

# INFORM

International News on Fats, Oils, and Related Materials

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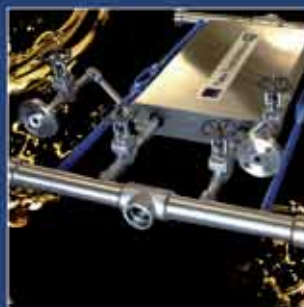
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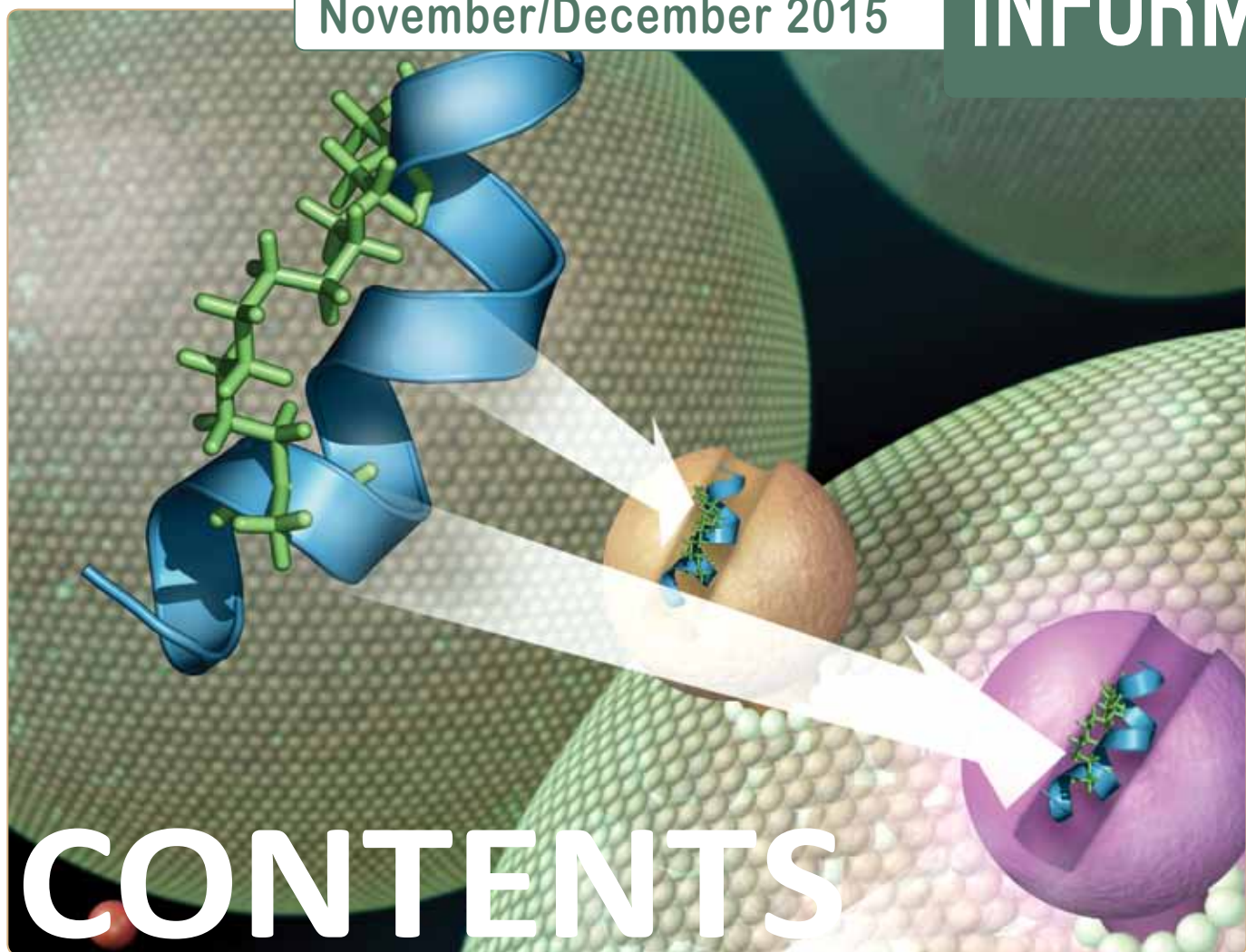


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Credit: Eric Smith, laboratory of Dr. Loren Walensky, Dana-Farber Cancer Institute

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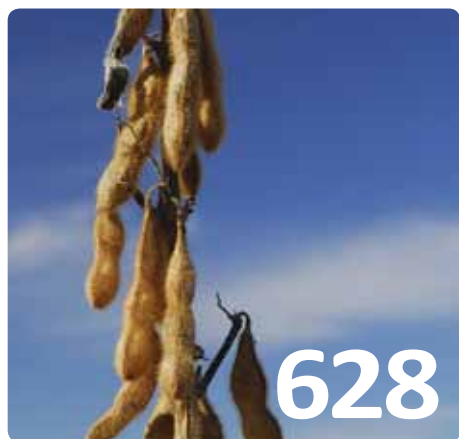
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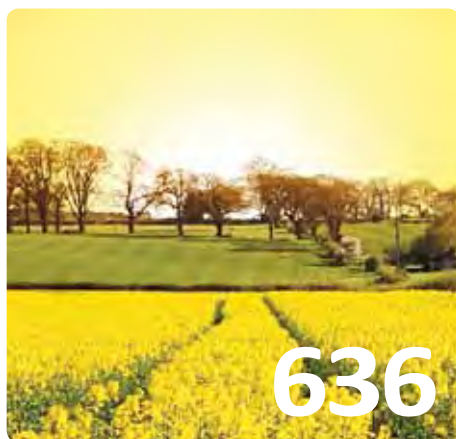
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AOCS Thomas H. Smouse

# Memorial Fellowship

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# The power of peptides

In every cell of the human body, millions of proteins are buzzing with activity. Some are massive pieces of machinery, relatively speaking, that direct chemical reactions; others compose the scaffolding that gives cells their shape; and some act as vehicles to carry products and messages throughout the body. There's no doubting their importance. But there is another class of molecule functioning in the shadow of these behemoths: peptides. The little siblings of proteins, peptides are smaller molecules composed of the same building blocks.

Despite their diminutive size—and often because of it—peptides have emerged as increasingly important biological entities capable of treating diseases, reducing inflammation, making foods more nutritious, killing microbes, and reversing aging. As scientists study the oversized properties of petite peptides, they are beginning to discover how to better isolate, study, and produce them. It is just the beginning for bioactive peptides.

- Peptides that affect how cells function—known as “biopeptides”—are an important subject of study within the field of proteomics.
- Many biopeptides have an effect on metabolism or how the body digests food, whereas others have functional properties that are useful in cosmetics.
- This article will get you up to speed on biopeptides, which are likely to become more plentiful in food and medicine as scientists discover how to better isolate, study, and produce them.

**Sarah C.P. Williams**

## BIOPEPTIDE BASICS

Proteins and peptides alike are made of strings of proteinogenic, or standard, amino acids—22 organic chemical building blocks found in the human body. Depending whom you ask, a protein—to gain its moniker—must have more than 20, 40, or 50 amino acids; an average protein in the human body, though, is much larger than this, with somewhere around 500 amino acids. A peptide is any string of at least two amino acids that has fewer than this designated cut-off—a few dozen building blocks rather than a few hundred.

The differences between proteins and peptides, while they are rooted in size, do not end with how large or small they are. Because peptides are so short, they generally do not fold into complex structures like larger proteins; instead of forming helices, sheets, large complexes, and globules, peptides remain a loose, two-dimensional string inside cells. And because of both their small size and lack of organized structure, peptides tend to be able to sneak through spaces where larger proteins can't fit—they can penetrate the walls of the intestines, human skin, and in some cases even the membranes surrounding cells. For drug makers or food engineers, this is one of the most appealing qualities of biopeptides; they can quickly get to the bloodstream to go where they are needed. In addition, short peptides can be simpler and cheaper to produce compared to complicated, larger proteins.

To make proteins, machinery inside a cell's nucleus first transcribes a strand of DNA (deoxyribonucleic acid) into a corresponding strand of messenger RNA (ribonucleic acid; mRNA). Next, in association with ribosomes outside of the nucleus, each triplet of mRNA, or codon, is translated into an amino acid that makes up a growing protein. Finally, the protein might be processed by the cell through the addition of chemical entities on the string of amino acids or the removal of sections of the strand.



When it comes to peptides, a few are produced through this classical protein-making pathway. But the vast majority come from elsewhere: the food we eat. When you drink a glass of milk or chow down a burger, your digestive system is faced with thousands of proteins from the food. In the process of digestion, many of these proteins are broken down into smaller, manageable chunks: peptides. Some of these have no immediate function—they will go on to be further processed so the body can later use their building blocks. But others are bioactive peptides; bioactive in the sense that they can cause a change to a cell's functioning. It is these that have caught researchers' attention.

## FIGHTING MICROBES

Twenty years ago, molecular biologist Maxwell Hincke of the University of Ottawa (Ontario, Canada) started studying the proteins that make up eggshells. He wanted to see whether studying how eggshells form could help inform research on teeth and bones. But he soon realized that the eggshell had even more potential than being a simple model system for calcification. Hincke recently spoke about the proteomic analysis of eggshell membranes at the 106th AOCS Annual Meeting and Industry Showcases (AM&IS) held May 3–6, 2015, in Orlando, Florida, USA.

"As the years went by, I saw that the eggshell isn't just a passive barrier," says Hincke. "If you step back, you see that the eggshell has all these mechanisms for protecting the life inside it."

His group started isolating molecules from the shells and studying their functions and discovered that eggshells—which are discarded by the ton at egg factories—are a plentiful source of bioactive peptides.

In particular, Hincke and his colleagues have homed in on eggshell biopeptides that fight microbes. "This is a completely novel source of antimicrobial peptides, so we're getting a different kind of insight than ever before," says Hincke. While all animals have proteins called beta-defensins that help fight disease,

for instance, Hincke has unearthed a new beta-defensin that is unique to eggs and may be able to treat bacterial infections that have classically resisted antibiotics.

The finding is an example of why it is so important to isolate and characterize biopeptides from new sources, Hincke says. "Nature is a huge experimental engine for developing molecules with

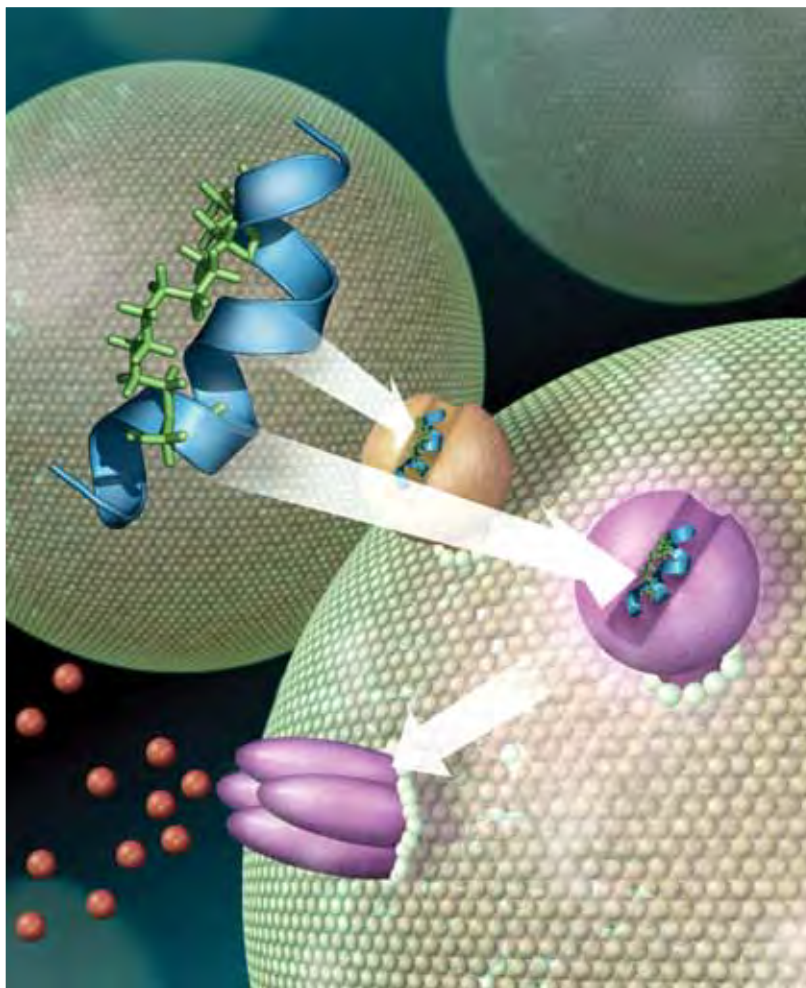
diverse properties," he says. "We can always go to the lab and manufacture synthetic peptides, but it speeds things up if first we can identify a natural mechanism [that] is the result of millions of years of evolution."

Often, Hincke says, peptides or proteins can be studied to find the shortest sequence of amino acids—within the larger molecule—that is required for something like an antimicrobial function. "One of our goals is always to define the minimal sequence activity," he says. "Then we can play around with that sequence to optimize the function."

Eggshells aren't the only place where scientists have found antimicrobial biopeptides. Hincke has now isolated some from chicken blood, and others have pinpointed them in the breakdown products of cow and goat milk, beef, and

whey. Each works a different way: some bust a microbe's outer membrane, whereas others interfere with the microbe's production of DNA or proteins. Some work against nearly all microbes, while others are more specific—a peptide from goat milk cheese, for instance, has been found to improve the symptoms of people infected with *Helicobacter pylori*.

There are likely more antimicrobial peptides that have yet to be discovered, Hincke points out. "Everything alive has to be able to resist bacterial invasion," he says. "And it's a big world out there."



**Mitochondrial apoptosis (cell death) in cancer is reactivated by targeting the BCL-2 protein family with hydrocarbon-stapled peptides.**

Credit: Eric Smith, laboratory of Dr. Loren Walensky, Dana-Farber Cancer Institute

## PEPTIDE DIET

Because many biopeptides are produced when food is broken down in the gut, it is not surprising that many of these mini-proteins have an effect on satiety, appetite, or how the body digests food. In an age of obesity, heart disease, and diabetes epidemics, it is obvious why scientists would be actively researching these effects of biopeptides.

"Dietary proteins have been known to induce satiety with different degrees but the mechanism has not been clear," says Hiroshi Hara, a professor of food science and nutritional biochemistry at Hokkaido University in Sapporo, Japan. Hara also presented at the recent AM&IS.

Often, research starts because scientists know that a food source is particularly good at making people feel full—or at lowering blood pressure or cholesterol. Then, researchers try to pin down what proteins or peptides are causing this effect.

For Hara, it began with soybeans. Researchers had shown that a soybean protein called  $\beta$ -conglycinin lowers cholesterol and decreases the incidence of atherosclerosis in mice, and suppresses hunger in humans. Hara wanted to know what shorter peptides within the  $\beta$ -conglycinin protein were responsible for the activity. So—as is standard in trying to isolate biopeptides—Hara's group digested the  $\beta$ -conglycinin protein in the lab and then tested the smaller fragments for their effectiveness at decreasing appetite in rats. The researchers identified a peptide ( $\beta$  51-63 peptide) that worked.

"Isolation and identification of the active structures in proteins like this are important to understand the mechanism," Hara says, and to work toward "application of the peptides for prevention of obesity or therapeutics of diabetes."

Hara's group is now studying peptides from other types of beans, as well as from sweet potatoes, which have been shown to temper fluctuations in blood sugar. Other researchers have found that fish and seafood are a plentiful source of bioactive peptides that modulate appetite, blood pressure, blood sugar, or cholesterol.

Toshiro Matsui, a professor of bioscience at Kyushu University in Fukuoka, Japan, has isolated tiny peptides—some only two amino acids long—from soybeans, egg whites, and teas—that apparently help prevent high blood pressure and clogged arteries. Matsui spoke about bioactive peptides at the May 2015 AM&IS.

"The advantage of peptides [over proteins] is their preferable intestinal absorption into our body," says Matsui. The peptides he has found, he believes, affect levels of calcium inside cells, which is key to how they signal. But other peptides exhibit similar effects by blocking a protein called angiotensin-converting-enzyme (ACE), causing blood vessels to relax. Matsui is still characterizing all the effects of the biologically active amino acid pairs that he has identified.

"Dipeptides could improve kidney dysfunction, suggesting that small peptides have still more unknown potential and physiological actions," he says. To better understand these roles, he has developed a method of labeling peptides to track their movement through the body.

Both Hara and Matsui say that scientists someday may be able to integrate the peptides they find into pills or even food itself to help prevent obesity and diabetes. But, as is the story for many newly discovered biopeptides, more work is needed to characterize the peptides before that can happen.

## BIOPEPTIDE BEAUTY

If you read the front of cosmetic containers—everything from anti-aging facial lotion to shampoo—you might think there is one area where biopeptides are already making a big splash in the commercial world. You will see "peptide-infused eye cream," "peptide lip therapy," and "peptide cleansing gel" at the beauty counter. But don't jump to conclusions just yet.

"In reality, most of the marketing you see for cosmetics—including claims about peptides—is exaggeration," says Perry Romanowski, a cosmetic chemist, vice president of Brains Publishing, and co-founder of the cosmetics blog The Beauty Brains. He spoke about the use and effectiveness of proteins and peptides in cosmetics at the AOCS AM&IS. In fact, he points out, if cosmetics did have bioactive peptides in them, they would no longer be just a cosmetic; they would have to be regulated by the US Food and Drug Administration (FDA) as a drug. That is because a cream or cleanser that interacts with the skins' metabolism—which a peptide would need to do to be dubbed "bioactive"—falls under the FDA's jurisdiction.

But Romanowski does admit the claims—in some cases—aren't just pulled out of nowhere. There are a number of biologically active peptides that research has shown might have anti-aging or wrinkle-removing properties. They are just not likely to be in over-the-counter creams any time soon. "It is really a gray area," he says.

Peptides advertised in cosmetics—both those that fail to provide their alleged function and those found in drugs that do—generally fall into one of a few categories. Neurotransmitter peptides, such as the botulinum toxin used in Botox™, affect the function of nerve cells and can reduce wrinkles by relaxing muscles in the face. Although Botox has been found highly effective, less potent versions of neurotransmitter peptides found in some new over-the-counter creams do not have much effect so far, Romanowski wrote in a recent blog post on peptides.

Other peptides can act as signaling molecules or enzymes, controlling the production or breakdown of larger proteins in the skin—including proteins such as collagen and elastin, which give skin its firmness. And a final set of peptides, carrier peptides, are said to transport small molecules such as copper and magnesium into the skin.

"Scientists have demonstrated through research that some of these do work," says Romanowski. A peptide called GHK-copper, for example, has been shown in the lab to make skin more firm. But whether the dose of it in your skin cream has that effect is not clear; and if it does, the FDA and other regulatory agencies around the world might be stepping in to regulate it.

Research on anti-aging peptides, Romanowski says, is moving forward particularly quickly. "I do think products are getting a bit better," he says. "But there's also a lot of fluff."

## WHERE DOES COLLAGEN COME IN?

Among the most recognizable proteins—along with insulin and hemoglobin—is collagen. Collagen is the subject of an active area of peptide research because it is a vital component of skin, tendons, ligaments, cartilage, and muscles in humans. Because of its many roles, researchers have been searching for smaller peptides, within the larger collagen proteins.

At Kyoto Prefectural University in Japan, Professor of Applied Biosciences Kenji Sato is among those isolating small peptides from collagen. He set out to see what peptides were circulating in a per-



son's bloodstream after ingesting collagen—or gelatin, the melted, unstructured form of the protein found in some foods. But he quickly ran into a problem that others have also run up against: Isolating tiny peptides from blood is tricky.

So Sato recently fine-tuned a technique to isolate the peptides he wanted to study. Using a chemical called phenyl isothiocyanate (PITC), Sato can isolate and identify the peptides that would have been missed using conventional analytical methods.

With this new technique, Sato has found almost a dozen di- and tripeptides that are present in the bloodstream after a person eats collagen. Of these, he has shown that a couple, including hydroxyprolyl-glycine and prolyl-hydroxyproline, can stimulate the growth of new skin cells. Aside from cosmetic uses (there are already collagen peptide “beauty drinks” on the market), Sato hopes that the peptides might eventually have medical use.

“I hope the collagen peptide can be used in the hospital as a ‘medical food’ to improve wound healing,” he says.

## MOVING FORWARD

Because peptides generally are the byproducts of digestion—and are, therefore, molecules that the human body has been exposed to for years—researchers hope that they will be found to be safer than new, engineered drugs that may have unexpected effects in the body. If the body has no problems digesting soybeans, they argue, then there is no reason a dose of a peptide that comes from soybeans should cause problems. But even if this is the case, biopeptides that are truly biologically active must be tested in rigorous trials to ensure that they do what they say and are safe for consumers and patients.

The good news for those who want to see biopeptides on their food and pharmacy shelves is that research on the molecules is picking up: the number of new peptides entering clinical trials each year jumped by 1,300% between the 1970s and 2000s.

Aside from further research on the peptides that have already been identified, work continues on developing new methods to characterize biopeptides by degrading proteins in new ways, separating the resulting fragments in new ways, and screening them for biological function in new ways. Moreover, scientists want to understand better what happens to peptides when they are in the body, using tracking methods such as the labeled peptides Matsui uses. Are there general rules that dictate when a biopeptide can be absorbed by the skin or the intestine, or when a peptide is further degraded instead of circulating in the body? Some guidelines on peptide properties are known, but researchers cannot yet predict the behavior of every peptide.

Every day, we ingest peptides found naturally in the food we eat and produce even more peptides made by our own bodies. So, when a product claims it contains peptides, it is nothing unusual. But, in the future, bioactive peptides may be even more plentiful in the food supply and medicine cabinet. It will just take more research to get there.

*Sarah C.P. Williams is a freelance science writer who covers biology, chemistry, and medicine for magazines, newspapers, websites, and institutions around the globe. Her work has appeared in the Los Angeles Times, Science, Nature Medicine, and New Scientist, among many other publications. She can be contacted at sarahcpwilliams@gmail.com.*

## A sampling of peptides being researched

| Peptide Name                                       | Source  | Application                    |
|--|---|--------------------------------|
| Lunasin  | Soybean, barley, wheat, rye                   | Anticancer                     |
| VSVFL  | Canola/rapeseed                               | Antihypertensive               |
| WNI/LNA NI/LDTDI/L                                 | Flaxseed                                      | Antihypertensive               |
| FCVLRP   | Marine shrimp ( <i>Acetes chinensis</i> )     | Antihypertensive               |
| MEGAQEAQGD   | Sea cucumber ( <i>Acaudina molpadioidea</i> ) | Antihypertensive               |
| Leu-Gln-Pro-Gly-Gln-Gly-Gly-Gln-Gln                | Sardine muscles                               | Antioxidant                    |
| Tyr-X-Phe-Leu-Gly-Leu-Pro-Gy-X-Thr                 | Cow placenta                                  | Immune Boosting                |
| Pro-Hyp  | Collagen                                      | Wound Healing and Inflammation |
| Ala-Phe-Asn-Ile-His-Asn-Arg-Asn-Leu                | Shellfish ( <i>Mytilus coruscus</i> )         | Anticancer                     |
| GLSDGEWQ   | Beef  | Antibacterial                  |
| Tyr-Pro-Phe-Pro-Gly-Pro-Ile-Pro-Asn-Ser-Leu        | Cow's milk                                    | Opioid agonist (pain relief)   |
| Pal-Lys-Thr-Thr-Lys-Ser (Palmitoyl pentapeptide-4) | Collagen                                      | Antiwrinkle                    |

**The biopeptides that researchers around the world have isolated come from diverse sources. Those listed here are all still under investigations for their potential to affect health.**

# Oleaginous yeast convert glycerol to triacylglycerol

Hiroshi Kurokawa

- Glycerol is a byproduct of biodiesel production, but glycerol derivatives are also key compounds of chemical synthesis.
- Researchers at the Lion Corporation in Tokyo, Japan, recently developed a strategy that uses oleaginous yeast to convert glycerol to oil.
- The fatty acid chain length distribution and physical properties of the oil are similar to those of palm oil, indicating that the oil could be used as an alternative to vegetable oil.

In 2050, the world's population is predicted to reach 9 billion people, and a food shortage is predicted. Accordingly, research to diversify inedible oil sources for industrial applications and fuel has been progressing. Recent green technology studies of biomass utilization have shown that oleaginous yeast is a leading candidate as an alternative to vegetable oil as a raw material for the surfactants used in detergents and biofuel production.

Meanwhile, research into petroleum-alternative fuels, such as biodiesel, is actively being conducted. Biodiesel is produced by the transesterification of vegetable oil and methanol, and glycerol is a key byproduct. Biodiesel is expected to be a renewable energy source, and expansion of its production is predicted. One way to prevent this expansion from impacting the food supply is to produce biodiesel from used cooking oils, chicken fat, brown grease, gutter oil, and other waste oils.

