in elderly men and women: The Kuopio Ischaemic Heart Disease Risk Factor Study

Ascoli, T.A.D., *et al., Eur. J. Clin. Nutr. 70*: 970-975, 2016, http://dx.doi.org/10.1038/ejcn.2016.59.

Fish intake and the long-chain omega-3 polyunsaturated fatty acids (PUFAs) in fish have been suggested to lower the risk of cognitive decline. We assessed whether serum long-chain omega-3 PUFAs eicosapentaenoic acid (EPA), docosapentaenoic acid (DPA) and docosahexaenoic acid (DHA) are associated with performance on neuropsychological tests in an older population and whether exposure to methylmercury, mainly from fish, or apolipoprotein-E4 (Apo-E4) phenotype can modify the associations. A total

of 768 participants from the population-based Kuopio Ischaemic Heart Disease Risk Factor Study were included. Cognitive function was measured using five neuropsychological tests: the Trail Making Test, the Verbal Fluency Test, the Selective Reminding Test, the Visual Reproduction Test and the Mini Mental State Exam. Multivariate-adjusted analysis of covariance and linear regression were used to analyze the cross-sectional associations. We found statistically significant associations between serum EPA+DPA+DHA and better performance in the Trail Making Test and the Verbal Fluency Test. The individual associations with EPA and DHA were similar with the findings with EPA+DPA+DHA, although the associations with DHA were stronger. No associations were observed with serum DPA. Pubic hair mercury content was associated only with a worse performance in the Trail Making Test, and mercury had only little impact on the associations between the serum PUFAs and cognitive performance. Apo-E4 phenotype did not modify the associations with PUFAs or mercury. Higher serum long-chain omega-3 PUFA concentrations were associated with better performance on neuropsychological tests of frontal lobe functioning in older men and women. Mercury exposure or Apo-E4 phenotype had little impact on cognitive performance.

## Glycolipid biosurfactants: main properties and potential applications in agriculture and food industry

Mnif, I., *et al., J. Sci. Food Agric. 96*: 4310–4320, 2016, http://dx.doi.org/10.1002/jsfa.7759.

Glycolipids, consisting of a carbohydrate moiety linked to fatty acids, are microbial surface active compounds produced by various microorganisms. They are characterized by high structural diversity and have the ability to decrease the surface and interfacial tension at the surface and interface, respectively. Rhamnolipids, trehalolipids, mannosylerythritol lipids and cellobiose lipids are among the most popular glycolipids. They have received much practical attention as biopesticides for controlling plant diseases and protecting stored products. As a result of their antifungal activity towards phytopathogenic fungi and larvicidal and mosquitocidal potencies, glycolipid biosurfactants permit the preservation of plants and plant crops from pest invasion. Also, as a result of their emulsifying and antibacterial activities, glycolipids have great potential as food additives and food preservatives. Furthermore, the valorization of food byproducts via the production of glycolipid biosurfactant has received much attention because it permits the bioconversion of byproducts on valuable compounds and decreases the cost of production. Generally, the use of glycolipids in many fields requires their retention from fermentation media. Accordingly, different strategies have been developed to extract and purify glycolipids.

## Phytyl fatty acid esters in the pulp of bell pepper (*Capsicum annuum*)

Krauß, S., *et al., J. Agric. Food Chem. 64*: 6306–6311, http://dx.doi.org/10.1021/acs.jafc.6b02645.

Phytyl fatty acid esters (PFAE) are esters of fatty acids with the isoprenoid alcohol phytol (3,7R,11R,15-tetramethylhexadec-2E-enol). In this study, PFAE were identified and quantified in bell pepper using gas chromatography with mass spectrometry (GC-MS). All red ($n = 14$) and yellow ($n = 6$) samples contained six or seven PFAE at 0.9–11.2 mg/100 g fresh weight. By contrast, PFAE were not detected in green bell pepper samples ($n = 3$). PFAE might eventually be a source for bioavailable phytol, which can be transformed into phytanic acid by humans. Phytanic acid cannot be properly degraded by patients who suffer from Refsum's disease (tolerable daily intake (TDI) ≤ 10 mg of phytanic acid). The phytol moiety of the PFAE (0.4–5.4 mg/100 g fresh weight) would contribute up to •50% to the TDI with the consumption of only one portion of bell pepper fruit pulp.

## Addition of aspirin to a fish oil-rich diet decreases inflammation and atherosclerosis in ApoE-null mice

Sorokin, A.V., *et al., J. Nutr. Biochem. 35*: 58–65, 2016, http://dx.doi.org/10.1016/j.jnutbio.2016.05.012.

Aspirin (ASA) is known to alter the production of potent inflammatory lipid mediators, but whether it interacts with omega-3 fatty acids (FAs) from fish oil to affect atherosclerosis has not been determined. The goal was to investigate the impact of a fish oil-enriched diet alone and in combination with ASA on the production of lipid mediators and atherosclerosis. ApoE$^{-/-}$ female mice were fed for 13 weeks one of the four following diets: omega-3 FA deficient (OD), omega-3 FA rich (OR) (1.8 g omega-3 FAs/kg·diet per day), omega-3 FA rich plus ASA (ORA) (0.1 g ASA/kg·diet per day) or an omega-3 FA deficient plus ASA (ODA) with supplement levels equivalent to human doses. Plasma lipids, atherosclerosis, markers of inflammation, hepatic gene expression and aortic lipid mediators were determined. Hepatic omega-3 FAs were markedly higher in OR (9.9-fold) and ORA (7-fold) groups. Mice in both OR and ORA groups had 40% less plasma cholesterol in very low-density lipoprotein-cholesterol and low-density lipoprotein fractions, but aortic plaque area formation was only significantly lower in the ORA group (5.5%) compared to the OD group (2.5%). Plasma PCSK9 protein levels were approximately 70% lower in the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OR and ORA groups. Proinflammatory aortic lipid mediators were 50%–70% lower in the ODA group than in the OD group and more than 50% lower in the ORA group. In summary, less aortic plaque lesions and aortic proinflammatory lipid mediators were observed in mice on the fish oil diet plus ASA vs. just the fish oil diet.

## Industrial Applications

### Oilseed crop crambe as a source of renewable energy in Brazil

Bassegio, D., *et al., Renew. Sust. Energ. Rev. 66*:311–321, 2016, http://dx.doi.org/10.1016/j.rser.2016.08.010.

Crambe (*Crambe abyssinica* Hochst. Ex R. E. Fries) is an oilseed with the potential for cultivation in Brazil as a source of renewable energy in areas with tropical conditions. Crambe oil contains erucic acid, giving it economic importance for use in the electricity sector. Crambe oil can be used in insulating fluids and in the manufacture of chemical products and biodiesel without competing with human food production. As it is a new crop in Brazil, the scientific literature on crambe is still scarce. This review discusses general aspects of agricultural production and the potential applications of crambe associated mainly with the manufacture of biodiesel. The topics discussed in this review include: (i) genetic improvement of crambe; (ii) agronomic practices of production, pests, diseases, harvesting, storage, and economic viability; (iii) industrial use of crambe oil as an electrical insulator and in biodiesel; (iv) detoxification of the co-product generated during oil extraction for use in animal feed and use of the co-product in the natural bioremediation of contaminants; and (v) the challenges of increasing crambe production. Crambe has a remarkable future as a source of renewable energy in Brazil. Strategies to boost its cultivation, such as the identification of improved varieties and optimization of production system logistics, marketing, and resource allocation, should be adopted.

### Epoxy monomers obtained from castor oil using a toxicity-free catalytic system

Parada Hernandez , N.L., *et al., J. Mol. Catal.*, online first, 2016, http://dx.doi.org/10.1016/j.molcata.2016.08.005.

In order to obtain monomers from vegetable source, the castor oil epoxidation process was investigated. The catalytic system used in this work, $H_2O_2$/alumina/ethyl acetate, can be considered as a green system, free of heavy metals and toxic solvents. These characteristics make the system appropriate for the purpose of this study since they increase the probabilities of obtaining a biomaterial with the desired specifications regarding toxicity. Reaction conditions of castor oil epoxidation were optimized using methyl ricinoleate as a model compound. In order to identify the operating region, it was developed an experimental design $2^3$ with 17 assays (6 axial points and central point in triplicate) in which, methyl ricinoleate, hydrogen peroxide and catalyst initial quantities in the reaction mixture were the studied variables. The system showed great efficiency with 100% of selectivity in the methyl ricinoleate epoxide production. In optimized conditions, it showed conversion of 99% in 6 h. It was obtained a conversion of 94%, an epoxidation percentage of 84 and a selectivity of 89% toward the epoxides for the castor oil epoxidation. These results show the efficacy of the catalytic system used in this work. Epoxidized castor oil structure was confirmed by FTIR, Raman and $^1$H NMR techniques.

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