Hiroshi Kurokawa, a researcher in the Functional Material Science Research Laboratories at the Lion Corp. (Tokyo, Japan) and Takafumi Naganuma at the University of Yamanashi (Kofu, Japan), have taken a different approach that focuses on the glycerol that is generated as a byproduct of biodiesel production from vegetable oil. Glycerol derivatives have been studied as key compounds of chemical synthesis.

Using such studies as a starting point, Kurokawa and Naganuma, have developed a strategy to convert glycerol to oil using "oleaginous yeast" by way of a glycerol byproduct.

The yeast produces oil by using carbon sources such as glucose, xylose, and glycerol, among others. The yeast can accumulate triacylglycerol to an amount of up to 50% of its cellular dry weight. Thus, the oil content of the yeast is superior to that of various food crops. Efficient oil production can be realized by combining the use of yeasts, carbon sources, and appropriate fermentation conditions. In this way, the yeast can produce oil with properties similar to vegetable oil, and the yeast-produced oil can be considered to be non-edible oil materials that do not compete with food. To practically realize the production of such non-edible oil sources, it is first necessary to improve the total oil production volume and productivity rate.



Therefore, Kurokawa and colleagues studied the appropriate fermentation conditions required to produce high oil production from glycerol using the yeast strain *Lipomyces starkeyi* CBS 1807 (Fig. 1), which is the type strain of the species. To establish a high-density culture, the researchers evaluated the effect of the nitrogen source on cell growth and oil content as well as the effect of glycerol concentration on the total oil amount. The control of the nitrogen source was an important factor to consider, since it promotes a phase change in the yeast: The presence of a nitrogen source promotes cell growth, and nitrogen deficiency promotes oil accumulation.

On the other hand, there is a positive correlation between glycerol consumption and total oil production, irrespective of culture conditions. Therefore, it is necessary to increase glycerol consumption to increase total oil production. However, glycerol consumption is inhibited by high initial glycerol concentrations with regards to consumption speed. Therefore, the control of low glycerol concentration would accelerate glycerol consumption.

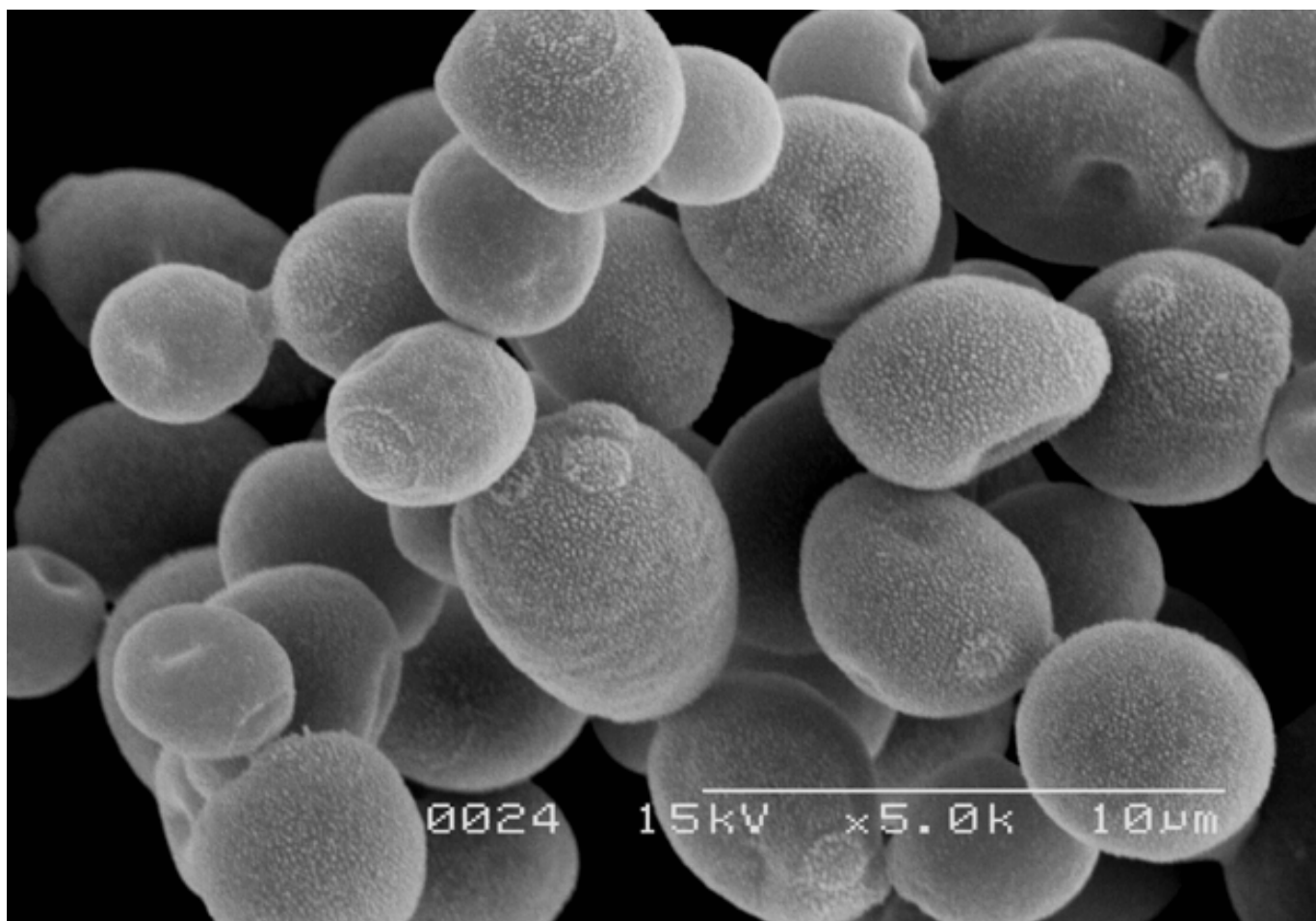
Based on the fundamental studies conducted to date, high-cell density culture fermentation was conducted in a five-liter bioreactor, known as a jar fermenter. As a result, the total oil production volume and productivity rate were approximately 60 grams/liter and 0.45 grams/liter/hour, respectively. To consider the potential commercialization of this oil, a 90-liter pilot-

scale fermentation experiment was conducted. As a result, the oil productivity was equivalent to that obtained at the five-liter scale. The researchers also confirmed the fatty acid chain length of the yeast-produced oil. The chain length distribution was mainly 32.8% palmitic acid and 51.1% oleic acid, which was similar to that of palm oil. Other physical properties were also similar. Therefore, the yeast-produced oil can be used as an alternative to vegetable oil.

Moving forward, the researchers will continue to improve the production speed and to utilize crude glycerol as a raw material. If it is possible to use crude glycerol, the total cost can be reduced. However, organic impurities in crude glycerol inhibit yeast growth. If this problem can be resolved, it will facilitate the practical use. The researchers have over 400 wild *Lipomyces* strains, and are continuing to screen and breed them.

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*Hiroshi Kurokawa is a researcher in the Functional Material Science Research Laboratories at the Lion Corp. in Tokyo, Japan. He joined Lion after graduating in 2007 from the Department of Chemistry, School of Science and Engineering at Waseda University in Japan. For the past several years, he has been searching for a new oil source that is an alternative to vegetable oil.*



**FIG. 1.** The yeast strain *Lipomyces starkeyi* CBS 1807 10μm at 5,000 X magnification

# Enhancing stability of olive oils by enrichment with natural ingredients

**Antonella Cavazza, Paola Salvadeo, Carmen Laganà, Stefania Corti, Claudia Mancinelli, and Claudio Corradini**

Olive oil is an important component in the Mediterranean diet. Several positive health benefits are attributed to the oil, but its shelf life is limited since the bioactive compounds in the oil degrade during storage, which diminishes its organoleptic and healthful properties. For these reasons, the control of olive oil shelf life is an important area of research, and some recent studies have also used mathematical modeling to predict it. The main effects reported to occur in olive oils during storage are linked to the lipidic and polyphenolic fractions, and involve oxidative and hydrolytic degradation of several compounds.

- **The healthful properties of olive oils strongly decrease during storage due to oxidative and hydrolytic degradation of bioactive compounds.**
- **Aromatic plants, such as rosemary and oregano, and spices such as red chilli pepper powder are rich in compounds with strong antioxidant properties.**
- **A screening of olive and extra-virgin olive oils enriched with small amounts of essential oils or red chilli pepper powder was performed through a rapid analysis that measures oxidative stability without preliminary fat separation. Results showed an enhancement of shelf-life in all experiments.**

The oxidative stability of an olive oil seems to be highly correlated to its initial content of natural antioxidants.

Consequently, the addition of natural substances has been proposed as a way to protect foods from deterioration and to prolong their shelf life. Indeed, there is a historical basis for this approach as aromatic plants, which contain essential oils with important antimicrobial, antioxidant, and other biological activities, have been used since ancient times as natural preservatives in food flavorings, pharmaceuticals, cosmetics, and perfumes.

Essential oils from oregano and rosemary, for example, have been widely used in such diverse types of food products as cheese, olive oil, and mackerel oil, where they provide higher resistance to oxidation and increased thermal stability. Oregano contains high percentages of the phenols carvacrol and thymol (known for antimicrobial and antioxidant activity) as well as the monoterpene hydrocarbons p-cymene and c-terpinene, which are flavor and perfume ingredients. The flavonoids of oregano are also a group of compounds with antioxidant activity. Rosemary essential oil contains carnosic acid, carnosol, and rosmarinic acid, which impart effective antioxidant properties.



Spices have also been proposed as a way to protect food from oxidation. In particular, red chilli pepper powder (*Capsicum* ss. vv.) is a remarkable source of antioxidants, including flavonoids (quercetin, luteolin), phenolic acids, carotenoids (carotene, capxanthin, zeaxanthin), and vitamins (vitamin A, ascorbic acid, and tocopherols). The major pungent components of red chilli pepper powder are capsaicinoids. These compounds exhibit an interesting antioxidant activity, which has been found to prevent the oxidation of oleic acid at cooking temperatures as well as the formation of lipid hydroperoxides from the autoxidation of linoleic acid.

Scientists in the Department of Chemistry at the University of Parma (Parma, Italy) and Velp Scientifica (Usmate, Italy) recently conducted a screening to evaluate the effects of adding various natural products known for their antioxidant properties to virgin olive and extra-virgin olive oils. Two essential oils (rosemary and oregano) were added to olive oil, and two spices (sweet and hot red chilli pepper powders) were added to extra-virgin olive oil (Fig. 1).

The degree of lipid oxidation of an oil can be measured by chemical or physical methods (direct methods) that are often

time consuming, expensive, and require trained people. The level of oxidation of fatty foods can be also determined with indirect methods through tests which measure the fatty food's stability under accelerated conditions (such as elevated temperatures) that allow autoxidation measurements to be obtained in a few hours instead of weeks or months. Generally, these accelerated tests provide an oxidation curve, characterized by an Induction Period (IP), which measures the time required to reach the starting point of oxidation that corresponds either to a level of detectable rancidity or a sudden change in the rate of oxidation.

In this screening, the oxidative stability of oil samples—both at the beginning and during storage—was investigated by accelerating the oxidation process through the use of Velp Scientifica's Oxitest reactor, an instrument that speeds up the oxidation process based on two accelerating factors: temperature and oxygen pressure. Compared to similar and conventional technologies, the Oxitest reactor is easy to use, as it allows the analysis to be performed directly on the food (liquid, solid, or pasty) without previous fat separation.



**FIG.1.** Olive oil and an example of essential oil bordered by three sources of essential oils: red chili pepper, rosemary, and oregano



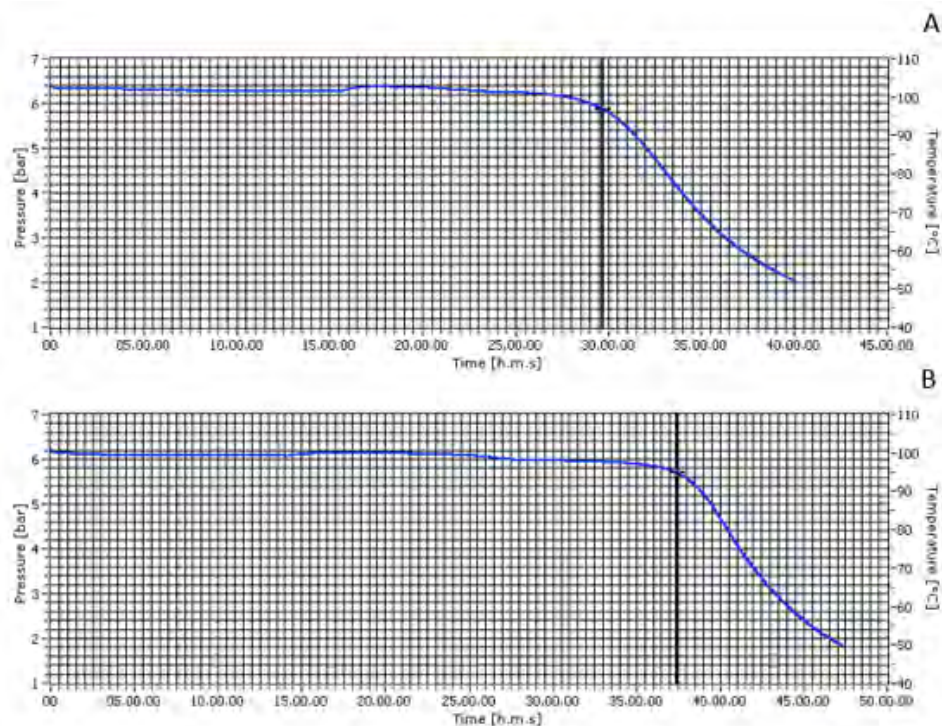
**FIG. 2.** Oxitest reactor

The instrument, shown in Fig. 2, measures the absolute pressure change inside two independent, closed, and thermostated chambers, monitors the oxygen uptake by reactive components in the samples, and automatically generates an Induction Period (IP) value (Maiocchi, P. and U. Bersellini, 2007). The longer the IP was, the higher the stability against oxidation over time.

A series of preliminary experiments indicated that the quantities sufficient for observing a significant effect on stability were 1% w/w for red pepper powder and 3% w/w for essential oils, respectively.

Oil stability was measured on the blank oils (to which nothing was added) and on enriched oils just after the adding the natural product, to evaluate a possible immediate effect in protection from oxidation. Measurements of the extra-virgin oil samples were repeated after aging them in a dark room at room temperature for one year.

Fig. 3 is a graph of induction time obtained for blank olive oil (A) and for olive oil enriched with rosemary essential oil (B), where the difference in the flex point of the curve can be seen.



**FIG. 3.** IP of blank olive oil (A) and olive oil samples enriched with 3% of rosemary essential oil (B)

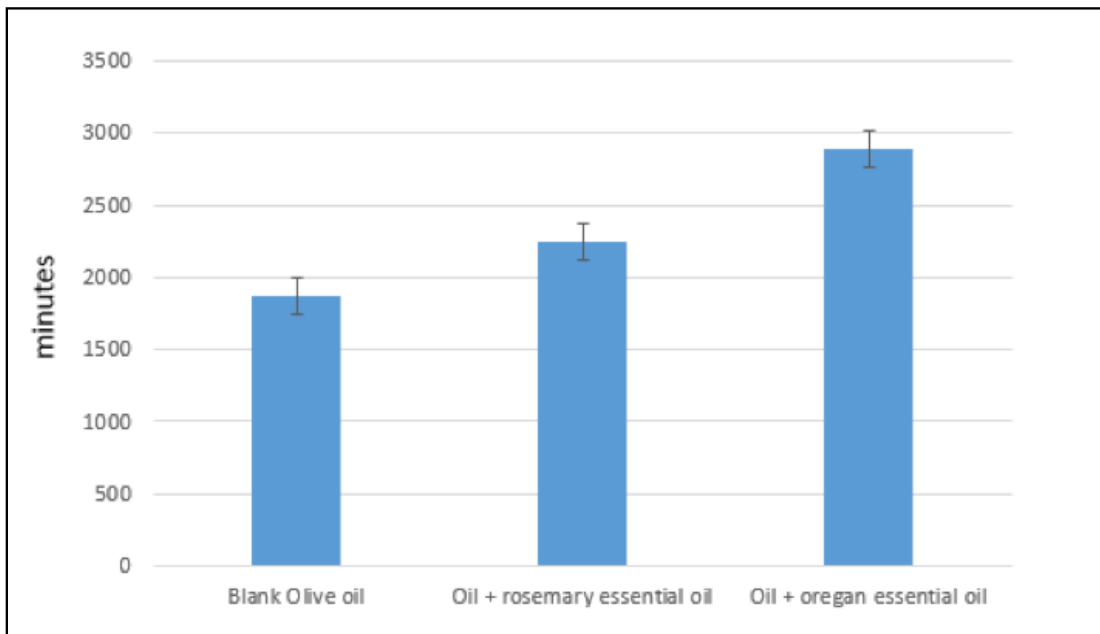


Results about samples examined are reported in Figs. 4 and 5. All samples enriched with essential oils and spices showed a longer IP than blank oils, thus confirming the antioxidant activity exerted by the natural product that was added.

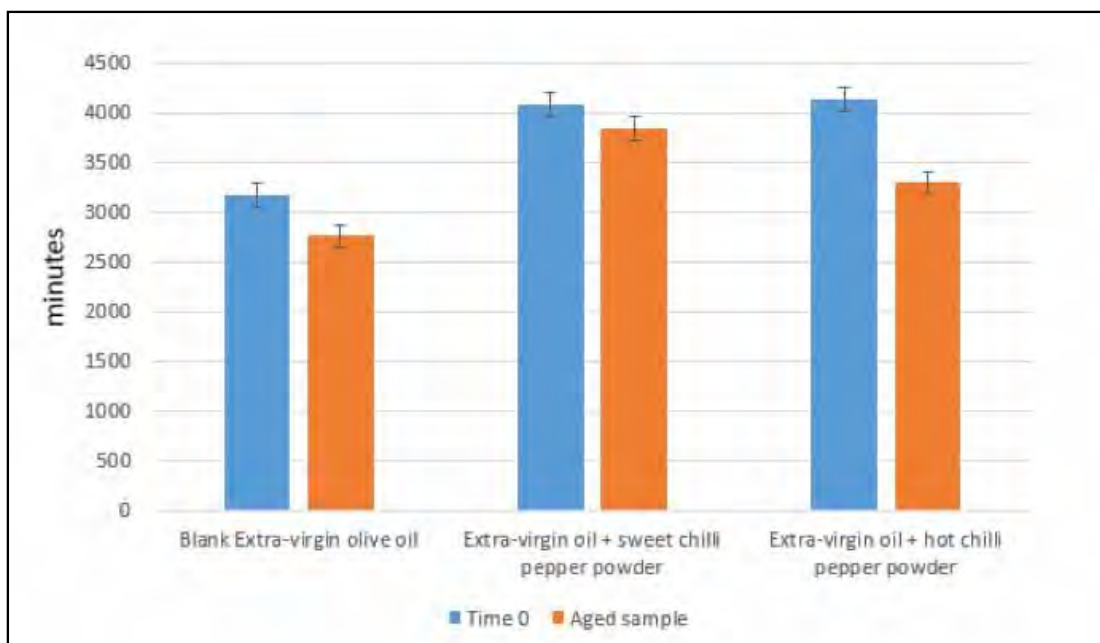
Previous studies had shown rosemary to be more effective than oregano in stabilizing olive oil—most likely because oregano essential oil has a higher content of volatile antioxidants, such as carvacrol, which may have partially evaporated during storage (Antoun *et al*, 1997). However, in our study, the evaporation of volatile components of oregano essential oil did not affect the

final result, and oregano demonstrated stronger antioxidant activity than rosemary oil. This is most likely because the experiments were performed in a closed thermostated chamber, where any evaporated volatile compounds would remain in contact with the oil sample. Thus, minimizing evaporation as a factor provided a more realistic evaluation of the effects exerted by the essential oils.

In measuring the stabilizing effects of the red pepper powders that had been added to the extra-virgin oil, we observed that the increase in IP became more pronounced after aging, suggesting



**FIG. 4.** IP (minutes) of blank olive oil and olive oil samples enriched with 3% of essential oil



**FIG. 5.** IP (minutes) of blank extra-virgin olive oil and extra-virgin olive oil samples enriched with 1% of spices

# Upgrading glycerol for high-value-added applications

Franck Dumeignil

Since the biodiesel boom began in about 2005, the glycerol supply has increased considerably, from about 0.5 million cubic meters (Mm<sup>3</sup>) in 2005 to about 2.5 Mm<sup>3</sup> in 2013, due to glycerol being a byproduct of biodiesel production through the transesterification of triglycerides.

- The smart and efficient conversion of glycerol to high-value-added chemicals is key to ensuring the sustainability of biodiesel production.
- In a true biorefinery approach that maximizes profitability, using cheap crude glycerin in a chemical process is not necessarily less costly than using more expensive refined glycerol; using intermediate qualities can be a profitable trade-off.
- The deactivation of catalysts during coking can be tackled from different angles, by fine-tuning the chemical properties of the catalysts and/or by designing a smart adapted process.

More than 2,000 possible applications of glycerol are reported in the literature. As a pure refined compound, glycerol tastes like sugar, which makes it, for example, a good sweetening agent in toothpastes. It can also be chemically upgraded to yield large-volume compounds such as acrolein and its derivatives. However, although several advanced glycerol processes have been developed on a laboratory/demonstration scale, few have reached commercial-scale production. One exception is the epicerol® process Solvay developed to produce epichlorohydrin from glycerol. This process is actually an inversion of an initial process that was developed for producing glycerol from epichlorohydrin when glycerol was in short supply (<http://tinyurl.com/nlahny2>).

The commercialization of other advanced glycerol processes is hampered by several factors. One is uncertainty regarding the glycerol supply caused by unpredictable fluctuations in regulations. The revised incorporation rate for biofuels in Europe, which was reduced from an initial target value of 10% by 2020 to 7%, has had repercussions throughout the biodiesel sector. Several existing units in France have closed, and some newly built units in Germany have postponed or discontinued operations. Such fluctuations are a threat to future investments in such projects.

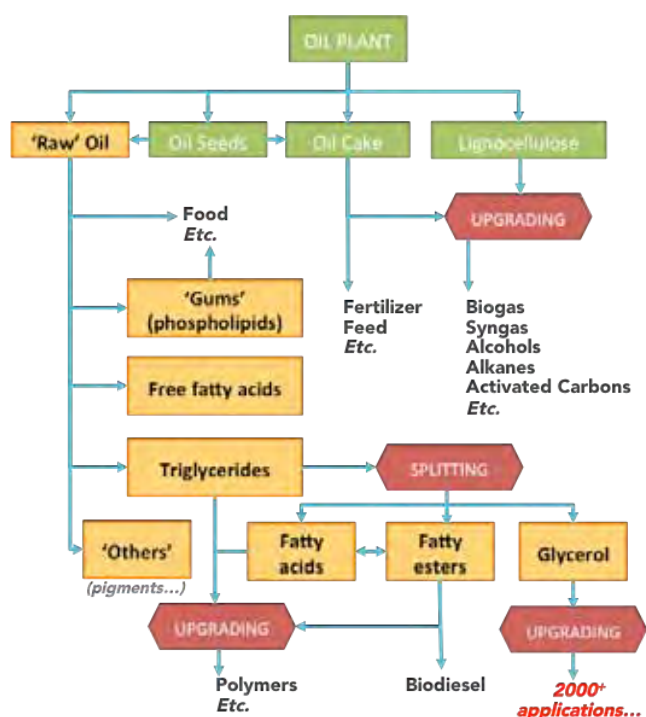
Other hurdles to commercialization include the high cost of purifying the “dirty” glycerol obtained from the transesterification process and the stability of the heterogeneous catalysts used for gas-phase glycerol upgrading. Such catalysts are quickly deactivated by coking (the formation and deposition of undesirable carbonaceous compounds on the catalyst’s surface), making their industrial applicability questionable.

The following is a global perspective of these factors and possible solutions, based on work done by our research group.

## BETTER ECONOMICS THROUGH DIVERSIFICATION AND INTEGRATION

The commercial challenges described above arise from the historical singular function of biodiesel units, which were exclusively dedicated to biofuels production. The downside of this approach is that biofuels must be cheap to be acceptable to the market, and competitive cost pressures result in low margins and an overall economy that is largely dependent on government incentives and subsidies. In contrast, about 90% of the raw materials used in the





**FIG. 1.** Schematics of a plant-oil-biorefinery value chain that integrates fuel and chemicals production

petroleum industry (namely oil) is used to produce fuels and the remaining 10% or so to produce high-value chemicals that account for approximately 90% of the industry's overall profits. Believing that an industrial unit will survive only on biofuels production is thus an illusion that will undoubtedly sooner or later fade away.

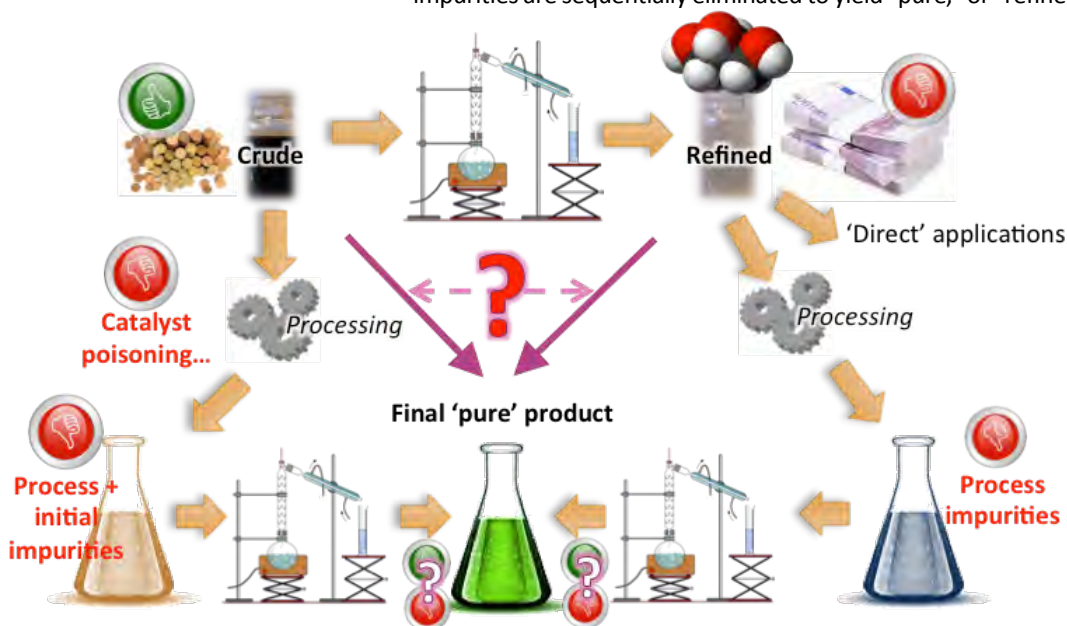
Diversifying outputs could open the door to many business opportunities. Such a biorefinery approach could, in its most evolved configuration, simultaneously accept multiple types of biomass, treated by multiple processes, to generate multiple products. Specifically concerning the topic of the present article, this approach, as outlined within the framework of the EuroBioRef PCRD7 European program ([www.eurobioref.org](http://www.eurobioref.org)), seeks to

optimize the value of glycerol by streamlining an additional chemical production unit with a biodiesel unit. This can be further extended to vegetable oil (and also animal and used oils) biorefineries that could produce high-value-added products from fatty compounds, glycerol, and any other coproducts along the value chain (Fig. 1).

My research team is currently involved in the large-scale PIVERT (Institute for Energetic Transition) project, which aims to design a plant-oil biorefinery through such an integrated and diversified concept (<http://www.institut-pivert.com/?lang=en>). Note that plant-oil-derived fatty compounds are already used to make polymers. For example, both Arkema and Solvay use ricinoleic acid from castor oil as a raw material to produce RILSAN® (<http://www.rilsan.com/en/>) and Technyl® eXten (<http://www.technyl.com/en/solutions/featured-product/technyl-exten/index.html>), respectively, as commercial products with superior technical properties. Extending this concept to a variety of chemicals and other value-added products, such as activated carbons (as exemplified in the EuroBioRef project), would provide the basis for a product line that, together with conventional fuel applications, would make the whole integrated process more economical.

## GLYCEROL VS. GLYCERINE

As mentioned previously, the biodiesel industry is the main source of glycerol today. However, the glycerol produced in a biodiesel unit is a dirty, complex mixture containing methanol, organic compounds, salts, base residues, and other impurities. Known as "crude glycerol," or "glycerin," this mixture is often subjected to a downstream multiple-step purification process in which the impurities are sequentially eliminated to yield "pure," or "refined,"



**FIG. 2.** Should I use crude or refined glycerol in my process to make it more profitable? The answer is not straightforward: The best option will most probably consist on a trade-off between an acceptable degradation in chemical catalytic performances in the presence of impurities and the easiness of separation of the desired final product(s) to get the required final quality at reasonable costs.

commercial glycerol. Unfortunately, this extensive purification process has a significant cost that affects the cost of the final product obtained through subsequent chemical conversion. It is worth mentioning that some research efforts are currently exploring alternative biodiesel production processes that would directly generate “pure” glycerol as a co-product, but such technologies are not likely to be implemented soon, and meanwhile we must cope with substantial amounts of crude glycerol.

The advantageous price of crude glycerol compared to that of pure glycerol (which costs three- to five times as much) raises the question of whether it is possible to design chemical conversion processes that are tolerant to the impurities present in crude glycerol streams. The answer to this question is not straightforward for the reasons illustrated in Fig. 2 (page 625).

Even if the downstream application of the final desired product is unaffected by the presence of impurities, which is highly unlikely, a purification step would still be needed after the chemical conversion—not only to separate the byproducts from the upgrading technology, but also to eliminate any unaltered or converted initial impurity.

In the case of a liquid phase process, even if an operator chose to perform only one global purification at the end of a procedure using crude glycerol as a raw material to eliminate all the impurities in one shot, it is most likely that the catalyst used for the chemical upgrading would not tolerate some of the impurities present in the initial stream. This would degrade chemical performances and yields, burdening the overall efficiency of the approach.

One intermediate solution, if technically possible, would be to define, generate, and use intermediate qualities of glycerol (“semi-refined” glycerol) in which only the impurities harmful to specific catalysts are removed prior to the reaction—or, alternatively, to design catalysts that would be more tolerant to such impurities.

Our research group recently evaluated the effect of various glycerol impurities on the performances of model catalysts used for glycerol liquid phase partial oxidation to high-value-added compounds. The catalysts we examined consisted of monometallic particles of Pt, Au, Pd, and Ag supported on alumina. In all cases, we observed that the matter organic non glycerol (MONG) stuck to the catalyst and blocked the active sites, causing further foaming in the presence of a base. The initial catalytic performances could be recovered by washing the catalyst with inorganic solvents, which sufficiently flushed out the MONG. However, in designing a sustainable process, MONG should obviously be removed from the initial stream.

In contrast, the presence of residual methanol from the transesterification process was observed to be beneficial, as it facilitated the smooth dissolution of the oxidant (molecular oxygen) from the gas phase to the reactive liquid medium, thereby enhancing the oxidation process in the liquid phase. The base residues remaining after transesterification also had a positive effect. This was not surprising given that basic conditions are usually beneficial to the glycerol liquid phase partial oxidation reaction.

The presence of inorganic salts that arise from the neutralization of the homogeneous base used in the transesterification process did not have any significant effect on the reactivity of the catalysts. However, we more recently identified trace amounts of sulfur compounds (the nature and origin of which are still unclear) in the streams that had

a negative effect. Our findings also indicated that some trace metals could also have an effect on the reactivity of the system, but more research is needed to clearly unravel the nature and extent of their action. Finally, among those catalysts we studied, the Pd-based one was the most tolerant to the glycerol impurities. Moving forward, it would be interesting to investigate the effects of impurities on bimetallic systems and/or when using alternative supports (other than alumina), as this might help us identify even more tolerant catalytic formulations.

In designing an optimal operation, such considerations and parameters could ultimately be used to identify the best tradeoff, requiring process designers and reaction developers to work together to choose the best combination of purification options, catalytic activity, and stability to meet required technical specifications (per customers and regulations) and product price point.

Although such considerations are useful for liquid phase processes, they are less relevant for gas phase processes, as vaporizing the impurities together with glycerol to feed a continuous reactor would be impractical due to the heterogeneity of the stream. However, some combined processes that accept crude glycerol as a raw material using specifically designed evaporation/separation devices to selectively bring glycerol vapors to the catalytic conversion reactor have been reported (Dubois, J. –L. (Arkema), WO 200812908, 2008, and Kijenski, J., *et al.*, (Inst. Chemii Przemyslowe), EP 1860090, 2007.)

## HETEROGENEOUS CATALYSTS COKING

Heterogeneous catalysts coking is a common issue when addressing gas-phase upgrading of biomass-derived platform molecules. Biomass-derived oxygenated compounds have a high reactivity, which can lead to uncontrolled polymerization reactions between chemical intermediates. Such reactions produce carbonaceous compounds, known as “coke,” that chemically bind to the surface of the catalysts, blocking the active sites and drastically degrading catalytic performances. Another complicating factor to consider is that pure glycerol starts to decompose at about 300 °C, while glycerol atmospheric pressure gas-phase conversion processes typically take place at temperatures between 250 and 350 °C. Nevertheless, glycerol is conventionally introduced as an aqueous solution in the reactor, as it would be too challenging to attempt vaporization of this highly viscous compound under its non-diluted pure form.

Roughly, two strategies for coping with catalyst deactivation issues were proposed in the academic literature and in patents:

1. introducing small quantities of oxygen in the feed, either continuously or in pulses, with optional introduction of an oxygen-splitting functionality to the catalyst to facilitate continuous elimination of the coke by “burning.”
2. using a fluid catalytic cracking-like (“FCC”) process in which the catalyst is continuously circulated between two reactors. The reaction takes place in the first reactor, while regeneration by coke burning takes place in the second.

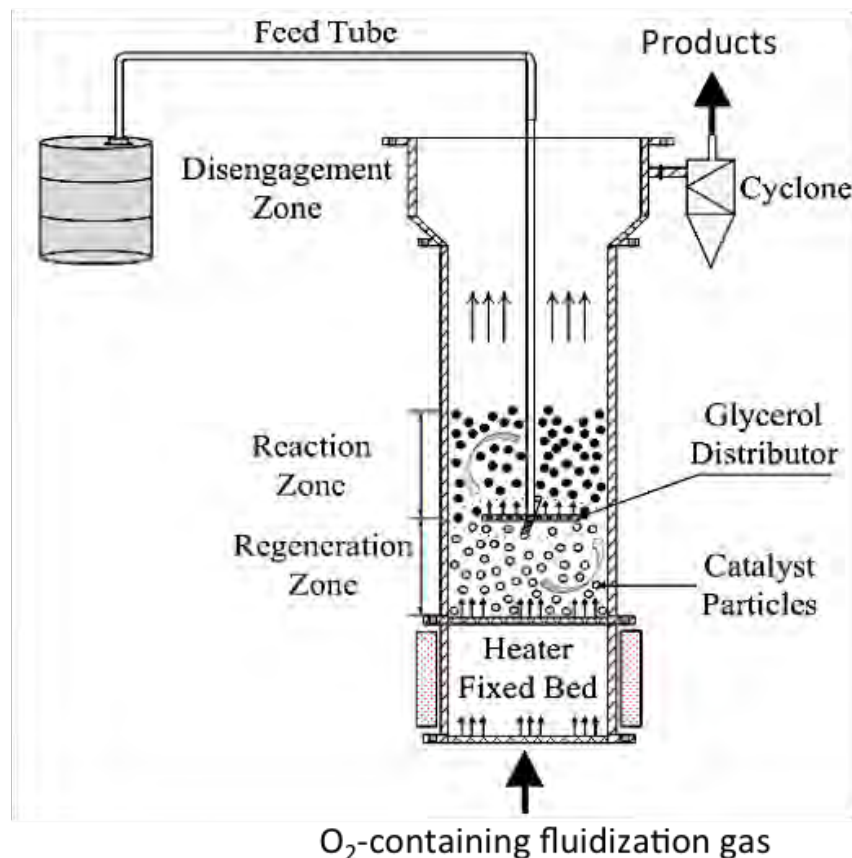
Our research team has developed two additional strategies that can be applied to the conversion of glycerol to acrolein, which is an important intermediate in the chemical industry. The first strategy involves fine-tuning the catalyst’s acidity—a critical parameter for the conversion reaction. Indeed, the catalyst must be sufficiently

acidic to enable the glycerol double dehydration reaction to acrolein, but not too acidic, as that would promote the undesired formation of cokes.

The required balance was achieved by designing a composite catalyst based on a rather conventional acidic active phase known as silicotungstic acid, which we supported on an original carrier composed of  $\text{ZrO}_2$  (zirconia) nanocrystallites dispersed in a nano-designed honeycomb-like structure of silica (called an "SBA-15"). The silica structure was designed to be resistant to the hydrothermal conditions under reaction, and its pore-size diameter was set at an optimal value for avoiding the confinement effects that would lead to undesirable side-coking reactions. The use of an SBA-15 as a host matrix for the  $\text{ZrO}_2$  support further enables conferring to the whole catalytic system mechanical properties that  $\text{ZrO}_2$  alone, while being a very good support from a chemical point of view for this reaction, could not afford. With this innovative composite catalytic system, we could significantly extend the life of the silicotungstic acid active phase, by substantially limiting its coking.

The second strategy we used was based on continuous regeneration in a specifically designed single reactor. It consists of a two-zone fluidized bed reactor (TZFBR; Fig. 3), in which the catalyst is continuously mixed (fluidization) by an upflow gas stream. A glycerol cane is plunged into the middle of the reactor from above. In the process we designed, the fluidization gas contained oxygen to regenerate the catalyst. To avoid oxidation of the reactants and products as well as creation of explosive atmospheres, the system must be perfectly controlled so that all of the oxygen is consumed just before the place where glycerol is introduced. With an optimized system, we were able to substantially extend the life of a conventional silicotungstic acid catalyst directly supported on a silica (without  $\text{ZrO}_2$  modification), thus proving our concept.

There is still work to do, and we are enhancing these technologies in the lab and at demonstration scale with industrial partners. We have also designed an efficient liquid-phase continuous process with Japanese partners; are working with Philip Xiaofei Ye, a professor at the University of Tennessee (Knoxville, Tennessee, USA) to develop an alternative catalysts regeneration technology based on cold plasma; and have designed a new glycerol



**FIG. 3. Schematic principle of the two-zone fluidized bed reactor (TZFBR) used for continuous regeneration of the catalyst**

value chain based on allyl alcohol from glycerol that presents many advantages compared to the conventional value chain mimicking the petroleum one (patents filed).

Many groups in the World are working on new technological solutions, and it is only a matter of time before novel technologies for increasing the value of glycerol hit the market and generate a new and more profitable value chain interwoven with biodiesel industry.

*Franck Dumeignil, is a full professor at the University of Lille, France, and Deputy Director of UCCS, UMR CNRS 8181. He coordinated the large-scale FP7 EuroBioRef project and is currently involved in biorefineries-related national projects, including PIVERT and REALCAT. He can be contacted at [franck.dumeignil@univ-lille1.fr](mailto:franck.dumeignil@univ-lille1.fr).*





# Antioxidants from soy hulls

Latoya Whitley, Rachael Woods, and Uruthira Kalapathy

- Soy hull is widely available and contains substantial amounts of polyphenols.
- These polyphenols are challenging to extract because they are covalently bound to cell wall polysaccharides and require harsh pretreatments.
- Our research group at Claflin University in Orangeburg, South Carolina, USA, demonstrated that extracting the polyphenols at room temperature simplified the processing requirements, which could make soy hull extract an attractive alternative to synthetic antioxidants.

Soy hull, rich in dietary fiber, makes up about 8% of the soybean by dry weight. Substantial amounts of polyphenols are found to be covalently bound to cell wall polysaccharides in the hulls, and hence require harsh pretreatments prior to extraction.

Previous research conducted in our laboratory showed that polyphenols from soy hull can be extracted from soy hull using 70% ethanol at 70–80°C (1). However, a higher temperature could cause undesirable chemical changes and may not be economically viable. The study described in this article demonstrated that polyphenols can be extracted at room temperature from soy hull to avoid multiple filtrations.

Previous research conducted in our laboratory showed that polyphenols from soy hull can be extracted from soy hull using 70% ethanol at 70–80°C (1). However, a higher temperature could cause undesirable chemical changes and may not be economically viable. The study described in this article demonstrated that polyphenols can be extracted at room temperature from soy hull to avoid multiple filtrations.

## EXTRACTION OF POLYPHENOLS

Soy hull samples were procured from Carolina Soya LLC (Estill, South Carolina, USA). These samples were ground in a household blender for 1–2 min at maximum speed and then transferred to a coffee grinder for further grinding (2–3 min) until a fine powdered form was obtained. All extractions were performed using 1:5 ratio of mass (g) of soy hull to volume (ml) of solvent. Based on the previous work, 70% ethanol was used as the extraction solvent. All samples were placed in a mechanical shaker at 200 rpm during incubation. Three sets of extractions were performed as shown in Table 1. The filtrates were separated after incubation by vacuum filtration and stored at 4°C until further analysis.

## QUALITATIVE ANALYSIS OF SOY HULL EXTRACT BY FTIR

To confirm the presence of phenolic compounds in soy hull extracts, a portion of the soy hull extracts were adsorbed onto dried silica by incubation of 1 g dried silica in 25 ml of soy hull extract for 30 min in a mechanical shaker, followed by air drying under a fume hood. The adsorbed silica was then mixed with potassium bromide to give a 2% silica mixture and analyzed using Diffuse Reflectance Infrared Fourier Transform (DRIFT) spectroscopy. The DRIFT spectrum of the extracts was also compared with that of 100 ppm catechin.

## TOTAL PHENOLIC CONTENT DETERMINATION

The total phenolic content was determined using the Folin-Ciocalteu method (2) and standard catechin solutions in 70% ethanol. Spectrophotometric absorbance of reaction mixtures at 726 nm was used to generate a catechin calibration curve and determine total phenolic content in soy hull extracts expressed as catechin equivalents (mg of catechin/g of sample). All quantitative analyses were performed in triplicates and the averages of triplicate measurements along with standard deviations were reported.

## ANTIOXIDANT CAPACITY BY ORAC ASSAY

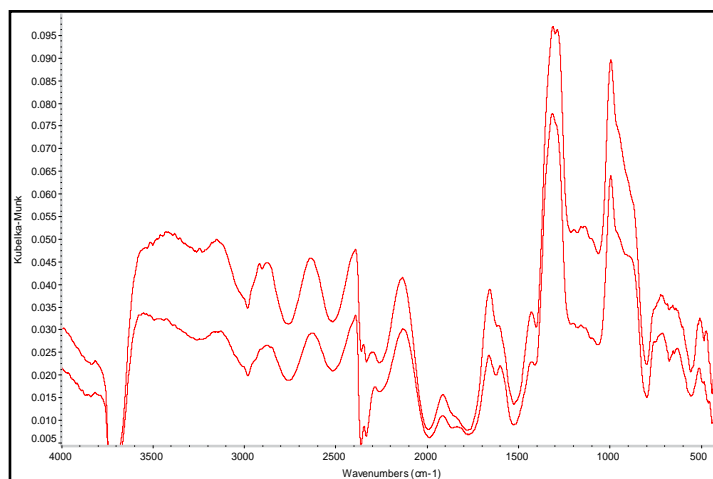
The oxygen radical absorbance capacity (ORAC) assay involves hydrogen atom transfer reactions where the antioxidant donates a hydrogen atom to the free radical thus inhibiting or retarding oxidation. The ORAC assay requires three components: a synthetic free radical generator, a fluorescent probe, and an antioxidant (3). In the present study, fluorescein was used as a fluorescent probe to measure antioxidant capacity of soy hull extract in comparison to the refer-

ence compound Trolox. The free radical generator was 2, 2'-azobis 2-methylpropionamide, dihydrochloride (AAPH). The fluorescence intensities of the reaction mixtures were measured at 1 min intervals for 30 min using an excitation wavelength of 495 nm and an emission wavelength of 520 nm. The fluorescence intensities were normalized and represented graphically using Excel®. The final ORAC values were determined using the area under the curve (AUC). The relative ORAC value was calculated by the following equation expressed as Trolox equivalents:

$$\text{The relative ORAC value} = [(AUC_{\text{Sample}} - AUC_{\text{Blank}}) / (AUC_{\text{Trolox}} - AUC_{\text{Blank}})] \times (\text{Trolox concentration} / \text{Total Phenolic content in the extract}).$$

## RESULTS AND DISCUSSION

Fig. 1 displays the DRIFT spectra obtained for soy hull extract and 100 ppm catechin. Both spectra share similar peaks at positions 1,000; 1,310; 1,665; 1,910; and 2,130  $\text{cm}^{-1}$ . The peaks at 1,000; 1,310; and 1,665  $\text{cm}^{-1}$  were due to C-O stretching, O-H bending, and C=C ring stretching vibrations, respectively. The bands at 1,910 and 2,130  $\text{cm}^{-1}$  were due to overtones and combination bands. The similarity in peaks in the two spectra was evident and confirmed the presence of phenolic compounds in the soy hull extract.



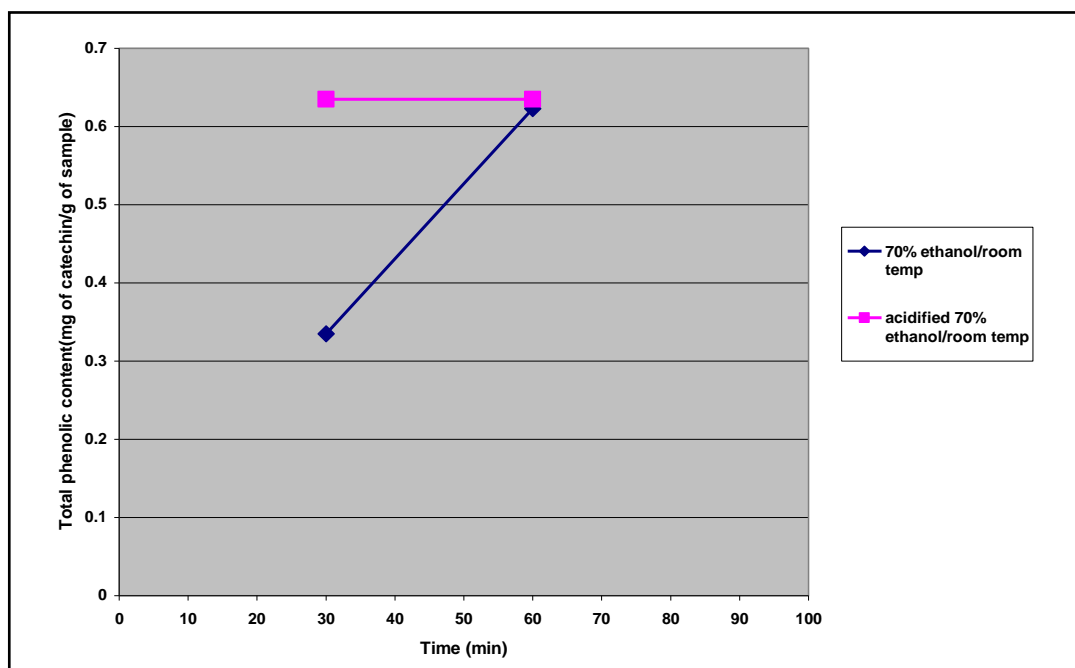
**FIG. 1. Diffuse Reflectance FTIR Spectra of soy hull extract and 100 ppm catechin**

The extraction of phenolic compounds from soy hull by 70% ethanol at 70°C and higher or using acidic condition posed a unique problem. The extracts recovered turned cloudy and formed a light brown precipitate after overnight storage. This fine precipitate was

**TABLE 1. Conditions for extraction of phenolic compound extractions of soy hull**

| Extractions | Controlled variables   | Manipulated variables                           |
|-------------|--|---|
| 1           | Extraction temperature: room temperature (23°C) Solvent: 70% ethanol | Extraction time: 30 and 60 min                  |
| 2           | Extraction time: 60 min Solvent: 70% ethanol                         | Temperature: 70°C, 80°C and 23°C                |
| 3           | Extraction time :60 min Extraction temperature: 23°C                 | Solvent: 70% ethanol and 70% ethanol/ 0.03N HCl |

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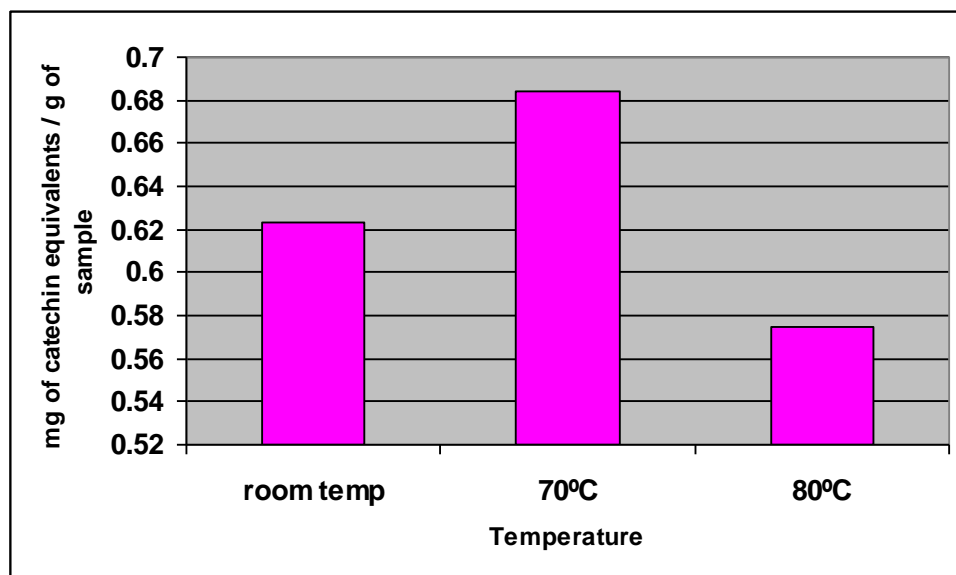
**FIG. 2. Effect of extraction time and acidic conditions on extraction efficacy**

separated from the filtrate after re-filtration with micro-filter (47  $\mu$ m) paper resulting in a transparent pale yellow solution. Extracts resulting from extraction with acidified 70% ethanol were also a cloudy brown and were also re-filtrated with micro-filter paper.

The cloudy precipitate formation was most probably due to slow precipitation of pectin that had been released by the digestion (4). Acidic or high temperature conditions during extraction with alcohol were expected to remove pectin that might have been present in the soy hull. Fig. 2 shows the effect of extraction conditions on the extraction efficacy at room temperature. For an extraction time of 60 min, the ethanol extraction attained a similar extraction efficacy to that of extraction under acidic condition. The total phenolic contents of soy hull extracts in 70% ethanol with 30 min and 60 min extraction times were  $0.34 \pm 0.06$  mg/g and  $0.62 \pm 0.10$  mg/g, respectively. The total phenolic contents of soy hull extracts in 70% ethanol under acidic conditions with both 30 min and 60 min extraction times were both  $0.64 \pm 0.10$  mg/g.

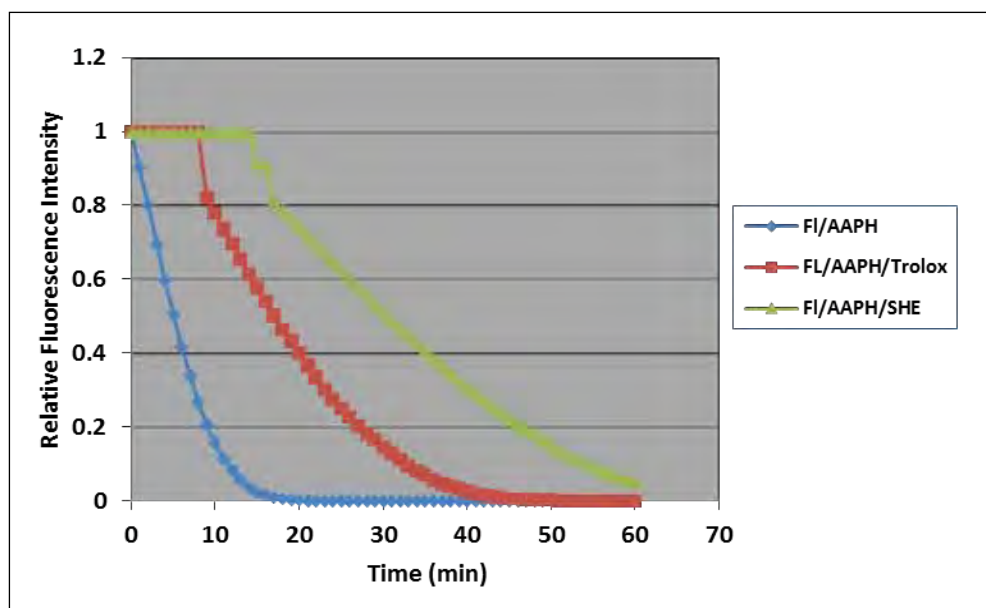
For a 60 min extraction with 70% ethanol, the total phenolic contents of soy hull extract at room temperature (23°C), 70°C, and 80°C are shown in Fig. 3. The differences in the total phenolic contents at different extraction temperatures were insignificant. However, an exposure to heat, oxygen, acid, or alkaline conditions has been shown to affect the

recovery of phenolic compounds from plant samples. Although the concentration of phenolic compounds in soy hull extract was lower compared to tea and grape products, the antioxidant activity of these compounds at lower concentration levels can make them a viable natural source.



**FIG. 3. Effect of extraction temperature on extraction efficacy**





**FIG. 4.** Fluorescent decay curves of fluorescein (0.48µm), fluorescein with Trolox (0.25 ppm), and fluorescein with soy hull extract (0.22 ppm) for ORAC assay. FL- Fluorescein, SHE- soy hull extract, AAPH - 2, 2'- azobis 2-methylpropionamide, dihydrochloride

Based on these observations, only 60 min room temperature extraction with 70% ethanol was further explored in the antioxidant capacity studies. As indicated in the previous section, there were no significant differences between the extraction efficiencies for room temperature and higher temperature extractions or for 60 min extraction with 70% ethanol and 70% ethanol with acidic conditions. However, an initial 60 min pretreatment with 0.1N HCl at 90°C to breakdown the pectin followed by addition of ethanol (to obtain 70% concentration) and removal of pectin precipitate after overnight storage, resulted in an extract with a higher concentration of polyphenol ( $1.2 \pm 0.1$  mg/g of soy hull). Since the scope of this work was to establish the use of a simple room temperature extraction process, the extraction using an acidic pretreatment at a higher temperature was not further explored in this study.

The antioxidant capacities of Trolox and soy hull extracts are compared in Fig. 4. A visual observation of decay curves suggests that, qualitatively, soy hull extract may be a better antioxidant than Trolox. Quantitative ORAC assay was  $2.23 \pm 0.29$  Trolox equivalent and was comparable with other antioxidants.

It is shown that the phenolic compounds can be extracted from soy hull using 70% ethanol at room temperature. Although the concentration of phenolic compounds in soy hull extract may seem to be very small compared to that in commercial sources, such as green tea and grape skin, the antioxidant strength of the extract makes it a prime candidate for applications. Further, soy hull's low cost and availability, as well as its simple processing requirements, can make soy hull extract an attractive alternative to synthetic antioxidants.

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Rachel Woods works at Physicians Choice Laboratory Services in Charlotte, North Carolina, USA.

Both are former students of Uruthira Kalapathy, interim chair and associate professor of the Department of Chemistry at Claflin University in Orangeburg, South Carolina, USA, and associate editor of the *Journal of the American Oil Chemists' Society*. Kalapathy can be contacted at [ukalapat@claflin.edu](mailto:ukalapat@claflin.edu).

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# Can **camelina** compete as a **feedstock** for **biobased products**?

**J.S. McLaren and X.S. Sun**

Camelina (*Camelina sativa*) is related to mustards in the Brassica family. Like its mustard relatives, it produces a small seed with a relatively high oil and protein composition. The plant, which historically was cultivated in several European countries, is also known as false flax, linseed dodder, and gold-of-pleasure. In recent times, camelina has been grown sporadically as a specialty niche crop, with soybeans, sunflower, and canola/rapeseeds being much more prominent as oilseed crops. Camelina seed composition on a dry weight basis is typically about 29% carbohydrate, 39% oil, 29% protein, and 3% ash. The oil is relatively high in oleic acid (18-20% 18:1) and  $\alpha$ -linolenic acid (34-39% 18:3), a composition similar to that of linseed oil.

- During the early 2000s, farmers in the Northern Plains of North America had high hopes that camelina would be the next cash crop for biofuels.
- From 2005–2007, the number of acres in Montana planted in camelina more than quadrupled, but the increase was short-lived due to low yields and the inability to compete with petroleum-based fuels.
- Since then, a multidisciplinary team of scientists from academia and industry spent two years studying whether the economic viability of camelina as a biofuel could be enhanced by optimizing the production system and developing byproducts. This article describes what they learned.



During the early 2000s, interest in growing camelina was renewed in the Northern Plains of North America, where it was believed that the plant would grow well under drier, low-input, conditions, and that the oil could be used as a feedstock for biodiesel. According to US Department of Agriculture (USDA) data, the number of acres planted with camelina in Montana increased from 5,000 in 2005 to over 20,000 in 2007. Today, fewer than 2,000 acres in Montana are planted with camelina. The short-lived spike in production acres was driven by expectations on economics, agronomy, and utilization that could not be met. According to USDA data, the 10-year average commercial yield for camelina is about 650 lbs/acre (at 8% moisture). Some reports indicate that the test jet fuel made from camelina oil cost over \$50/gallon, while petroleum-based jet fuel currently costs less than \$2/gallon. Clearly, it is difficult to see how camelina could be economically viable as a biofuel feedstock, given such relatively low yields and high costs.

What if camelina could be useful for products with much higher value than biodiesel alone? That is one of the main questions addressed by the Enhancing economic viability of camelina as a bio-feedstock: Optimization and demonstration of the production system and bioproduct development project (<http://tinyurl.com/pwv3z7a>), a multidisciplinary research effort funded by the US Department of Energy (DOE) and US Department of Agriculture's (USDA's) National Institute of Food and Agriculture's joint Biomass Research and Development Initiative (BRDI). The project's integrated research topics include agronomic studies to optimize field production, engineering evaluations for high-efficiency crushing and separations, advanced chemistry to develop biobased products from camelina protein and oils, economic analysis across the integrated system, and life cycle analysis from cradle-to-grave to assist in choosing the best option for each stage of product generation.

With members from both academic and industrial organizations, the research team spans a broad range of disciplines with a unique mix of highly experienced individuals. The overall project leader is Susan Sun, university distinguished professor of grain science and industry at Kansas State University (Manhattan, Kansas, USA). Other team members include Donghai Wang, Augustine Obour, Yonghui Li, Shing Chang, Terrie Boguski, Blase Leven, and Larry Erickson (from Kansas State University); Chengci Chen, Anton Bekkerman, Kent McVay, and Peggy Lamb (from Montana State University, Bozeman, Montana, USA); Thomas Foulke (from University of Wyoming, Laramie, Wyoming, USA); David Yang and Ningbo Li (from SBT Inc.); Gary Iverson (from Montana Gluten Free Processors); Jim McLaren (from StrathKirn Inc.); and two postdoctoral and nine graduate students. Following two years of intensive research, sev-

eral important results have been obtained which help clarify the opportunities and challenges for camelina.

The group's field agronomy work indicated that although camelina can be grown under drier conditions, yield levels are impacted by moisture level with a typical range from 350 lbs/acre to over 1,500 lbs/acre (under 10 inches to more than 20 inches rainfall) under commercial conditions. It should be noted that research plot trials typically have considerably higher yields (can be over 2,000 lbs/acre) than those obtained at commercial scale. This small plot effect is well-known for almost all crops. However, camelina appears to have a large range which may indicate that there is considerable potential to improve yields with appropriate practices and technologies. Both winter and spring types can be grown, but spring plantings are often more consistent due to less variation from winter kill.

Other factors were also found to impact yield. For example, the optimal planting date was early- to mid-April; the varietal performance differed depending on location, planting time, and rainfall; and the response to nitrogen was most evident below 50 lb N/acre, while no response to sulfur was observed [1].

Considerable research was done to explore the potential for camelina to replace the traditional fallow period in a dryland wheat-based cropping system [2]. In these plot-level experiments, the seed yield of camelina was 808 lbs/acre and that for wheat after fallow was 2,464 lbs/acre. When grown after camelina, the wheat yield was decreased by 13% indicating that camelina had a negative rotational impact compared to the fallow period. Economic analysis showed that the 808 lbs/acre camelina did not offset the loss of wheat yield plus the additional variable production costs, when current market prices were used. Thus, obtaining higher camelina yields and/or a higher market value for camelina products remains a challenge to be resolved before camelina could effectively compete in an altered cropping system. Advantages found for the camelina-wheat rotation included higher soil organic matter, higher overall precipitation use efficiency, and increased protection against soil erosion.

Research on the initial processing and separation of camelina seed components has provided promising enhancements compared to conventional oilseed processing methods. In addition to the oil and meal fractions, a high purity gum was isolated that has unique properties compared to commercial alginate gum. The further separation of proteins from the meal fraction was achieved by the chemical cleavage of sulfur bonds and unfolding of the camelina proteins [3]. Pilot trials are underway to evaluate a proprietary membrane separation technology for protein separations.



The various fractions from camelina seed have undergone considerable chemical research to evaluate a range of potential applications:

- Camelina protein has shown potential for use in wood adhesives [4]. Subsequent research is being done on improving the wet strength via cross-linking and other chemical modifications, such as the grafting of hydrophobic fatty acids to the protein polypeptides. Enzyme and nanoparticle modifications that were successful with soybean protein adhesives are being applied to the camelina protein adhesives.
- Camelina oil has been used to synthesize various intermediate chemicals [5]. Adhesives and coatings have been developed from these intermediates through various polymerization pathways. For example, high-gloss transparent wood varnishes were made from acrylated camelina oil through a UV-polymerization process. The camelina coatings showed better hardness, scratch resistance, mechanical strength, and heat resistance than similarly prepared soybean coatings. Pressure-sensitive adhesives from epoxidized camelina oil and camelina acrylic polyol also showed good adhesion performances, with potential applications for tapes and labels. Other pathways for conversion of camelina oils into biobased epoxies and water-borne polyurethanes, and interactions with proteins for wood adhesives, are being explored.

Currently, research is helping to optimize a facility design that has been completed for the possible future production of camelina protein-based adhesives. This type of bio-based adhesive has potential for use in wood products such as plywood.

Life cycle assessment has been applied to each stage of the system from growing the crop through to post product use (Fig. 1), in order to identify areas for potential improvement and to assist in decisions between optional approaches in producing and processing camelina. For example, the majority of the environmental impact arises from producing the crop at the field stage, making yield an important variable. Within this stage, the highest impact factor is the manufacture of nitrogen fertilizer which indicates that improving nitrogen use efficiency would have a positive effect on the environmental impact profile.

Economic studies showed that the breakeven price for camelina,

manufacture the adhesive from the camelina meal (\$0.20/lb), plus a 20% gross profit, the result is a cost of around \$0.86/lb camelina protein-based adhesive.

Current adhesive pricing is around \$0.30 for urea-formaldehyde (UF) and \$0.75/lb for phenol-formaldehyde (PF) resin. Methylene diphenyl diisocyanate (MDI) resin cost is higher at \$1.5/lb. Thus, if the camelina protein-based adhesive has the same performance as UF or PF resins then the camelina cost would be too high. The camelina cost might be acceptable if the protein-based adhesive performance were the same as MDI. The on-going research is attempting to improve the adhesive performance as much as possible, and to compare the relative performance which, in turn, will determine the economic viability of the camelina production.

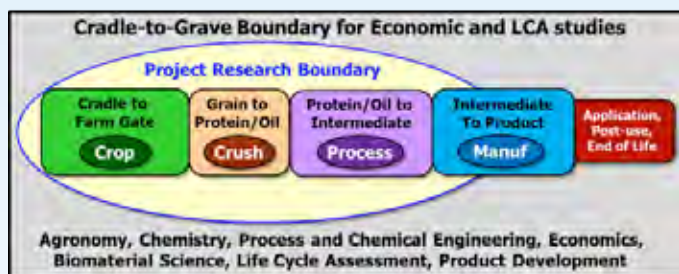
Another enhancement would be to improve the value of the camelina oil fraction, either by finding an industrial use or by altering the composition to enhance the nutritional use. Potentially valuable oils uses are being explored within the project. Also, there are projects underway by other groups that have genetically engineered camelina to have specialty omega-7 fatty acids [6], or improved omega-3 fatty acid composition [7], both of which will increase the oil value.

Thus, the research work continues via this multidisciplinary approach with the goal of improvements along the production-processing route. Increasing yield, enhancing the adhesive performance, evaluating additional bio-products, and maximizing the value from the oil are all valid targets. Taken together, improvements in all of these areas may collectively allow the creation of a camelina system that is commercially viable and environmentally acceptable.

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**FIG. 1.** Cradle-to-grave life cycle assessment system boundary and disciplines involved.

replacing fallow in a wheat rotation, was around \$0.16/lb seed using plot yields of 1,336 lb/acre. As noted previously, the average commercial yield is recorded by the USDA at 650 lb/acre, which would result in an approximate translation to \$0.32/lb seed for the breakeven price assuming similar crop inputs. This price then translates into a cost of \$0.60/lb for camelina meal if that were the only product. However, the oil also has some value. Based on a 36% oil composition and based on a soyoil price of \$0.22/lb, the camelina oil would be worth \$0.08/lb seed. Subtracting this oil value from the breakeven price results in a need for the meal to be worth \$0.52/lb. Taking a rough calculation of the energy and processing costs to

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# Hydroxylated canola biowaxes: nanocellulose composite microspheres for controlled release of aroma agents

Waxes are long-chain hydrocarbon compounds with diverse applications that include food coatings and packaging, wood coatings, industrial lubricants, adhesives, candles, ski lubricants, cosmetics, pharmaceutical formulations, among others. The global market for waxes (valued at \$8.2 billion in 2013) is expected to grow by about 5% by 2020. Although petroleum waxes currently represent more than 60% of the wax market segment, driven by environmental consciousness, the market for waxes derived from renewable feedstocks is expected to expand in the next few years.

**Samuel Mugo**

- Hydroxylated waxes have desirable physicochemical properties that are useful in cosmetics and other applications.
- Microparticles fashioned from a hydroxylated wax composite made of canola oil and nanocellulose were recently evaluated as an advanced material for the encapsulation and controlled release of the aroma compound vanillin.
- The use of canola straw nanocellulose enhanced the properties of the wax composite, while allowing single crop to be used as a feedstock source.

Natural waxes occur in the form of esters comprising a long-chain fatty alcohol esterified to a long-chain fatty acid. Current sources of natural wax esters, such as beeswax, carnauba wax, and jojoba oil, are relatively expensive and not produced on a scale that is capable of sustainably supplying the growth in demand for high-quality biowaxes. Naturally occurring waxes also lack chemical moieties, such as hydroxyl groups. This limits the dynamic interactions with other molecules that make some materials “functional.”

Castor oil is the most prevalent naturally occurring hydroxylated triglyceride from which hydroxylated biobased waxes could be produced. However, castor cultivation is largely confined to tropical environments, and concerns over the toxin ricin further limit castor oil's use as a feedstock.

Canola, camelina, and flax oils are highly unsaturated (>90%), and therefore amenable to a variety of chemical modifications, making them excellent feedstock candidates for the production of high-value, functional biowaxes. Canola is particularly a well-placed feedstock due to its large-scale production in North America, with 2.5 million hectares of land seeded with canola in Alberta, Canada, per year (Fobert, P.R., *et al.*, *Biofuels*, *Bioprod. Biorefin.*, 2: 206–214, 2008; Lokotsch, W., *et al.*, *JAOC* 73: 1459–1464, 1996). Consequently, we are developing technologies for the conversion of canola oils to serve the growing specialty functional wax market.

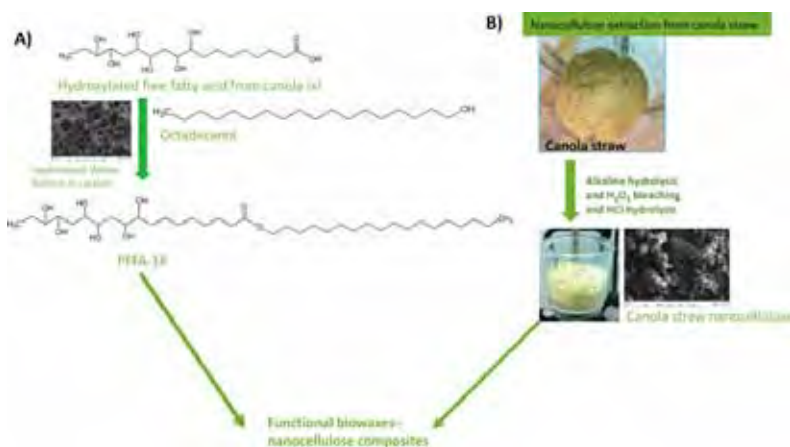
## PREPARATION OF CANOLA PFFA-18

Our research group in the Department of Physical Sciences at MacEwan University (Edmonton, Alberta, Canada) demonstrated the production of hydroxylated wax esters from canola oil (branded here as PFFA-18). As partly delineated in Fig. 1a, producing canola PFFA-18 involved a two-step process: 1. plant-oil epoxidation and ring opening to produce hydroxylated triacylglycerol (a highly viscous, clear oil resembling castor oil) and 2. hydrolysis





to a hydroxylated free fatty acid (a viscous grease similar to lanolin) and then sulfonated biochar (or carbon) esterification with a long-chain alcohol, such as octadecanol. Each of these individual steps resulted in an intermediate product with unique properties and functions that are currently being investigated. Furthermore, we achieved our goal to obtain multiple lipid products from a single plant feedstock.



**FIG. 1 (a) Preparation of canola PFFA-18 biowax; (b) Preparation of nanocellulose from canola straw**

Sulfonated willow biochar (carbon) was found to be a very effective solid acid catalyst for the esterification reaction, with 97% conversion achieved after five hours. This effectiveness can be attributed to the high surface area of this material. The biochar is also renewable and cost effective, as it is typically generated from plant waste.

The largescale production of canola oil in North America generates huge volumes of canola straw, which is of minimal use. However, it had been hypothesized that the value of this byproduct could be increased by using it to produce nanocellulose materials.

In the last few years, nanocellulose has been studied as a highly functional sustainable biomaterial, with many proposed applications, such as reinforcing of polymers and coating applications (Mitch, J., *Chem. Eng. News Archive* 92: 9–12, 2014). Our group has since dem-

onstrated that adding canola straw nanocellulose to hydroxylated canola waxes could enhance their properties.

## PREPARATION OF CANOLA STRAW NANOCELLULOSE

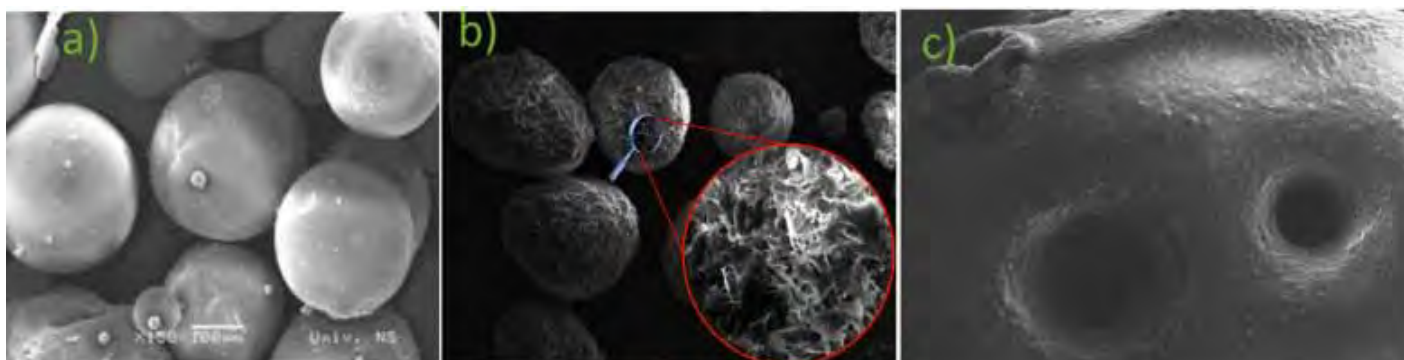
The process for extracting nanocellulose from canola straw is depicted in Fig. 1 b. Canola straw was ground and lignin removed via alkaline treatment. This was followed by bleaching and hydrochloric acid hydrolysis at 95°C. Since, pressure homogenization was not used, the resulting material was microfibrillated cellulose. However, the cellulosic material that we obtained provided a good proof-of-concept for using such material for as additives to enhance the properties of wax ester products.

## PREPARATION OF PFFA-18-NANOCELLULOSE COMPOSITE MICROSPHERES

Using differential scanning calorimetry (DSC), PFFA-18 was found to be a low-melting-point wax (27°C) with high emollient properties—features that would be useful in cosmetics, for example. The thermal properties of bulk PFFA-18 was different from the microsphere format. PFFA-18 microparticles were prepared using a hot-water-emulsion method followed by nitrogen chilling, which produced microspheres of ~ 300  $\mu\text{m}$ . Using DSC, we observed that the melting point of these microspheres increased to 33°C.

To reinforce the biowax, nanocellulose was used as an additive in the PFFA-18 at 10%. The microparticles were similarly prepared, which increased the melting point to 43°C. This was not surprising, as the hydroxylated character of both the biowax and nanocellulose results in intermolecular hydrogen bonding between them. Fig. 2 (page 638) shows the scanning electron microscopy images of the PFFA-18 and the PFFA-18/nanocellulose composite microspheres. It is evident that nanocellulose increases the surface area of the biowax microspheres. It is also notable that the biowax microspheres have unique porosity characteristics that could conceivably make them useful for encapsulation and controlled release of bioactive agents.

CONTINUED ON NEXT PAGE



**FIG.2. (a) Electron microscopy images of PFFA-18 wax microspheres; (b) and (c) Electron microscopy images of PFFA-18 wax-nanocellulose composite at different magnifications**

## BIOWAXES MICROCAPSULES CONTROLLED RELEASE OF VANILLIN

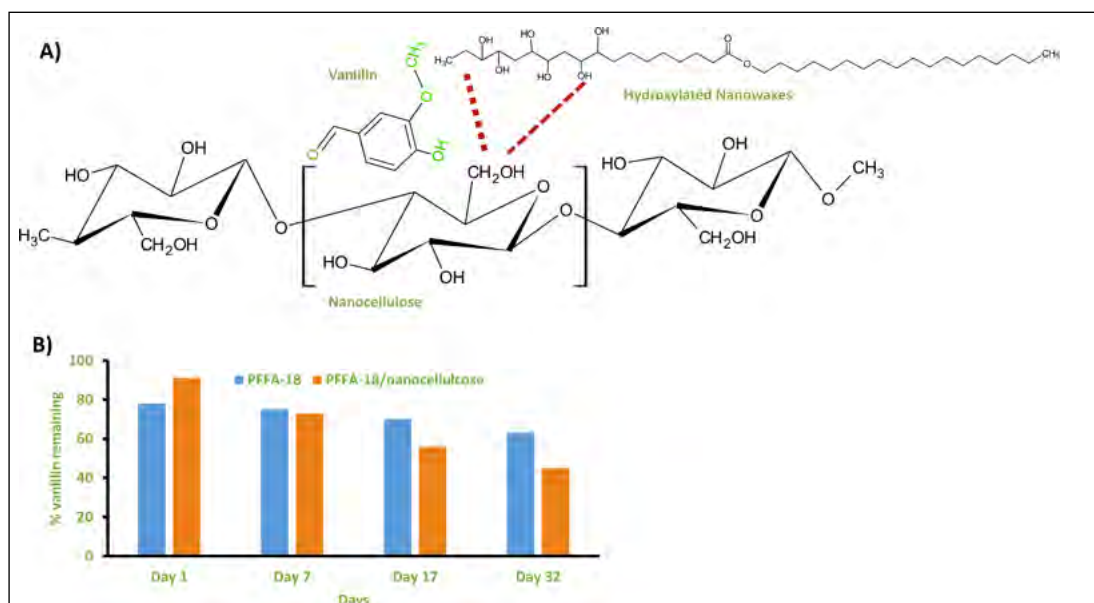
As depicted in Fig. 3a, the wax microspheres were fabricated and studied for their ability to immobilize and stabilize vanillin as well as their ability to control release.

The controlled release of vanillin in the PFFA and PFFA/nanocellulose microspheres was monitored for more than one month by spectrophotometry. The content of vanillin retained in the microspheres overtime is summarized in Fig. 3b. On day 1, the vanillin content encapsulated in PFFA-18 and PFFA/nanocellulose composite microspheres was ~77% vanillin and 91% respectively. As shown in Fig. 3b, the composite microspheres more consistently released the vanillin compared to the PFFA-18, which strongly retained the vanillin over time. This could be explained by the large porosity we observed in the morphology of the composite microspheres attributed to the defects introduced by the presence of nanocellulose.

## ONGOING WORK

The microspheres studied so far are fairly large. We expect that decreasing the size to nanoscale would significantly change the tribological and other physicochemical properties of the hydroxylated biowaxes. Consequently, the interaction between nanowaxes and nanocellulose is expected to be distinctively different. Moving forward, we are using a coaxial electrospray process to produce the nanowaxes, and conducting in-depth kinetic studies of the loading and release of other molecules, such as fungicides and antioxidants, for food packaging applications.

*Samuel Mugo is an associate professor in the Department of Physical Sciences at MacEwan University, Edmonton, Alberta, Canada. He can be contacted at [mugos@macewan.ca](mailto:mugos@macewan.ca).*



**FIG 3. (a) Structural illustration of vanillin and PFFA-18/nanocellulose composite hydrogen-bonding interaction; (b) PFFA-18 and PFFA-18/nanocellulose microspheres for controlled release of vanillin.**



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GKD-USA Inc.  
Hudson Tank Terminals  
Corp.  
Huntsman Corp. Australia  
Integro Foods Australia Pty  
Ltd.  
Intertek Agri Services  
Ukraine  
Iranian Vegetable Oil  
Industry Assn.  
ITS Testing Services (M) Sdn  
Bhd  
Kemin Industries Inc.  
Kolb Distribution Ltd.  
Kuala Lumpur Kepong Bhd  
Liberty Vegetable Oil Co.  
Lovibond North America  
Lovibond Tintometer  
Modern Olives  
MSM Milling PL  
Myande Group Co. Ltd.

Nippon Yuryo Kentei Kyokai  
Northstar Agri Industries  
Nutriswiss AG  
OLVEA Fish Oils  
Pattyn Packing Lines NV  
Pearl Crop Inc.  
Peerless Holdings Pty Ltd.  
Perry Videx LLC  
Pompeian Inc.  
Process Plus LLC  
Puerto Rico Dept. of  
Agriculture  
Rothsay  
Sanmark Ltd.  
Sea-Land Chemical Co.  
Silverson Machines Ltd.  
Sime Darby Jomalina Sdn  
Bhd-North Port  
SNF Holding Co.  
Solvent Extractors Assn. of  
India  
Spectrum Organic Products

SPX Flow Technology  
Copenhagen AS  
Storino's Quality Products  
Sun Products Corp.  
Sunset Olive Oil LLC  
Thanakorn Vegetable Oil  
Products Co. Ltd.  
TMC Industries Inc.  
Tsuno Food Industrial Co.  
Ltd.  
Unilever R&D Port Sunlight  
Lab  
Vegetable Oils & Fats  
Industrialists Assn. Turkey  
Ventura Foods LLC  
WILD Flavors Inc.  
Wilmar Biotech R&D Center  
Co. Ltd.  
Wilmar International Ltd.

*As of September 21, 2015*

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# 2015–2016 AOCS Approved Chemists

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

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**Tami Brown:** Aflatoxin in Corn Meal (test kit), Oilseed Meal

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**James Houghton:** Edible Fat, Trace Metals in Oil, Palm Oil

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**Abdul Bath:** Gas Chromatography

## Admiral Testing Services, Inc.

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**Renato M. Ramos:** Oilseed Meal, Unground Soybean Meal, Soybean, Soybean Oil, NIOP Fats and Oils, Aflatoxin in Corn Meal (test kit)

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Unground Soybean Meal

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Gas Chromatography

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Unground Soybean Meal

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**Oilseed Lab:** Gas Chromatography, Oilseed Meal, Soybean

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**Giorgio Cardone:** Olive Oil (Parts A, B and C), Olive Oil Sensory Panel

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**Katrina Sanderson:**

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**Kent Karsjens:** Oilseed Meal, Unground Soybean Meal, Soybean, Fish Meal, Soybean Oil, Aflatoxin in Cornmeal Test Kit, Nutritional Labeling

**Keith Persons:** Tallow and Grease, Cholesterol, AOCS/GOED Nutraceutical Oils, Marine Oil, Marine Oil Fatty Acid Profile, Nutritional Labeling

**Anders Thomsen:** Tallow and Grease, Oilseed Meal, Unground Soybean Meal, Soybean, Cholesterol, Fish Meal, AOCS/GOED Nutraceutical Oils, Marine Oil Fatty Acid Profile, Soybean Oil, Aflatoxin in Corn Meal Test Kit, Nutritional Labeling  
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**Cheah Ping Chong:** Palm Oil

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**Mumtaz Haider:** Tallow and Grease, Oilseed Meal, Soybean, Gas Chromatography, NIOP Fats and Oils, Aflatoxin in Corn Meal (test kit)

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**Sandra Holloway:** Aflatoxin in Corn Meal (test kit)

**Intertek Agri Services**

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**Frank Tenent:** Oilseed Meal, Unground Soybean Meal

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**Dulcinea Mendoza:** Specialty Oils

**Land O'Lakes**

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**Julie Honsa:** Edible Fat, Gas Chromatography, trans Fatty Acid Content

**Lipid Analytical Labs**

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[www.lipidanalytical.com](http://www.lipidanalytical.com)  
**Jerry Piekarski:** Cholesterol, AOCS/GOED Nutraceutical Oils

**Lysi hf**

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**Arnar Halldorsson:** AOCS/GOED Nutraceutical Oils, Marine Oil, Marine Oil Fatty Acid Profile

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**www.mpob.gov.my**  
**Dr. Hazimah Abu Hassan,**  
**Mrs. Hajar Musa:** Palm Oil,  
Gas Chromatography, trans  
Fatty Acid Content

**Merieux Nutrisciences**

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Canada  
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**Jocelyn Alfieri:** Cholesterol,  
Gas Chromatography, trans  
Fatty Acid Content, Aflatoxin  
in Peanut Paste (test kit),  
Marine Oil Fatty Acid Pro-  
file, Specialty Oils, Nutritional  
Labeling

**Modern Labs and Survey Co., Inc.**

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**Timothy S. Meyer:** Tallow and  
Grease

**Modern Olives Laboratory Services**

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(Parts A, B and C), Olive Oil  
Sensory Analysis

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**Mike Clayton:** Tallow and  
Grease

**National Beef Packing Company**

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**Sherry Robertson:** Tallow  
and Grease

**New Jersey Feed Lab, Inc.**

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**www.njfl.com**  
**Pete Cartwright:** Oilseed  
Meal, Gas Chromatogra-  
phy, Fish Meal, AOCS/GOED  
Nutraceutical Oils, Marine Oil,  
Marine Oil Fatty Acid Profile

**Northstar Agri Industries**

Kennedy, MN 56733 USA  
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**Lawrence Brosius:**  
Trace Metals in Oil

**NSF Inassa S.A.C.**

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**Carmen Catter de Bueno:** Fish  
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raphy, Olive Oil (Part A, B and  
C), trans Fatty Acid Content,  
Olive Oil Sensory Panel

**Nu-Mega Ingredients Pty. Ltd.**

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Fatty Acid Profile

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**Nutreco Canada Shur Gain**

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**Jana Pogacnik:** Oilseed Meal,  
Cholesterol, Nutritional Label-  
ing, Marine Oil, Unground  
Soybean Meal

**Olam Food Ingredients UK, LTD**

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Analysts Olam Food Ingredi-  
ents: Gas Chromatography

**Olvea Fish Oils**

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**Melanie Delvaux:** Marine Oil  
Fatty Acid Profile

**Omega Protein, Inc.—Health and Science Center**

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**Nancy Roman:** Marine Oil  
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**Owensboro Grain Edible Oils**

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**OGEO Lab:** Gas Chromatogra-  
phy, Soybean Oil, trans Fatty  
Acid Content

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**Maria Garzon:** Olive Oil Parts  
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Marine Oil, Marine Oil Fatty  
Acid Profile, trans Fatty Acid  
Content, Phosphorus in Oil

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**Steven Dominguez:** Aflatoxin  
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**Proteinas Basicas**

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**Richardson Oilseed**

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als in Oil, Vegetable Oil for  
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Trace Metals in Oil,  
Phosphorus in Oil

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**Robert Carr:** Oilseed Meal,  
Soybean

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Corn Meal (test kit)

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Sovena Oilseeds Laboratory:  
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Phosphorus in Oil, Trace Metals in Oil

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**John Dillard and Gordon**

**Whitbeck:** Tallow and Grease (MIU only), Unground Soybean Meal, Aflatoxin in Corn Meal (test kit), Oilseed Meal

**2015–2016 AOCS**

**Certified Laboratories**

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

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## Welcome New Members



### AOCS is proud to welcome our newest members\*.

\*New and reinstated members joined from July 1 through September 30, 2015.

Stefan Abel, Cape Peninsula University of Technology  
Roland Abigor, Nigerian Institute for Oil Palm  
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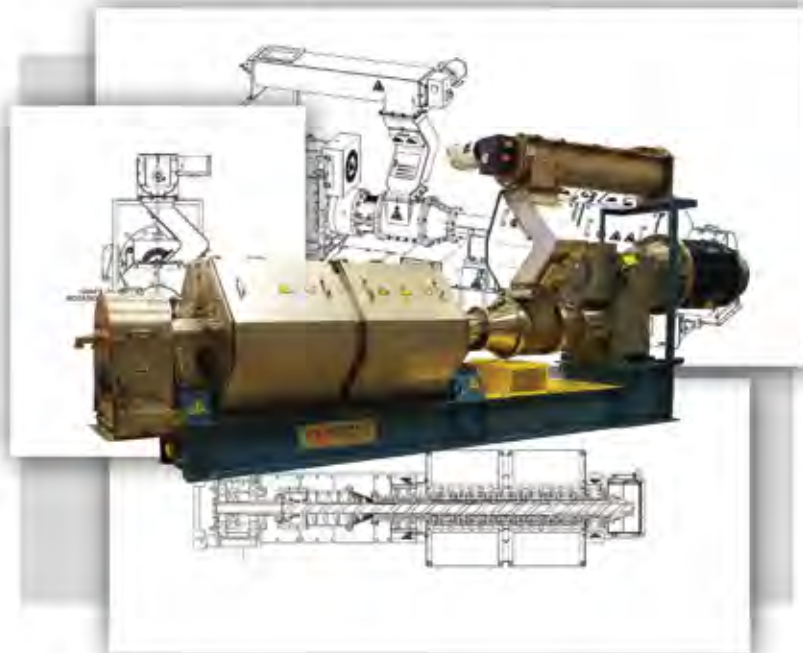
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# AOCS MEETING WATCH

**May 1–4, 2016. 107th AOCS Annual Meeting & Expo, Calvin L. Rampton Salt Palace Convention Center, Salt Lake City, Utah, USA. <http://annual-meeting.aocs.org>**

**October 5–7, 2016. World Conference on Fabric and Home Care—Singapore 2016, Shangri-La Hotel, Singapore. <http://singapore.aocs.org>**

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

Also, be sure to visit AOCS' online listing of industry events and meetings at <http://tinyurl.com/industry-calendar>. Sponsoring organizations can submit information about their events to the web-based calendar by clicking a link and completing a web form. Submission is free. No third-party submissions, please. If you have any questions or comments, please contact Patrick Donnelly at [patrick.donnelly@aocs.org](mailto:patrick.donnelly@aocs.org).

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## STATISTICAL ANALYSIS FROM MINTEC

Loraine Hudson

Rapeseed oil prices remain low due to competitive prices for alternative vegetable oils. Global production in 2015/16 is forecast at 26.0 million metric tons (MMT), down 4% year-on-year following a record harvest last year. Consumption is forecast to exceed production at 26.8 MMT, down 1% year-on-year. Consequently, global ending stocks are expected to fall by 24% year-on-year to 2.7 MMT.

Sunflower oil prices have risen slightly but remain low due to a general downward trend in vegetable oil market and the weak Russian and Ukrainian currencies. Global production in 2015/16 is forecast at 15.0 MMT, down 1% year-on-year. Falling production in Ukraine, down 3% year-on-year at 4.2 MMT, will be offset by rising production in Russia, up 3% year-on-year at 3.7 MMT.

Palm oil prices have fallen as low crude oil prices have reduced demand for biofuels. Events in China over the summer, the world's third largest importer, have also raised concerns about lower demand. Global production of palm oil in 2015/16 is forecast at a record 65.2 MMT, up 6% year-on-year. Ending stocks are estimated at 9.5 MMT, up 5% year-on-year.

Soybean oil prices fell due to low demand in the biodiesel market. Prices have also fallen due to forecasts for a record global soybean crop this season, up 5% year-on-year at 50.7 MMT. Consumption is expected to rise by 5% year-on-year to 50.2 MMT.

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# Transforming the “art” of formulation to **dynamic fluid “engineering”**

Pasha Hammond

This article is based on a presentation Charles Hammond, winner of the 2015 Samuel Rosen Memorial Award, gave at the 106th AOCS Annual Meeting & Industry Showcases in Orlando, Florida, USA.

Where are we in the process of transforming the “art” of formulation to dynamic fluid “engineering”? I believe that as individuals and discreet systems we know what we are doing. However, the language around surfactants has yet to become universally defined. Consequently, at present, concepts cannot be effectively shared and communicated.

- The language of any given field can hinder progress.
- New ideas often cause tension by challenging existing beliefs.
- The language of enhanced oil recovery is not sufficient to explain realities in the field.

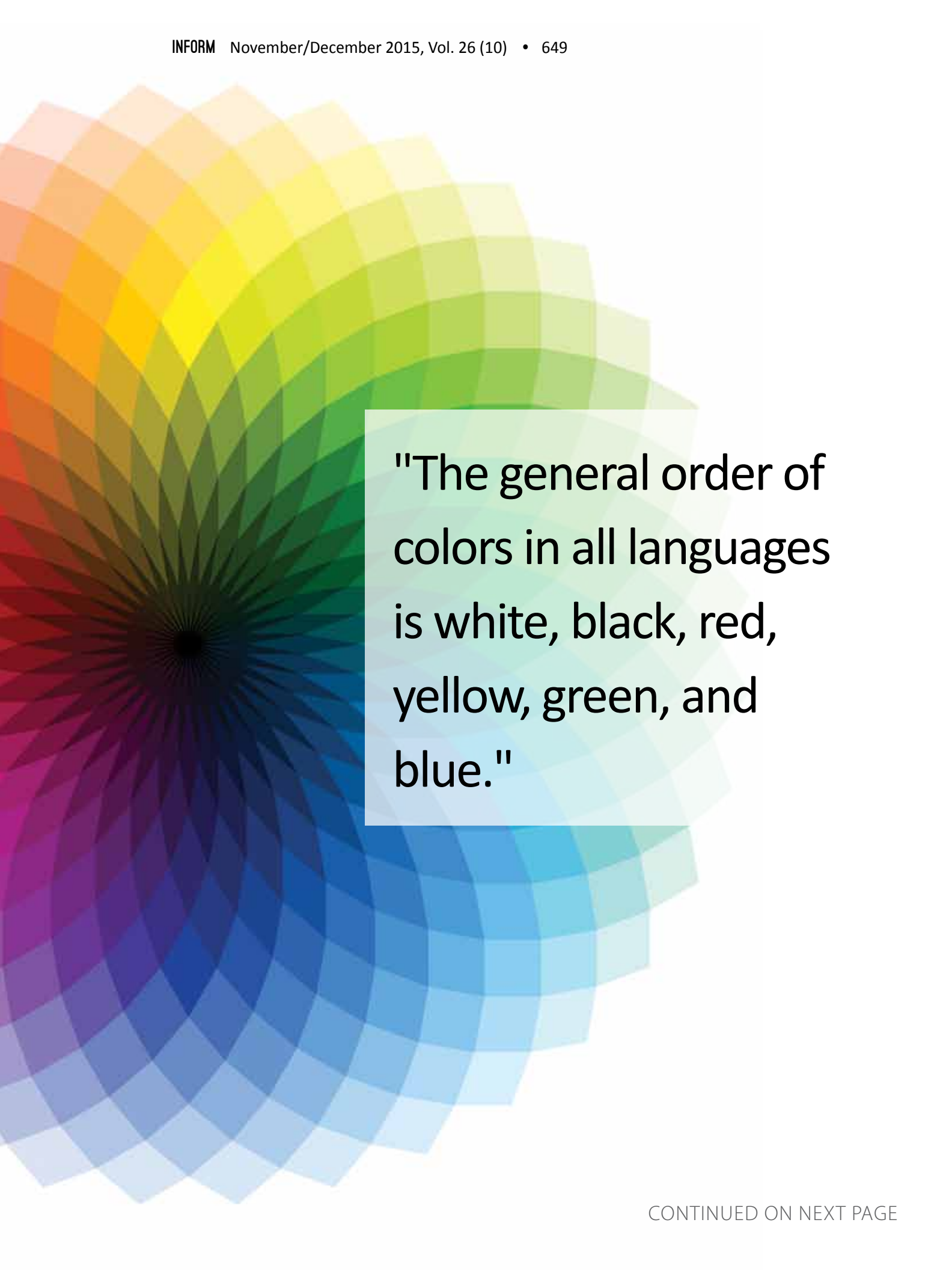
Let’s draw an analogy by examining color theory and language. The colors were often named once they could be synthetically made. The general order of colors in all languages is white, black, red, yellow, green, and blue. Until the 1600’s, it was thought that colors shown through a prism were created by the prism. Around 1666, Newton was bored after having been sent home due to a plague sweeping through Britain, and discovered that it was the light, as opposed to the prism, that contained the colors. It was not until 1810 that the theory of colors was published. In an area of Africa named Tiv, only three colors are named (Fig. 1)

| English | Tiv   |
|---------|-------|
| green   | ii    |
| blue    |       |
| gray    |       |
| brown   | nyian |
| red     |       |
| yellow  |       |

FIG. 1. English and Tiv color charts

How can three colors, two of which are different tones of one color, be wrapped into a color wheel? They can’t. Therefore it is not possible for people in Tiv to understand or apply color theory, because they are not equipped with the language to facilitate the idea (<http://tinyurl.com/cre7eyr>).



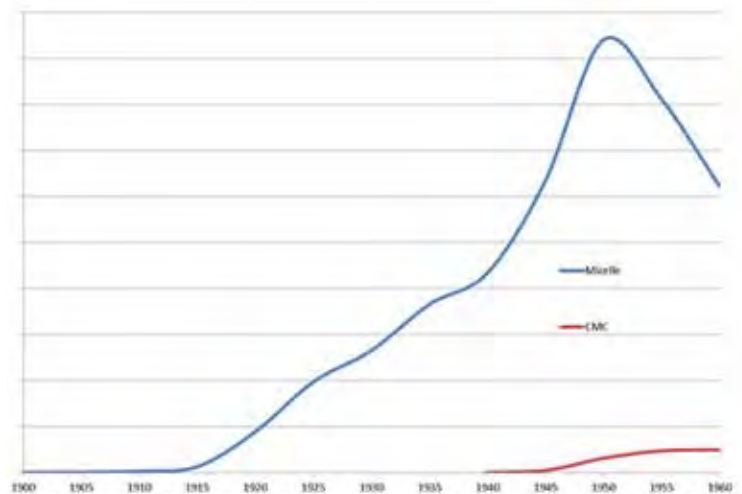


"The general order of colors in all languages is white, black, red, yellow, green, and blue."

The language we use defines what we view as reality. So, we must look at the language of surfactants to determine if it allows us to have the necessary discussions to advance the field by sharing ideas and understanding.

## THE EARLY LANGUAGE OF SURFACTANTS

Google's N-Gram viewer can probe the use of words in literature over time. In looking at the terms "colloidal solution" and "micelle" in Ngram viewer (Fig. 2), it is easy to see a rise in the term "colloidal solution" until about 1917, when the term "micelle" was introduced (<http://tinyurl.com/mevc696>). A micelle is defined as an aggregate of molecules in a colloidal solution, such as the molecular aggregates formed by detergents, and is a much better description than the previous term. After the transition from colloid to micelle, it took another 25 years for terms like "phase behavior" and "critical micelle concentration" to show up. These more complex ideas could not be developed until the basic language and terminology were defined, and this extends to all aspects of the surfactant field such as the names of the molecules involved. The molecules making micelles were discussed using terms such as "hydrophobic" and "lipophilic" or, as in Winsor's case in 1954, "amphiphilic molecules." In the mid 1950's, the hyphenated term "surface-active agents" and the shortened equivalent, "surfactants," were adopted to



**FIG. 2.** Use of the terms colloidal solution (CS) and micelle in literature over time (Source: Google N-Gram viewer)

describe molecules that partition to the interface. However, this is still a misnomer because any molecule could be a surfactant in the right environment. (One could envision the term used to describe molecules that are added to formulations with the intention to function at the interface.) Thus, one could consider that the word "surfactant" correlates to the ability to place molecules at the interface as intended, analogous to the naming of a color correlating with the ability to synthesize the color.

## Tips from **inform|connect**

*Tips from inform|connect is a regular Inform column that features tips and other discussion highlights from the community forum board at <http://www.informconnect.org/home>.*

**Q:** A professional in the textile aftercare industry who works at a company that uses liquid silicone in the cleaning of consumer wear had some questions about activated bleaching earth. The company where he works uses activated bentonite clays to filter the silicone. The activated clay works extremely well, removing the fugitive dye, odors, and other particles in the cleaning medium. Because activated clay is also used to filter and purify olive oils and other food-grade oils, the textile aftercare professional was interested in learning how, exactly, bleaching earth is used in the food industry. He noted that the clay has a very short effective life and was hoping to learn how the food industry deals with that issue.

**A:** A community member replied that activated bleaching earth is used for nearly all oils except extra virgin olive oil and provided the following summary: The oil is treated with 0.5–4.0 wt% earth by adding the earth to a vessel containing the oil; the oil temperature is usually close to 100°C; the contact time is 15–30 minutes; and the slurry is subsequently filtered using a pressure leaf filter. He explained that some companies use a precoat, such as diatomaceous earth (kieselgur), but that circulating the slurry across the filter until the filtrate is clear saves on the cost of kieselgur. The textile aftercare professional also learned that he could find more information on the Lipid Library website at <http://tinyurl.com/BleachingEarth>.

## THE LANGUAGE OF SURFACTANTS TODAY

The misnomer “microemulsion” is a prime example of where the language is today. Neither micro nor an emulsion, the term gained traction in the 1970's when it was used to describe the point when surfactants self-organize in solution to a bi-continuous, semi-structured low-energy state that in some cases correlates to ultra-low interfacial tension. Such systems were developed to improve crude oil recovery out of rock formations in the earth. In some circles, the definition was expanded to describe the systems that range from oil in water to water in oil to bicontinuous, are thermodynamically self-organizing, and are referred to as Winsor type I, II, III, and IV. Interestingly, Winsor purportedly did not endorse the use of the term microemulsion, and he refused to serve as a coauthor for a book if microemulsion was used in the title.

One can appreciate that a word with complex meaning would simplify discussions. However, the word microemulsion is such a misnomer that it causes more confusion than it solves. Even among learned, skilled colleagues in the art, it seems as though every discussion involving the term must be preceded with a discussion and oftentimes a debate about what a microemulsion is. This is akin to the discussions that arise when playing monopoly with someone for the first time. Do the fines go into free parking or not? What are the rules around trading properties when it's not your turn? The need to establish “house rules,” such as what the definition of a microemulsion is, muddles discussions and leads to confusion and miscommunication. For cooperation and the growth of new ideas to flourish, everyone needs to play from the same rulebook at all times. Isn't it time we purposefully begin to create new words that can be effectively defined, searched, and taught to manage the language so that all colors are named and the art of formulation may be transformed into dynamic fluid engineering?

*Pasha Hammond is the son of Charles Hammond and is currently attending the Math and Science Academy at Dulles High School. Charles Hammond can be reached at [CHammond@flotekind.com](mailto:CHammond@flotekind.com).*

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# How new chemicals are identified

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

## Charlotte Niemiec

As countries start to regulate chemicals, one of the first things they do is figure out what is already on the market and what is new.

In most cases, they then introduce controls aimed at new substances—preferring to deal with the more complex question of existing chemicals separately.

There is no globally accepted definition of a “new” chemical, but at ChemCon Asia in June 2015, Karon Armstrong provided a roundup of the general criteria.

Firstly, a substance must not be listed on an inventory or listed as a category on an inventory. It must not be a component of an article or an impurity, or a naturally occurring substance.

Chemicals imported for R&D purposes do not qualify as new chemicals, nor do polymers that are eligible for exemption. Chemicals imported in low volumes or for export purposes only are also not considered new chemicals. If these criteria are fulfilled, a chemical must be registered as new. However, companies are falling at the first hurdle. It is not always easy to determine if a chemical is new simply by checking inventories. This is the case, for instance, if a substance has only recently been notified or notified as confidential. While the option to make confidential notifications is necessary to protect chemical identity, it is another hoop to jump through when determining if a chemical is new.

Timing is another factor. After notification approval of a substance, it is published in the *Inventory Gazette*. Sometimes, a notified chemical is immediately added to an inventory. This is most common if it has not been noti-

fied confidentially. Official listing takes place three to five years after approval. Until then, a new chemical cannot be manufactured or imported by a party other than the notifier until a new manufacturer or importer submits a secondary notification. After a substance is notified, others may use the same substance without additional notification.

There are 12 main global inventories, belonging to Australia, Canada, China, the EU, Japan (two inventories), Korea, New Zealand, the Philippines, Switzerland (separate from the EU), Taiwan, Thailand, and the United States.

Companies must check the inventory of the country to which they import or manufacture. If a chemical appears on the national inventory, it is not a new chemical and must be notified. In some countries, if a substance appears on another country's inventory, a simplified notification can be submitted.

## NOTIFYING A NEW CHEMICAL

The EU's REACH Regulation [Regulation on Registration, Evaluation, Authorisation and Restriction of Chemicals] is unique in that it does not distinguish new from existing chemicals by inventory listing. If a substance is being imported in amounts over one metric ton per year and is not registered, each legal entity must register the substance by submitting an inquiry dossier to the European Chemicals Agency (ECHA). ECHA's response will detail what data [are] required to register the substance, what data [are] already available, and if there are any other registrants.

In the United States, the Environmental Protection Agency's New Chemicals Program enforces a ban on the production or use of a new chemical before it enters the market. Manufacturers and importers must complete a premanufacture notice (PMN) and submit this at least 90 days prior to manufacture of the chemical.

New chemical procedures in Asia span the board from mature to almost nonexistent. Korea's newly imple-

mented Act on the Registration and Evaluation of Chemicals (K-REACH) is similar to EU REACH in its scope and requires new chemicals to be registered in much the same way.

Japan's regulations are also well established: All manufacturers or importers of new chemicals must file a notification three months in advance and substances are subject to testing. Notifications must be in Japanese and, once approved, they are added to the Inventory of Existing and Notified Substances (ENCS) three to five years later. There are separate notifications under Japan's Industrial Safety and Health Law and substances notified on this inventory are often added after just one year. Japan defines a new chemical as one that is not listed on the ENCS inventory, is not a class I- or II-specified substance, a designated substance, or on a published list of notified new "safe" substances. Manufacturers often have to wait years before the substance is added to the inventory and, during that time, only the original notifier can manufacture or import the substance.

China's Ministry of Environmental Protection (MEP) Order 7 requires companies to submit new chemical substance notifications irrespective of annual tonnage. The requirements apply to new substances, those used in preparations or articles intended to be released, and also new substances used as ingredients or intermediates for pharmaceuticals, pesticides, veterinary drugs, cosmetics, food additives, and feed additives.

## CONFIDENTIAL BUSINESS INFORMATION (CBI)

One of industry's prevailing concerns is that of protecting the identity of chemicals used in products. To tackle this problem, most countries allow chemicals to be notified confidentially and inventories are often split into public and confidential sections.

Taiwan's Chemical Substance Inventory, for example, has both a public and confidential section. Notifiers have automatic confidentiality on volume in the public section, and notified chemicals are added after five years. To claim CBI, a separate application must be submitted that will allow the CAS number and chemical name to be concealed. Chemicals are instead identified by serial number and generic name, and are kept confidential for five years, with the option to extend for another five years.

The United States has a similar system. There is a confidential section in its Toxic Substances Control Act (TSCA) inventory but, to appear on the list, a detailed, written response to 14 questions must be submitted, along with a CBI claim.

The EU, on the other hand, requires all hazardous substances to be declared on safety data sheets and it is almost impossible to keep chemical names confidential. The EU has two main inventories, the European Inventory of Existing Chemical Substances, which is a static inventory of around 100,000 substances, and the European List of Notified Chemical Substances, which contains around 4,300 company-specific new chemical notifications.

China and Korea have no separate, confidential inventories, but generic names appear on a public listing along with a reference number. In Korea, CBI can only be claimed for nonhazardous substances, and these substances must not appear on any other public inventories. Neither of Japan's inventories [has] confidential sections, although the authorities do have some provisions to protect disclosure of newly listed substances.

Chemicals are often listed on an inventory more quickly if a company foregoes claiming CBI.

Nevertheless, due to the lack of global harmonization of CBI, companies are at an impasse when trying to claim it in a country that allows for this, while notifying in another that requires full disclosure of a hazardous substance.

## POST-NOTIFICATION

There are further requirements after notification, which differ country by country. In China, records must be kept of new chemical notification, quantity, flow status, and its impact until it is published in the inventory.

In Japan, notifiers must report actual volumes of the chemical and comply with labeling and hazard information laws. The government can direct manufacturers and importers to investigate long-term toxicity for humans, top predators, and flora and fauna.

In the Philippines, a Notice of Commencement must be submitted to the Environmental Management Bureau with a bill of landing, import entry, and tax revenue declaration.

Both new and existing substances may need to be notified a second time (a secondary notification), if new information is received on the substance, such as an increase or change in exposure, an increase in tonnage, a switch to domestic manufacture instead of import, a change in manufacturing method, or a change in polymer characteristics.

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*Charlotte Niemiec is the Asian desk editor for Chemical Watch, a regulatory news service with headquarters in London. This article is reprinted from the July-August 2015 Global Business Briefing, ©2015. It is reproduced by permission of CW Research Ltd. at [www.chemicalwatch.com](http://www.chemicalwatch.com).*



JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY

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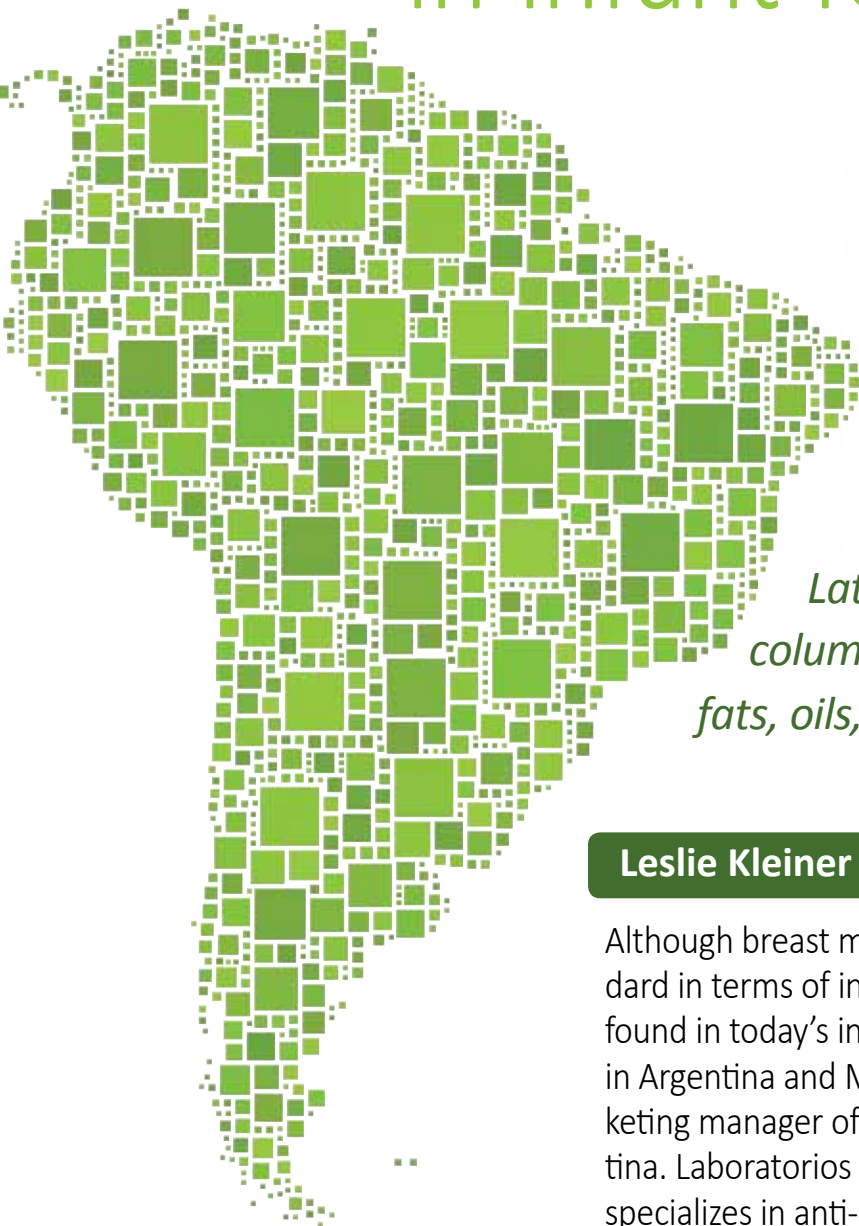
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# Developments in infant formula



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

## Leslie Kleiner

Although breast milk has long been considered a gold standard in terms of infant nutrition, many developments can be found in today's infant formulas. To learn about infant formula in Argentina and Mercosur, I interviewed Ariel Melamud, marketing manager of Laboratorios Argentina, Buenos Aires, Argentina. Laboratorios Argentina is a pharmaceutical company that specializes in anti-inflammatory, gastroenterological, and cardio-metabolic drugs, as well as infant nutrition.

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](mailto:LESLIE.KLEINER@roquette.com).





**Q: WHAT TRENDS ARE YOU SEEING IN THE INFANT FORMULA MARKET IN ARGENTINA AND MERCOSUR?**

**A:** Currently, the biggest tendency in infant formula lies in approximating the ingredients to those naturally found in breast milk and adding micro and macro nutrients that have important health benefits for the infant. Some of these nutrients are the fatty acids arachidonic acid (ARA) and docosahexaenoic acid (DHA), which have been clinically proven to improve cognitive, gastrointestinal, and immunological function. Furthermore, a new development in the history of infant formula is that of incorporating prebiotics and probiotics to the formulation.

**Q: PREBIOTICS, PROBIOTICS, AND NEWLY DEVELOPED SYMBIOTICS .... COULD YOU EXPAND ON THE DEVELOPMENT OF THE SYMBIOTIC INFANT FORMULA NUTRIBABY, AND ITS BENEFITS FOR INFANTS?**

**A:** Recently, it was found that breast milk contains probiotic components. The benefits of probiotics are broad and have been reported to aid in diseases related to premature birth [such as enteritis necroticans] as well as in immunological processes. In today's worldwide market, there are some infant formulas that contain either prebiotics or probiotics. However, few formulations provide both symbiotic components. With our team of nutritionists and related scientists, we have performed extensive R&D for the development and commercialization of "Nutribaby," the only symbiotic [galacto-oligosaccharides and Bifidobacterium lactis] infant formula to date in Argentina and Mercosur.

**Q: THERE ARE VARIOUS STUDIES ON THE SYNTHESIS OF STRUCTURED LIPIDS WITH INCREASED PALMITIC ACID CONTENT AT THE SN-2 POSITION OF THE TRIACYLGLYCEROLS (TAGS). THIS MODIFICATION OF THE TAGS IS INTENDED FOR BOTH IMPROVED PALMITIC ACID ABSORPTION AND INFANT NUTRITION. IN ARGENTINA AND MERCOSUR, ARE THESE TYPES OF STRUCTURED LIPIDS USED FOR INFANT FORMULA DEVELOPMENT?**

**A:** Indeed, structured lipids with increased palmitic acid content at the sn-2 position are a new achievement for the development of infant formula. These structured lipids could arise from palm oil or milk fat, among other sources, and could be used as valuable components of infant formula.

**Q: FOR THE NEXT FIVE YEARS, HOW DO YOU FORESEE THE GROWTH OF INFANT FORMULA CONSUMPTION IN ARGENTINA AND MERCOSUR?**

**A:** The infant formula market in Argentina and Mercosur is under constant growth, and I expect this trend to continue. In many Latin American countries, the infant formula market is the most important market within the pharma/ nutraceutical segment.



# Fatty acid distributions within glycerolipids in plant tissues

*Lipid Snippets is a regular Inform column that features select content from The AOCS Lipid Library (<http://lipidlibrary.aocs.org/>).*

Once more, the triacylglycerols of the commercial seed oils are those to have been subjected most frequently to detailed structural analyses. In general, there tends to be little difference between the compositions of positions *sn*-1 and *sn*-3 of the glycerol moiety, but the saturated fatty acids are concentrated in the primary positions and the unsaturated are in greatest abundance in position *sn*-2. In some instances, there appears to be a higher proportion of longer-chain fatty acids (C<sub>20</sub> to C<sub>22</sub>) in position *sn*-3 than in position *sn*-1, and sometimes the more unusual fatty acids are concentrated in position *sn*-3.

The positional distributions of fatty acids in many of the glycerophospholipids of plants seem to resemble those of animal tissues in that the saturated fatty acids are concentrated in position *sn*-1 and the unsaturated in position *sn*-2. However, phosphatidylglycerol from spinach leaves and the 'model' plant *Arabidopsis thaliana*, is unusual in that the major molecular species contains linolenic acid in position *sn*-1 and *trans*-3-hexadecenoic acid in position *sn*-2 [1,2]. Data for both glycolipids and glycerophospholipids are listed in Table 1.

In those plants containing 16:3(*n*-3), the monogalactosyldiacylglycerols consist mainly of the 16:3-18:3 combination (with all the 16:3 in position *sn*-2), while the digalactosyldiacylglycerols have the more common 18:3-18:3 species. The distinctive compositions of the phosphatidylglycerol and monogalactosyldiacylglycerol are believed to result from the existence of a primitive prokaryotic biosynthetic pathway in addition to the more usual eukaryotic pathway in the chloroplasts [3]. This is discussed further in our web page on glycosyldiacylglycerols.

**Table 1. Composition (mol %) of fatty acids in positions *sn*-1 and *sn*-2 of mono- and digalactosyldiacylglycerols, phosphatidylcholine, phosphatidylethanolamine and phosphatidylglycerol from leaves of *Arabidopsis thaliana* [1].**

| Lipid class | Position     | Fatty acids |      |       |       |      |      |
|-------------|--------------|-------------|------|-------|-------|------|------|
|             |              | 16:0        | 16:3 | 18:0  | 18:1  | 18:2 | 18:3 |
| MGDG        | <i>sn</i> -1 | 2           | 1    | trace | trace | 4    | 93   |
|             | <i>sn</i> -2 | trace       | 70   | trace | trace | 1    | 28   |
| DGDG        | <i>sn</i> -1 | 15          | 2    | trace | 2     | 3    | 76   |
|             | <i>sn</i> -2 | 9           | 3    | trace | trace | 4    | 83   |
| PC          | <i>sn</i> -1 | 42          |      | 4     | 5     | 23   | 26   |
|             | <i>sn</i> -2 | 1           |      | trace | 5     | 47   | 47   |
| PE          | <i>sn</i> -1 | 58          |      | 4     | 5     | 15   | 18   |
|             | <i>sn</i> -2 | trace       |      | trace | 2     | 60   | 38   |
| 3f-16:1     |              |             |      |       |       |      |      |
| PG          | <i>sn</i> -1 | 22          | -    | trace | 9     | 13   | 55   |
|             | <i>sn</i> -2 | 23          | 41   | trace | 1     | 8    | 8    |

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## Continued from page 623

that the protective effect of the antioxidant ingredients is more effective during storage. It is also interesting to note that at time zero both pepper varieties exerted a similar activity, while after ageing the sweet pepper shown higher activity than the hot one. These data suggest that the antioxidant activity is not connected to the capsaicinoids content, but is probably due to the combined action of all the antioxidants compounds occurring in the pepper variety.

It can be concluded that a perishable food such as olive oil can be protected by adding natural products at very low percentage, without significantly affecting its organoleptic properties. In particular, the strong effect recorded by the addition of only 1% of sweet red pepper suggests that this fruit could have important applications in canned food, as it should not significantly affect the overall flavor of the final product.

*Claudio Corradini is an associate professor in the Department of Chemistry at the University of Parma (Parma, Italy), where Antonella Cavazza is a researcher, Paola Salvadeo is a research fellow, and Carmen Laganà is a doctoral student.*

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# PATENTS

## Type A gelatin capsule containing PUFA in free acid form

Sachetto, J.-P., *et al.*, Chrysalis Pharma AG, US9012501, Apr. 21, 2015

A pharmaceutical formulation comprising at least one omega-3 polyunsaturated fatty acid in free acid form or a pharmacologically acceptable derivative thereof is contained in a soft gelatin capsule characterized in that the capsule comprises gelatin extracted by an extraction process comprising acid pretreatment of a collagen source. One advantage of the present invention over a soft to gelatin capsule containing the same formulation but comprising gelatin extracted by an extraction process comprising alkali pretreatment of the collagen source is that the present invention does not harden significantly over time and thus has a longer shelf life.

## Cocoa and sugar agglomerate for flavored beverages and method of forming same

Wang, X., The Hershey Co., US9017750, Apr. 28, 2015

A method of forming cocoa and sugar base agglomerates is disclosed in which cocoa powder and sugar is mixed with an aqueous ethanol solution to form a dough and the dough is dried to form agglomerates of a porous dried dough having less than 0.5% bwt residual ethanol. The dried dough may be broken into pieces or formed into pieces by cutting prior to drying. The resulting pieces of the agglomerate disintegrate instantly in a cold aqueous liquid, such as water or milk, to form a flavored beverage.

## Shortening composition

Doucet, J., Dupont Nutrition Biosciences APS, US9017752, Apr. 28, 2015

The present invention relates to a shortening system for use as dough fat or filling fat. The shortening system contains unhydrogenated or nonhydrogenated vegetable oil, such as a highly unsaturated, nonhydrogenated, or unhydrogenated vegetable oil, e.g., soybean oil, sunflower oil, corn oil, ricebran oil, or cottonseed oil and a minimum or minor amount (e.g., by weight about 3–10%, advantageously about 3–7%, more advantageously about 3–6% or about 3–5% or less than about 6% or less than about 8% of an emulsifier composition. The emulsifier composition comprises a monoglyceride and/or diglyceride, an alpha tending emulsifier and an ionic emulsifier. Methods for making and using such a shortening system and products from the use of such a shortening system are also disclosed.

## Oil or fat composition containing diglycerides

Saito, K., *et al.*, Kao Corp., US9017753, Apr. 28, 2015

Provided is an oil or fat composition rich in diacylglycerol, including a high content of oleic acid in a constituent fatty acid thereof and having a good appearance at normal temperature. Specifically, the oil or fat composition satisfies the following items (1) to (4): (1) the oil or fat composition includes diacylglycerol in an amount of 20 mass% or more; (2) constituent fatty acids of the diacylglycerol include oleic acid in an amount of 30 mass% or more; (3) the mass ratio of the content of (B) 1,3-dipalmitin diacylglycerol to the content of (A) 1,2-dipalmitin diacylglycerol, [(B)/(A)], in the diacylglycerol is 1.45 or less; and (4) the mass ratio of the content of (D) 1,3-diacylglycerol to the content of (C) 1,2-diacylglycerol, [(D)/(C)], in the diacylglycerol is 1.2 or more.

## Methods for enzymatic decolorization of chlorophyll

Lam, D., *et al.*, DSM IP Assets B.V., US9017990, Apr. 28, 2015

The invention provides the invention provides compositions and methods for the enzymatic treatment (“bleaching” or “de-colorizing”) of chlorophyll-comprising compositions, e.g., algae preparations, chlorophyll-containing or chlorophyll-contaminated feeds, foods or oils, for example, vegetable oils, including oils processed from oilseeds, such as canola (rape-seed) oil or soybean oil, or oil fruits, such as palm oil. In one aspect, the invention provides methods using a chlorophyllase enzyme for the enzymatic hydrolysis of chlorophyll in an algaal, an animal (e.g., a fish) or plant preparation, a food or an oil. In one aspect, the chlorophyllase is immobilized onto a silica. The invention also provides compositions of manufacture and detergents.

## Fatty acid pharmaceutical foam

Abram, A.Z., *et al.*, Stiefel Research Australia Pty Ltd., US9023863, May 5, 2015

The present invention provides a foamable composition comprising water and an organic solvent, wherein the organic solvent comprises a fatty acid. The composition may further comprise a pharmaceutically active agent. The composition of the invention is also useful for the treatment of a dermatological disorder in a mammal by the topical administration of the composition.



## Purification of crude glycerol

Macret, R., *et al.*, Rhodia Poliamida e Especialidades LTDA, US9024081, May 5, 2015

Crude glycerol obtained from raw materials, such as the glycerol obtained during the production of biodiesel or glycerol obtained during the conversion of fats or oils, is purified by forming a dioxolane therefrom by reacting the crude glycerol with a ketone or aldehyde, separating the dioxolane thus formed, converting the dioxolane into purified glycerol and ketone/aldehyde, and recovering the glycerol thus purified.

## Methods and apparatus for controlling moisture in plant oils and liquid biofuels

Matsumura, M., Knobbe Martens Olson and Bear LLP; Ohana Investment Works LLC, US9028697, May 12, 2015

The moisture absorption capacity of biofuels can be more or less 10 times that of fossil diesel oil, causing biofuels to form acids that induce metal corrosion and form deposits in the fuel tank and pipe lines. Methods for removing moisture from stored biofuels and plant oils are described wherein glycerol is used as a solvent to extract the moisture from the bioliquid or oil, comprising the steps of placing the biofuel or oil in fluid contact with glycerol, incubating for a time, and then removing the glycerol. A cellulose ester dialysis or other semi-permeable membrane may be used to prevent the glycerol from contaminating the biofuel while allowing moisture to pass. Crude glycerol produced as a byproduct of biodiesel production may be used in the method of the disclosed invention with good result. Preferred embodiments of apparatus that employ the method of the subject invention are described.

## Method for producing oil containing highly unsaturated fatty acid using lipase

Doisaki, N., *et al.*, Nippon Suisan Kaisha, Ltd., US9029584, May 12, 2015

A method for lowering content of a sterol in a highly unsaturated fatty acid-concentrated oil in a method to concentrate a highly unsaturated fatty acid by using a lipase reaction, may include removing the free form of the sterol from a raw material oil that includes a highly unsaturated fatty acid-containing glyceride prior to performing the lipase reaction; and thereafter concentrating the highly unsaturated fatty acid in the glyceride by using a lipase that is less reactive for the highly unsaturated fatty acid.

## Burner for unprocessed waste oils

Leue, T.S., Homestead Engineering Inc., US9033698, May 19, 2015

A system and techniques for waste oil combustion are provided. The techniques include straining waste oil, separating contaminants from the strained waste oil to produce segregated layers of waste oil, selectively drawing a segregated layer of the waste oil, entraining the drawn waste oil to a burner for combustion, and regulating flow rate to produce a controllable amount of heat output. The system includes a container for straining waste oil, a storage drum for separating contaminants from the strained waste oil via gravity separation to produce segregated waste oil, an oil uptake channel for entraining the waste oil from the storage drum, a fuel regulator for controlling rate of flow of the waste oil from the storage drum to a burner head via the oil uptake channel, and a burner head control for repositioning a burner head under the input stream to produce a controllable amount of heat output.

## Product based on conjugated linoleic acid and a method for the manufacture thereof

Lorenzon, M., SILA S.R.L., US9034385, May 19, 2015

A product based on conjugated linoleic acid (CLA) comprises an inner core in which the conjugated linoleic acid is substantially concentrated as well as a coating for covering and protecting the inner core; the coating in turn comprises a fraction greater than 80% bwt relative to the coating of a lipid matrix formed by glycerides of C16, C18, C20 and C22 saturated fatty acids.

## Compositions and methods for making and modifying oils

Lam, D., *et al.*, DSM IP Assets B.V., US9034612, May 19, 2015

The invention provides novel methods for making or modifying oils, e.g., plant animal or microbial oils, such as vegetable oils or related compounds, that are low in a particular fatty acid(s), for example, low linoleic oils, linolenic oils, low palmitic oils, low stearic oils or oils low in a combination thereof.

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](mailto:scott.bloomer@adm.com).



# EXTRACTS & DISTILLATES

## Lipid-lowering activity of natural and semi-synthetic sterols and stanols

Taha, D.A., *et al.*, *J. Pharm. Pharm. Sci.* 18: 344–367, 2015.

Consumption of plant sterols/ stanols has long been demonstrated to reduce plasma cholesterol levels. The objective of this review is to demonstrate the lipid-lowering activity and anti-atherogenic effects of natural and semi-synthetic plant sterols/ stanols based on evidence from cell-culture studies, animal studies and clinical trials. Additionally, this review highlights certain molecular mechanisms by which plant sterols/ stanols lower plasma cholesterol levels with a special emphasis on factors that affect the cholesterol-lowering activity of plant sterols/ stanols. The crystalline nature and the poor oil solubility of these natural products could be important factors that limit their cholesterol-lowering efficiency. Several attempts have been made to improve the cholesterol-lowering activity by enhancing the bioavailability of crystalline sterols and stanols. Approaches involved reduction of the crystal size and/or esterification with fatty acids from vegetable or fish oils. However, the most promising approach in this context is the chemical modification of plant sterols/ stanols into water soluble disodium ascorbyl phytostanyl phosphates analogue by esterification with ascorbic acid. This novel semi-synthetic stanol derivative has improved efficacy over natural plant sterols/ stanols and can provide additional benefits by combining the cholesterol-lowering properties of plant stanols with the antioxidant potential of ascorbic acid.



## Diets with high-fat cheese, high-fat meat, or carbohydrate on cardiovascular risk markers in overweight postmenopausal women: a randomized crossover trial

Thorning, T.K., *et al.*, *Am. J. Clin. Nutr.* 3: 573–581, 2015, <http://dx.doi.org/10.3945/ajcn.115.109116>.

Heart associations recommend limited intake of saturated fat. However, effects of saturated fat on low-density lipoprotein (LDL)-cholesterol concentrations and cardiovascular disease risk might depend on nutrients and specific saturated fatty acids (SFAs) in food. We explored the effects of cheese and meat as sources of SFAs or isocaloric replacement with carbohydrates on blood lipids, lipoproteins, and fecal excretion of fat and bile acids. The study was a randomized, crossover, open-label intervention in 14 overweight postmenopausal women. Three full-diet periods of 2-wk duration were provided separated by 2-wk washout periods. The isocaloric diets were as follows: 1) a high-cheese (96–120-g) intervention [i.e., intervention containing cheese (CHEESE)], 2) a macronutrient-matched nondairy, high-meat control [i.e., nondairy control with a high content of high-fat processed and unprocessed meat in amounts matching the saturated fat content from cheese in the intervention containing cheese (MEAT)], and 3) a nondairy, low-fat, high-carbohydrate control (i.e., nondairy low-fat control in which the energy from cheese fat and protein was isocalorically replaced by carbohydrates and lean meat (CARB)). The CHEESE diet caused a 5% higher high-density lipoprotein (HDL)-cholesterol concentration ( $P = 0.012$ ), an 8% higher apo A-I concentration ( $P < 0.001$ ), and a 5% lower apoB:apo A-I ratio ( $P = 0.008$ ) than did the CARB diet. Also, the MEAT diet caused an 8% higher HDL-cholesterol concentration ( $P < 0.001$ ) and a 4% higher apo A-I concentration ( $P = 0.033$ ) than did the CARB diet. Total cholesterol, LDL cholesterol, apoB, and triacylglycerol were similar with the 3 diets. Fecal fat excretion was 1.8 and 0.9 g higher with the CHEESE diet than with CARB and MEAT

diets ( $P < 0.001$  and  $P = 0.004$ , respectively) and 0.9 g higher with the MEAT diet than with the CARB diet ( $P = 0.005$ ). CHEESE and MEAT diets caused higher fecal bile acid excretion than did the CARB diet ( $P < 0.05$  and  $P = 0.006$ , respectively). The dominant type of bile acids excreted differed between CHEESE and MEAT diets. Diets with cheese and meat as primary sources of SFAs cause higher HDL cholesterol and apo A-I and, therefore, appear to be less atherogenic than is a low-fat, high-carbohydrate diet. Also, our findings confirm that cheese increases fecal fat excretion. This trial was registered at [clinicaltrials.gov](http://clinicaltrials.gov) as NCT01739153.

## The relationship of antioxidant components and antioxidant activity of sesame seed oil

Yin Wan, *et al.*, *J. Sci. Food Agric.* 95: 2571–2578, 2015, <http://dx.doi.org/10.1002/jsfa.7035>.

Although sesame seed oil contains high levels of unsaturated fatty acids and even a small amount of free fatty acids in its unrefined flavored form, it shows markedly greater stability than other dietary vegetable oils. The good stability of sesame seed oil against autoxidation has been ascribed not only to its inherent lignans and tocopherols but also to browning reaction products generated when sesame seeds are roasted. Also, there is a strong synergistic effect among these components. The lignans in sesame seed oil can be categorized into two types, i.e. inherent lignans (sesamin, sesamol) and lignans mainly formed during the oil production process (sesamol, sesamolol, etc.). The most abundant tocopherol in sesame seed oil is  $\gamma$ -tocopherol. This article reviews the antioxidant activities of lignans and tocopherols as well as the browning reaction and its products in sesame seed and/or its oil. It is concluded that the composition and structure of browning reaction products and their impacts on sesame ingredients need to be further studied to better explain the remaining mysteries of sesame oil.

## Validation of the omega-3 fatty acid intake measured by a web-based food frequency questionnaire against omega-3 fatty acids in red blood cells in men with prostate cancer

Allaire, J., *et al.*, *Eur. J. Clin. Nutr.* 69: 1004–1008, online first, March 2015, <http://dx.doi.org/10.1038/ejcn>.

The objective of this study was to evaluate the ability of a web-based self-administered food frequency questionnaire (web-FFQ) to assess the omega-3 ( $\omega$ -3) fatty acids (FAs) intake of men affected with prostate cancer (PCa) against a biomarker. The study presented herein is a sub-study from a phase II clinical trial. Enrolled patients

afflicted with PCa were included in the sub-study analysis if the FA profiles from the red blood cell (RBC) membranes and FA intakes at baseline were both determined at the time of the data analysis ( $n=60$ ). Spearman's correlation coefficients were calculated to estimate the correlations between FA intakes and their proportions in the RBC membranes. Intakes of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) were highly correlated with their respective proportions in the RBC membranes (both  $r_s=0.593$ ,  $P<0.0001$ ). Correlation between alpha-linolenic acid (ALA) intake and its proportion in RBC was not significant ( $r_s=0.130$ ,  $P=0.332$ ). Correlations were observed between fatty fish intake and total  $\omega$ -3 FAs ( $r_s=0.304$ ,  $P=0.02$ ), total long-chain  $\omega$ -3 FAs ( $r_s=0.290$ ,  $P=0.03$ ) and DHA ( $r_s=0.328$ ,  $P=0.01$ ) in RBC membranes. This study has shown that the web-FFQ is an accurate tool to assess total long-chain  $\omega$ -3 FAs, EPA and DHA but not ALA intake in clinical trials and epidemiological studies carried out in men with PCa.

## Characterization of the aroma-active, phenolic, and lipid profiles of the pistachio (*Pistacia vera* L.) nut as affected by the single and double roasting process

Rodríguez-Bencomo, J.J., *et al.*, *J. Agric. Food Chem.* 63: 7830–7839, 2015, <http://dx.doi.org/10.1021/acs.jafc.5b02576>.

The pistachio (*Pistacia vera* L.) nut is one of the most widely consumed edible nuts in the world. However, it is the roasting process that makes the pistachio commercially viable and valuable as it serves as the key step to improving the nut's hallmark sensory characteristics including flavor, color, and texture. Consequently, the present study explores the effects of the single-roasting and double-roasting process on the pistachio's chemical composition, specifically aroma-active compounds, polyphenols, and lipids. Results showed the total polyphenol content of increased with the roasting treatment; however, not all phenolic compounds demonstrated this behavior. With regard to the aroma and aroma-active compounds, the results indicated that roasting process results in the development of characteristics and pleasant aroma of pistachio samples due to the Maillard reaction. With regard to lipids, the pistachio roasting treatment reduced the concentration of CN38 diacylglycerides while increasing the amount of elaidic acid.



## Manipulation of lipids in animal-derived foods: Can it contribute to public health nutrition?

Ian Givens, D., *Eur. J. Lipid Sci. Technol.* 117 : 1306–1316, online first, September 2015, <http://dx.doi.org/10.1002/ejlt.201400427>.

Foods derived from animals are an important source of nutrients for humans. Concerns have been raised that due to their SFA content, dairy foods may increase the risk of cardiometabolic disease. Prospective studies do not indicate an association between milk consumption and increased disease risk although there are less data for other dairy foods. SFA in dairy products can be partially replaced by cis-MUFA through nutrition of the dairy cow although there are too few human studies to conclude that such modification leads to reduced chronic disease risk. Intakes of LCn-3 FA are sub-optimal in many countries and while foods such as poultry meat can be enriched by inclusion of fish oil in the diet of the birds, fish oil is expensive and has an associated risk that the meat will be oxidatively unstable. Novel sources of LCn-3 FA such as krill oil, algae, and genetically modified plants may prove to be better candidates for meat enrichment. The value of FA-modified foods cannot be judged by their FA composition alone and there needs to be detailed human intervention studies carried out before judgements concerning improved health value can be made.

## Transgenic plants as a sustainable, terrestrial source of fish oils

Napier, J.A., *et al.*, *Eur. J. Lipid Sci. Technol.* 117 : 1317–1324, 2015, <http://dx.doi.org/10.1002/ejlt.201400452>.

An alternative, sustainable source of omega-3 long chain polyunsaturated fatty acids is widely recognized as desirable, helping to reduce pressure on current sources (wild capture fisheries) and providing a de novo source of these health beneficial fatty acids. This review will consider the efforts and progress to develop transgenic plants as terrestrial sources of omega-3 fish oils, focusing on recent developments and the possible explanations for advances in the field. We also consider the utility of such a source for use in aquaculture, since this industry is the major consumer of oceanic supplies of omega-3 fish oils. Given the importance of the aquaculture industry in meeting global requirements for healthy foodstuffs, an alternative source of omega-3 fish oils represents a potentially significant breakthrough for this production system.

## Ahiflower oil: a novel non-GM plant-based omega-3+6 source

Cumberford, G. and A. Hebard, *Lipid Technol.* 27, Issue 9: 207–210, 2015, <http://dx.doi.org/10.1002/lite.201500044>.

Ahiflower oil, a novel and proprietary dietary oil with the richest effective combined essential fatty acids from a single non-GM plant, has been developed agronomically in a unique vertically integrated “soil to oil” model at commercial scale by Nature's Crops International. Ahiflower oil helps resolve a persistent dilemma in sustainable global omega-3 nutrition from marine sources while dramatically improving dietary omega-3 EPA conversion from plant sources and while supplying beneficial anti-inflammatory GLA not found in fish or algal oils. Having achieved key regulatory clearances in the US and EU, Ahiflower oil is launching in the latter half of 2015 in supplements and foods.

## Reduction of oil absorption during frying

Li, J. and L. Fan, *Lipid Technol.* 27 : 203–205, 2015, <http://dx.doi.org/10.1002/lite.201500040>.

Because of health concerns, consumers prefer fried food with low oil content and good sensory properties. Oil absorption during frying is affected by pretreatment, frying conditions and material properties. This paper reviews the oil absorption mechanism, influence factors and reduction methods during frying, pre-drying and coating methods for reducing oil absorption. It is evident that new frying technology, natural coatings, and combination frying, should ultimately lead to the production of healthier fried foods.

## Crude fat content and fatty acid profile and their correlations in foxtail millet

Zhang, A., *Cereal Chem.* 92: 455–459, 2015, <http://dx.doi.org/10.1094/CCHEM-12-14-0252-R>

Crude fat and fatty acid profile of 35 foxtail millets including seven varieties planted in five different regions of China were studied. The fat content ranged from 3.38 to 6.49% (averaging 4.51%). The major fatty acid in foxtail millets was linoleic acid (averaging 66.68%), followed by oleic acid (averaging 16.11%), palmitic acid (averaging 7.42%), stearic acid (averaging 6.84%), and linolenic acid (averaging 2.48%). Two-way ANOVA showed that fat content was significantly affected by millet variety and cultivation area ( $P < 0.05$ ). Fatty acids including linoleic acid, palmitic acid, stearic acid, and linolenic acid varied significantly in different foxtail millet varieties ( $P < 0.05$ ), except oleic acid ( $P > 0.05$ ). Fatty acids including linoleic acid, oleic acid, palmitic acid, and stearic acid did not change significantly in foxtail millets from different regions ( $P > 0.05$ ), except linolenic acid ( $P < 0.05$ ). Correlation analysis indicated that oleic acid was negatively correlated with palmitic acid and linoleic



acid ( $P < 0.05$ ), and linolenic acid was positively correlated with palmitic acid and linoleic acid but negatively correlated with stearic acid ( $P < 0.05$ ). The research showed that millets with good fat composition can be obtained through breeding techniques or cultivation management.

## Lipid Oxidation

### Active edible coating from chitosan incorporating green tea extract as an antioxidant and antifungal on fresh walnut kernel

Sabaghia, M., *et al.*, *Postharvest Biol. Technol.* 110: 224–228, 2015, <http://dx.doi.org/10.1016/j.postharvbio.2015.08.025>.

The effects of coatings made from chitosan (Chi) incorporating green tea extract (GTE) on lipid oxidation, fungal growth, and sensory properties of walnut kernels were studied. The Chi powder and GTE obtained from green tea were combined to obtain the final concentrations of Chi-GTE: 5-0, 5-5, 5-10, 10-0, 10-5 and 10-10 g L<sup>-1</sup> in an aqueous coating solution. Effective inhibition of lipid oxidation and fungal growth during storage of walnut kernels (18 weeks) was possible using the Chi10 coating combined with GTE. Different proportions of GTE had no significant effect ( $p < 0.05$ ) on lipid oxidation. Fungal growth was not detected during the storage period with Chi10 and all different proportions of GTE. Throughout the storage duration, coatings without GTE showed no significant ( $p < 0.05$ ) effect on sensory properties; however Chi10-GTE10, was significantly ( $p < 0.05$ ) unacceptable by panelist. The results suggested that Chi10-GTE5 could prolong the shelf life of walnut kernels.

### From alga to omega; have we reached peak (fish) oil?

Clayton, P.R. and S. Ladi, *J. R. Soc. Med.* 108: 351–357, 2015, <http://dx.doi.org/10.1177/0141076815599673>.

While the Inuit diet was highly cardio-protective and consuming oily fish within a Western diet is to a lesser degree, the case for purified fish oil supplements is less convincing. Purification of fish oil removes lipophilic polyphenols which likely contribute to the health benefits of oily fish; leaving the  $\omega 3$  highly unsaturated fatty acids exposed and prone to conferring oxidative and inflammatory stress. The authors believe that due to such issues as dietary shift, it may now be inadvisable to prescribe or sell purified  $\omega 3$  highly unsaturated fatty acids supplements, unless the appropriate co-factors are included.

### Development and characterization of multiple emulsions for controlled release of *Trichilia catigua* (Catuaba) extract

Alesandra, A., *et al.*, *Pharm. Dev. Technol.*, online only, September, 2015, DOI: 10.3109/10837450.2015.1081611.

Considering the antioxidant activity of the *Trichilia catigua* extract (TCE), the aim of the current study was to develop and characterize W/O/W multiple emulsions containing different vegetable oils as a platform to deliver a TCE. The extract displayed antioxidant activity (IC<sub>50</sub>) of 4.59  $\mu\text{g/mL}$  and total phenol content (TPC) of 50.84%. Formulations were prepared by the phase-inversion emulsification method and analyzed for morphological appearance, pH, conductivity, droplet size and distribution, content of active, rheological properties, in vitro release, skin permeation, and stability. Formulations prepared with canola oil were selected and displayed regular morphology, mean diameter 2.77  $\mu\text{m}$  (without TCE), 3.07  $\mu\text{m}$  with 0.5% and 3.23  $\mu\text{m}$  with 1.0% TCE. Rheometry (flow) showed pseudoplastic behavior with minimal thixotropy for both systems. TCE could be released from emulsions containing 1.0% and 0.5% TCE in a controlled manner for 16 and 23 h, respectively. The emulsions allowed good retention of TCE in the skin (stratum corneum, epidermis, and dermis). In a 180-d assessment of accelerated chemical stability, TPC was more reduced for the emulsions at 40 °C; other parameters remained stable. Multiple emulsions containing TCE were developed, exhibited good characteristics, and may be considered for future investigations as anti-aging formulations for the skin.

### Oxidative stability of a heme iron-fortified bakery product: effectiveness of ascorbyl palmitate and co-spray-drying of heme iron with calcium caseinate

Alemána, M., *et al.*, *Food Chem.*, online first, September, 2015, <http://dx.doi.org/10.1016/j.foodchem>.

Fortification of food products with iron is a common strategy to prevent or overcome iron deficiency. However, any form of iron is a pro-oxidant and its addition will cause off-flavours and reduce a product's shelf life. A highly bioavailable heme iron ingredient was selected to fortify a chocolate cream used to fill sandwich-type cookies. Two different strategies were assessed for avoiding the heme iron catalytic effect on lipid oxidation: ascorbyl palmitate addition and co-spray-drying of heme iron with calcium caseinate. Oxidation development and sensory acceptability were monitored in the cookies over one-year of storage at room temperature in the dark.

The addition of ascorbyl palmitate provided protection against oxidation and loss of tocopherols and tocotrienols during cookies preparation. In general, ascorbyl palmitate, either alone or in combination with the co-spray-dried heme iron, prevented primary oxidation and hexanal formation during storage. The combination of both strategies resulted in cookies that were acceptable from a sensory point of view after 1 year of storage.

## Microstructure and long-term stability of spray dried emulsions with ultra-high oil content

Sarkara, A., *et al.*, *Food Hydrocolloids* 52: 857–867, online first, January 2016 print date, <http://dx.doi.org/10.1016/j.foodhyd.2015.09.003>.

The aim of this study was to investigate the microstructure and long-term mechanical as well oxidative stability of a new class of spray dried emulsion containing ultrahigh oil content. Emulsion (20 wt% oil) stabilized by whey protein (1 wt%) was thermally cross-linked at 82 °C for 10 min and spray dried without any additional wall materials using inlet/outlet air temperature of  $105 \pm 2/65 \pm 2$  °C, respectively at a pilot scale. Confocal micrograph showed cohesive cross-linked whey protein film present at the oil-water interface and at the powder surface stabilizing the oil powder particles containing 95.3 wt% oil. The mean droplet size of parent emulsion (0.21, 0.38, 0.76, 2.31  $\mu\text{m}$ ) significantly influenced the mechanical stability of the resulting oil powder in terms of oil leakage (2.73, 0.93, 4.1, 7.54 wt%) upon compaction. Scanning electron microscopy revealed the level of surface oil and porous “sponge” like internal microstructure of the oil powder with polyhedral, closely packed droplets. Strong correlations existed between the mechanical properties of the oil powder and the oxidative stability over 5 months. The kinetics of oxidation of oil powder was higher than that of corresponding bulk oil with or without added antioxidants as evidenced by evolution of primary oxidation products (hydroperoxides) and secondary oxidation products (hexanal). This might be due to the multi-step processing (e.g. homogenization, thermal cross-linking, spray drying) as well as inability of the cohesive but permeable protein matrix to protect the ultra-high content of oil droplets from diffusion of oxygen and prooxidants.

## Influence of oil extraction method on properties of canola biodiesel, epoxies, and protein-based plastics

Wajira, A., *et al.*, *Ind. Crops Prod.* 77: 133–138, online first, December 2015 print date, <http://dx.doi.org/10.1016/j.indcrop.2015.08.050>.

Canola oil was extracted using a liquid carbon dioxide extraction process (LCE), laboratory screw-pressing, and industrial solvent extraction. Oilseed meal generated from these methods was used to extract proteins for protein-based plastics, and the oil was used to produce both epoxy resins and biodiesel. Protein isolates obtained from the LCE-generated meal produced plastics with higher toughness and elongation, but lower tensile strength and modulus, than

those using meal obtained from screw pressing and solvent extraction. The oils extracted using the LCE process produced cured epoxy resins with higher flexural modulus than those produced from solvent-extracted oil. However, dynamic mechanical properties such as storage and loss modulus showed no significant differences with respect to oil extraction method. Oils obtained from extraction methods had similar fatty acid profiles and produced biodiesel with no significant differences in properties.

## Increasing carotenoid bioaccessibility from yellow peppers using excipient emulsions: impact of lipid type and thermal processing

Liu, X., *et al.*, *J. Ag. Food Chem.*, accepted online, September 2015, <http://dx.doi.org/10.1021/acs.jafc.5b04217>.

Many phytochemicals from fruits and vegetables exert biological activities that may be beneficial to human health, but these benefits are not fully realized because of their poor oral bioavailability. The objective of this research was to establish the potential of excipient emulsions to increase carotenoid bioaccessibility from raw and cooked yellow peppers using a gastrointestinal model that included oral, gastric, and intestine phases. The influence of oil type (medium chain triglycerides, MCT; long chain triglycerides, LCT; and indigestible orange oil, OO) on microstructural changes, particle properties, lipid digestibility, and carotenoid bioaccessibility was investigated. Oil type had a major impact, with carotenoid bioaccessibility decreasing in the following order: LCT > MCT > OO > control (no oil). Conversely, thermal treatment (raw versus boiled) had little influence on carotenoid bioaccessibility. These results will facilitate the rational design of excipient emulsions that boost the bioavailability of phytochemicals in fruits and vegetables.

## Long-chain fatty acids elicit a bitterness-masking effect on quinine and other nitrogenous bitter substances by formation of insoluble binary complexes

Ogi, K., *et al.*, accepted online, September 2015, <http://dx.doi.org/10.1021/acs.jafc.5b03193>.

We have previously found that fatty acids can mask the bitterness of certain nitrogenous substances through direct molecular interactions. Using isothermal titration calorimetry, we investigated the interactions between sodium oleate and 22 bitter substances. The hydrochloride salts of quinine, promethazine and propranolol interacted strongly with fatty acids containing 12 or more carbon atoms. The  $^1\text{H-NMR}$  spectra of these substances obtained in the presence of the sodium salts of the fatty acids in dimethyl sulfoxide, revealed the formation of hydrogen bonds between the nitrogen atoms of the 8 bitter substances and the carboxyl groups of the fatty acids. When sodium laurate and the hydrochloride salt of quinine were mixed



in water, an equimolar complex was formed as insoluble heterogeneous needle-like crystals. These results suggested that fatty acids interact directly with bitter substances through hydrogen bonds and hydrophobic interactions to form insoluble binary complexes, which mask bitterness.

## Industrial applications

### Alternative solvents for *Moringa oleifera* seeds extraction

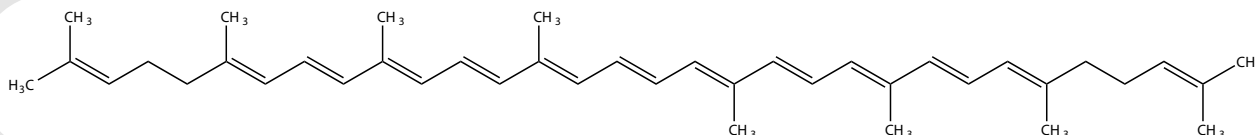
Efeovbokhan, V.E., *et al.*, J. Applied Sci. 15: 1073–1082, 2015, <http://dx.doi.org/10.3923/jas.2015.1073.1082>.

*Moringa oleifera* is a versatile plant and has wide applicability. Two critical factors that make the oil more readily available for use are its ease of processing techniques and production rate. Solvent extraction using various solvents is common and is usually more efficient than mechanical presses. Hence investigating the suitability of three solvents-hexane, Isopropyl Alcohol (IPA) and Petro-

leum Ether (PE) in the extraction of *Moringa* seeds from northern (sample 1) and southern (sample 2) parts of Nigeria was carried out using a soxhlet extractor between 2 and 12 h. The percentage yield of oil from the two samples was found to be dependent on the solvent used, the residence time and the source of the seed sample. Petroleum ether gave the highest yield of 49.38 and 37.57%, next was hexane with 44.94 and 34.71% while isopropyl alcohol gave 36.39 and 28.43%, for samples 1 and 2, respectively. For all solvents, sample 1 produced higher oil yield. The percentage oil yield increased with time reaching an optimum at between 8–10 h. From chromatographic analysis, besides other trace components, the predominant fatty acids present in the *Moringa oleifera* oil include oleic, stearic, palmitic, linoleic and palmitoleic acids. The overall composition of the oil indicated higher levels of unsaturated than saturated acids with oleic acid having the highest percentage composition of 68.8% in all the extracted oil samples. The results obtained from this investigation showed that the alternative solvents (IPA and PE) considered can potentially substitute n-hexane in *Moringa* oilseed extraction.

**Continued on page 669**

## FAST FACT



*Editor's Note: This excerpt is from Ingredients: A Visual Exploration of 75 Additives & 25 Food Products by science writer Steve Ettlinger and photographer Dwight Eschliman. This new book from Regan Arts (September 29, 2015; ISBN 978-194139331-4; \$35.00) demystifies some of the more controversial and common food additives. See [www.reganarts.com](http://www.reganarts.com) for more information.*

Every natural color has its most common, well-known food source, but bright red lycopene outdoes the others: It is almost exclusively linked with tomatoes, even though it is also found in paprika, pink grapefruit, guava, watermelon, asparagus (yes, asparagus), and rose hips (as well as a fungus, *Blakeslea trispora*). Interestingly enough, neither cherries nor strawberries have it; many red fruits are colored by anthocyanins. Only lycopene extracted from tomatoes is allowed to be used as a food colorant in the United States. Generally, this extraction involves a large chemical plant and soaking tomato pomace left over from making juice or sauce in a volatile solvent that is boiled off (chemically synthesized lycopene is acceptable as a nutritional supplement). Lycopene is a carotene, and as such shares a molecular formula and the E-number 160 (though with a differentiating letter) with the other carotenes, especially beta-carotene.

A phytochemical, lycopene's biological role involves helping photosynthesis along and creating beta-carotene. It joins a list of other natural pigments that are also good for you, including chlorophyll, beta-carotene, and anthocyanins. In fact, despite the health benefits being quite unclear, many people make a special effort to eat cooked tomatoes in order to consume as much lycopene as they can.

One of the most wonderful discoveries of food science was inspired by research into the Mediterranean diet in the early 1950s. Scientists noticed that many of the residents of a southern Italian village led long and healthy lives without much need for medical care. Among the many healthful things they ate (like wine, lots of fruit and veggies, and very little meat) were tomatoes cooked in olive oil. Now we have come to understand that lycopene is only soluble, and thus made more nutritious to us, in oil (or solvents). Raw tomatoes offer much less nutrient bioavailability. Without really knowing it, the Italian villagers had chosen the best way to make tomatoes' nutrients available through something akin to natural selection. (As of the 2000s, residents in the same area are not doing as well, attributed in part to their adoption of a modern sedentary lifestyle and overprocessed-food eating habits.)



# RENDERING IS RECYCLING



RENDERING IS AN ENVIRONMENTALLY FRIENDLY WAY TO RECYCLE MATERIAL THAT WOULD BE WASTED

## WHAT MATERIALS ARE RENDERED?

### Packing Plants

**147.2 MILLION** head of cattle, calves, hogs & sheep are slaughtered annually in the US



**10 BILLION** chickens and turkeys are processed each year in the US



**APPROXIMATELY 50%** of the animal is considered inedible by Americans and goes to renderers including: bones, fat, blood, feathers & some internal organs



### Farms

Some animals die on the farm from injury, old age, or other issues. Renderers prevent this from becoming a public health hazard by eliminating pathogens in the rendering process. Fallen animals only represent about 4.5% of rendered product



### Grocery Stores generate

Super Foods

**1.92 BILLION POUNDS**

of scraps, fat, bone, expired meat & used cooking oil annually

### Renderers collect



**4.4 BILLION POUNDS**

of used cooking oil per year in the U.S. and Canada

## WHAT ARE THE PRODUCTS OF RENDERING?

### Renderers collect:

**56 BILLION POUNDS**

of raw materials every year in the U.S. and Canada



If all renderable product was sent to the landfill, all available space would be used in

**4 YEARS**

### Renderers recycle these materials into:

**10 BILLION POUNDS**

of fat and oil products



**& 9 BILLION POUNDS**

of protein products annually



### Rendered products are ingredients in:



Pet & livestock feed



Biotfuel



Fertilizer

Plus Other products like soaps, lubricants, detergents, and more.

## RENDERING IS Green!

### Rendering is:

the process of breaking down animal by-products into fats & proteins



### Rendering is recycling!

Carbon- and nitrogen-rich materials are recycled into

**USEABLE PRODUCTS**



## RENDERING IS SAFE & ESSENTIAL

- Protects the environment
- Protects human health
- Protects animal health
- Sustainable and contributes to sustainability of animal agriculture
- Essential link in food chain



### Rendering reduces greenhouse gases.

Carbon dioxide, methane, and other greenhouse gas emissions from natural decomposition like in a compost pile or landfill are avoided.



A single decomposing dairy cow releases **1.2 METRIC TONS** of carbon dioxide.

Rendering avoids this!

Rendering these animal tissues has the same effect on greenhouse gas emissions as removing



**12,263,316 CARS FROM THE ROAD**



Continued from page 667

## Synthetic biology

### Metabolic engineering of oilseed crops to produce high levels of novel acetyl glyceride oils with reduced viscosity, freezing point and calorific value

Liu, J., et al., *Plant Biotechnol. J.* 13 :858–865, 2015, <http://dx.doi.org/10.1111/pbi.12325>.

Seed oils have proved recalcitrant to modification for the production of industrially useful lipids. Here, we demonstrate the successful metabolic engineering and subsequent field production of an oilseed crop with the highest accumulation of unusual oil achieved so far in

transgenic plants. Previously, expression of the *Euonymus alatus* diacylglycerol acetyltransferase (*EaDacT*) gene in wild-type *Arabidopsis* seeds resulted in the accumulation of 45 mol% of unusual 3-acetyl-1,2-diacyl-sn-glycerols (acetyl-TAGs) in the seed oil (Durrett et al., 2010 PNAS 107:9464). Expression of *EaDacT* in *dgat1* mutants compromised in their ability to synthesize regular triacylglycerols increased acetyl-TAGs to 65 mol%. Camelina and soybean transformed with the *EaDacT* gene accumulate acetyl-triacylglycerols (acetyl-TAGs) at up to 70 mol% of seed oil. A similar strategy of coexpression of *EaDacT* together with RNAi suppression of *DGAT1* increased acetyl-TAG levels to up to 85 mol% in field-grown transgenic Camelina. Additionally, total moles of triacylglycerol (TAG) per seed increased 20%. Analysis of the acetyl-TAG fraction revealed a twofold reduction in very long chain fatty acids (VLCFA), consistent with their displacement from the sn-3 position by acetate. Seed germination remained high, and seedlings were able to metabolize the stored acetyl-TAGs as rapidly as regular triacylglycerols. Viscosity, freezing point and caloric content of the Camelina acetyl-TAG oils were reduced, enabling use of this oil in several nonfood and food applications.



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# FT-NIR spectroscopy: a valuable tool for the edible oil industry

Dagmar Behmeri

Near Infrared (NIR) Spectroscopy has been recognized for decades as a well-established technique in the agricultural sector and is becoming more and more important in the food industry. Modern multi-purpose NIR spectrometers based on Fourier-Transform technology, so-called FT-NIR spectrometers (Fig. 1), can analyze both liquid and solid samples and are the ideal tool for the non-destructive and rapid analysis of oilseeds and finished oils throughout the entire manufacturing process. In addition, dedicated applications, such as the storage analysis of olive oil or monitoring of degradation processes in used frying oils, have been explored, showing a high potential for increasing the quality of oils, raising the efficiency of production, and providing cost savings.



**FIG. 1.** FT-NIR spectrometer MPA for the analysis of oils and oilseeds FT-NIR measures the absorption of near-infrared light of the sample at different wavelengths. The recorded spectrum is characterized by overtones and combinations of the fundamental molecular vibrations of molecules containing C-H, N-H or O-H groups, making FT-NIR spectroscopy first choice for the analysis of organic materials, such as oilseeds and edible oils.



**FIG. 2.** Analyzing soybeans in less than a minute for oil content and fatty acid composition

FT-NIR offers many advantages over classical wet-chemical and chromatographic analyses. It is quick, cost-effective, and safe, since no hazardous chemicals are used. It also eliminates the typical error sources of classical lab methods, such as those that occur during the sample preparation stage. With only one measurement, multiple components can be analyzed in less than one minute for both solid and liquid samples (Fig. 2). This will result in tighter quality control with higher sample throughput and lower analysis cost.

Although FT-NIR spectroscopy is not a technology for trace analysis of components such as toxins, it will help the producer to constantly monitor the quality of goods along the production chain—from checking the incoming raw materials to quality testing of the finished product. With the implementation of the US FDA Food Safety Modernization Act, this will soon be as important for the food industry as it is already today for the pharmaceutical industry.

## RIGHT FROM THE BEGINNING: ANALYSIS OF OILSEEDS

The analysis of oilseeds plays a major role in ensuring the right quality of food as well as of agricultural products. FT-NIR spectroscopy offers solutions for oil producers as well as for breeders. To optimize the oil pressing process, oil seeds, as well as the intermediate products, can be analyzed for oil and moisture content. The pressing process can therefore be optimized, leading to higher plant efficiency.

Looking at the plant breeding process, at each stage the breeder needs to choose the best seeds for propagation from one generation to the next. In the past, the breeder had to sacrifice some of these valuable seeds to test for the traits of interest. FT-NIR has become increasingly popular in the plant breeding-sector, as it is a non-destructive method of analysis, capable of measuring many

of the important traits. It allows the breeder to analyze bulk as well as single seeds quickly to determine the optimum seeds.

Depending on the use of oilseeds, different parameters are of interest. For example, FT-NIR spectrometers can analyze the content of oleic and linoleic acid inside the seed to determine high-oleic sunflower seeds. Canola breeders can obtain valuable additional information, such as fatty acid concentrations, including erucic acid as well as the glucosinolate content. These are crucial parameters for the palatability of the finished product.

## TAKING A CLOSER LOOK: QC OF FINISHED OILS

Since FT-NIR spectroscopy is capable of looking at the complete composition of the molecules, the composition of the oil in terms of fatty acid profile (C16:0, C18:0, C18:1, C18:2, C18:3, etc.) can be determined. This is generally important for the identification of different oils as well as for quality control.

Iodine Value (IV) is a good measure to predict the physical appearance and the processability of the fat. IV is defined as the grams of Iodine absorbed by 100g of fat or oil. The higher the IV is, the softer the fat. IV analysis by FT-NIR is acknowledged by AOCS (method Cd1e-01). Other parameters, such as oil acidity, can also be determined simultaneously from a single measurement. The more fatty acids are freed from the glycerine backbone, the more acidic the oil becomes. This is expressed as the free fatty acid (FFA) content. Moreover, the health-relevant trans-fatty-acid (TFA) content as well as physical parameters like color values or dropping point can be determined.

## MEDITERRANEAN SUPERFOOD: OLIVES AND OLIVE OIL

The vast majority of olives grown all over the world are used for the manufacture of olive oil. The value of an olive crop is mostly determined by the oil content. Thus, determining the exact oil content is essential for farmers and industry alike to estimate the value of the collected olives and to optimize future harvests. Traditionally, the oil content is determined by Soxhlet extraction. Since Soxhlet extraction is a wet chemical method requiring a large amount of solvents, it is becoming increasingly unacceptable. Moreover, the results are often operator dependent, and the procedure is slow compared to spectroscopic methods.

The amount of free fatty acids (FFA), expressed as acidity is, apart from organoleptic evaluations, a major criterion for the classification of the olive oil into the trade categories of “virgin” and “extra virgin”. According to the current EC regulation 1513/01, the maximum level of free acidity of an extra virgin olive oil must not be higher than 0.8 gram per 100 grams (0.8 %). The acidity level in the oil increases if the olives were



**FIG. 3. Rapid oil analysis with FT-NIR using disposable vials**

attacked by insects, were collected from the ground instead of straight from the tree, or if there was a time lag between harvests and processing.

However, classification of “extra virgin” is not the only important factor to evaluate the quality of an olive oil. Two recently developed methods, the amount of 1,2-diacylglycerols (DAGs) and the ratio of pyropheophytins (PPP), describing the degradation of the oil’s chlorophyll to pyropheophytin, have shown promising results in determining whether an oil is old, has been poorly stored, or possibly even refined. DAGs and PPP have been included by the Australian Olive Association in their regulations and are currently under investigation by the Olive Oil Commission of California (OOC). Both parameters can be analyzed with FT-NIR in 30 seconds (Fig. 3).

## TO GET WHAT YOU PAY FOR: TESTING FOR ADULTERATION

A common problem not only for the olive oil industry is the adulteration of high-priced olive oil with cheaper vegetable oils, such as sunflower oil or hazelnut oil. Today’s public awareness of the health benefit of olive oil makes adulteration economically attractive. From a chemical perspective, most oils are very similar and a blend is difficult to identify with common quick tests such as refractive index measurements. However different oils vary in their fatty acid profile, and FT-NIR spectroscopy can offer a valuable tool for determining other types of oil in olive oil down to a low-percentage range. A study of different olive oils adulterated with hazelnut oils showed a good correlation of FT-NIR results with the gravimetric method.

Another issue regarding adulteration is to determine the origin of plant oils to assign them to geographic regions for certification purposes. One example is the registered designations of origin (RDO) nomination of extra virgin olive oils of known region, province, and cultivar. Here FT-NIR is currently under review if it can help identifying fraud. First studies indicate that FT-NIR spectroscopy, together with chemometric algorithms, shows promising results.

## HOT TOPIC: MONITORING OF FRYING OIL QUALITY

In recent years, fried food has been stigmatized due to health concerns raised by various studies relating fried food to cardiovascular disease, obesity, diabetes, and other negative conditions. However, the fact is that the quality of the frying fat heavily influences the quality of the finished product. This becomes clear if you consider that a potato in its raw state has a fat content of less than 1%. Potato chips (crisps), on the other hand, can contain up to 35% fat, which clearly derives from the migration of the frying medium into the food.

This makes the importance of thorough quality control—in fast food restaurants and huge frying operations alike—very obvious. Whereas small frying operations mostly rely on experience and occasional quick-test solutions, such as visual test strips or simple conductivity measurements, it is of utmost importance to constantly monitor the frying medium in large-scale industrial fryers with high turnovers. Any deviation from the guaranteed quality will lead to loss in consumer trust and potential claims. Consequently, tight control of oil quality will offset the cost of replenishing the oil.

There are several recognized parameters that correlate well with the quality of the used frying oil, such as acid value (amount of free fatty acids) and anisidine value. Since most of the degradation products of frying oils are of polar character, the amount of total polar compounds (TPC) is a reliable quality indicator, together with the amount of di- and polymeric triacylglycerols (DPTG), which are formed during the frying process.

However, no single parameter is able to explain the frying process or the final food quality sufficiently. Ideally, a combination of indices giving a complete picture of the chemical substances formed during frying would be desirable. Hence, FT-NIR spectroscopy offers a fast, non-destructive, and cost-effective method for the analysis of used frying oils. This was acknowledged by the Deutsche Gesellschaft Für Fettwissenschaft (DGF), or German Society for Fat Science, which issued the Standard Method “FT-NIR Spectroscopy: Screening analysis of used frying fats and oils for rapid determination of polar compounds, polymerised triacylglycerols, acid value and anisidine value [DGF C-VI 21a (13)]” in September 2013.

## CONCLUSION

FT-NIR spectroscopy has the potential to substitute a wide range of classical analysis methods in the edible oil industry. Bruker Optics offers an array of ready-to-use calibrations for edible fats and oils as well as for oil seeds and olives to enable a quick and efficient start into this exiting technology. For more information, please contact [info.bopt.us@bruker.com](mailto:info.bopt.us@bruker.com) or visit <http://www.bruker.com/edible-oils>.

*Dagmar Behmer, member of Euro Fed Lipid and DGF, has an MSc degree in analytical chemistry and is responsible for the international support in the NIR & Process Technology department of Bruker Optics in Germany. Today, she is looking back on over 20 years of experience with NIR spectroscopy, focusing on food and agricultural applications.*



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# Common editorial mistakes in journal submissions

Michael K. Dowd

Congratulations; you've finished your experiments, reached interesting conclusions, and started to draft your report. Now it needs to be polished to perfection before sending it to the journal.

In the current state of scientific publishing, few publishers employ technical editors to revise reports. Instead, publishers state in the Instructions to Authors that this is the author's responsibility. While researchers generally take great efforts to design experiments, collect data, and generate results, they rarely put much into the last step of the process—communicating the results to others. Consequently, the quality of journal submissions over the years has deteriorated, much to the annoyance of reviewers and editors—and the unfortunate readers who wrestle with poorly written reports after publication. In the final part of this series, I focus on editorial problems frequently observed in submissions to the *Journal of the American Oil Chemist's Society (JAOCS)*. Grammatical errors, word misuse, wordiness, imprecise or ambiguous language, abbreviation misuse, and scientific style errors are all issues.

- In today's "Wild West" of scientific publishing, it is the author's—not the publisher's—responsibility to make sure that an accepted paper is well-written and in correct English.
- Given that most authors are not grammarians or technical editors, and also that English may not be their first language, it is easy to understand why the quality of journal submissions has deteriorated.
- In part three of our series on scientific publishing, JAOCS Senior Associate Editor Michael Dowd describes common editorial problems that regularly find their way into submitted manuscripts.





## GRAMMATICAL ISSUES

In the first article of this series, I mentioned the importance of clarity. Grammatical issues relate to this, in part, because of reader expectations. For instance, readers expect singular nouns to be followed by singular verbs. If that is not what happens, they can become confused about what was intended. I frequently find this error in two forms. First, it occurs when a series of qualifying elements (often of questionable value) is placed between the subject and verb, such that the subject gets lost and the verb is made to agree with a closely placed noun, as in: “The total value of all the products, including A, B, and C, *were* also calculated.” Reading the sentence without the qualifiers helps one see that the subject, “value,” is singular and requires the singular verb, “*was*.” Second, the problem also occurs when a number with units is the subject of a sentence. In this case, the verb should generally be singular, as in “Ten milliliters of A *was* added to B.”

Other common grammatical errors to look for include double negatives, sentence fragments, pronouns without clear antecedents, capitalization and punctuation issues, and so on. There is no easy fix for these issues except to spend a little time to understand the principles and to edit, edit, and edit.

Dangling modifiers, generally participial but also prepositional and infinitive phrases, are more problematic. Dangling modifiers

are words or phrases that do not connect logically to a noun in a sentence. At the very least, they force the reader to assume something about what the author was trying to say. At worse, they result in head-scratching ambiguities. The problem occurs frequently in technical writing because of the tendency to write in an impersonal manner. Consider the participial phrase in the sentence, “The ionization data was collected using mass spectrometry.” As there is no noun in the sentence capable of *using spectrometry*, no English instructor would accept this sentence as correct. Yet, you will find this construction every month in all three AOCS journals. Compare the sentence with “*We* collected ionization data using mass spectroscopy” or “Using a mass spectrometer, we collected ionization data.” Grammatically, these sentences are fine, although many scientists would try to avoid the use of “*we*” in their writing. The solution to this dilemma is not to use this construction excessively.

The pretentiousness in the use of a personal pronoun can be toned down to a degree by burying the pronoun within the sentence, as in the second example above. Alternatively, the spectrometer can be made the subject of the sentence, as in “A mass spectrometer was used to collect ionization data.” One could also consider substituting a preposition for the participle, i.e., “The ionization data was collected *with* a mass spectrometer,” which seems less

CONTINUED ON NEXT PAGE





offensive. Much more could be said on this issue, but I'll leave the topic with my one of my favorites borrowed from Schoenfeld [1]: *The rabbits were observed using binoculars.* My hope is that the rabbits also write better sentences.

## WORD MISUSE

There are too many examples to cover in this article, but here are a few. "Since" and "while" have strong connotations to time but are frequently used where "because" or "although" are better. "Unique" describe an absolute, so it is not possible for something to be *very* or *more* unique. Used correctly, the preposition "via" refers to a pathway, not a process. One can travel from A to C *via* B but not from A to C *via* a train. Nor can compounds be separated *via* chromatography. Avoid "and/or", which Bernstein [2] calls "a verbal and mental monstrosity." Instead, consider whether "and" or "or" alone will do. If not, consider "A or B or both."

Some words are closely related in meaning but not interchangeable, such as "fewer/less," "may/might," "continual/continuous," and "which/that." Many technical writing guides have a section or appendix that discusses such word pairs and how to choose the correct word [3,4].

## ABBREVIATIONS (AND ACRONYMS)

Although abbreviations are convenient shorthand tools for an author, they can be an annoyance for readers. Hence, authors should use these with care. Some abbreviations (DNA, NMR, NaOH, diam., e.g., and etc., and so on) can be used without definition. Generally, if it is an entry in the dictionary, you can use it without definition. Otherwise, abbreviations must be defined on first use and used consistently thereafter in the report. If an abbreviation is not used frequently, delete it. There is simply too little to gain for the reader, who has to store these extra bits of code in memory. Finally, use abbreviations sparingly. Overuse will make the document read as if it is in a different language. Also, because the abstract and the report are different documents, abbreviations must be defined and used independently in each.

## WORDINESS

Verbosity, which frequently results in ambiguous, poorly constructed, and convoluted sentences, is one of the biggest problems I see in submitted research reports. Big words, qualifiers, and long sentences sound grandiose, but can make work unreadable. The most effective technical writing is clear, uncluttered, and precise. This does not mean that long sentences are necessarily bad or poorly written, but that weak and imprecise writing is often characterized by wordiness. The English vocabulary is blessed with many words that can be used to convey subtle shades of meaning. Use the best word, but if a choice exists between a big and a small word, use the small word.

Superfluous words tend to weaken the words that surround them. Use adjectives only when they are needed for clarity, and be sparing with adverbs. Every "clearly," "very," "quite," "fairly," "effectively," and "relatively" in your draft should be viewed with suspicion. "Clearly" deserves special mention, as it is often used to gloss over points that may not be *clear* at all. While on the topic of adverbs, I'll mention the split infinitive. In line with the above discussion, this potential grammatical problem can often be solved simply by omitting the adverb. At times, however, the adverb does contribute something, and it may not be easy to reposition the modifier within the sentence to produce the same effect. Hence, sometimes it is better to accept the construction rather than generate an awkward sentence. Just keep this practice to a minimum.

Use nouns that evoke images, and verbs that evoke actions. Many nouns, such as "factor," "component," "approach," and (one of my pet peeves) "efficiency," do not evoke images. Although all of these words have their place, they are frequently used by writers having trouble saying what they mean. Pay attention to nouns that are derived from verbs, such as "measurement," "establishment," and "formation." They often force writers to use a weak verb, such as "resulted" or "produced," and prepositional fillers, all of which weaken sentences. Compare for example "The separation of A and B was effected by the addition of C" with "A was separated from B by adding C." On the topic of verbs, the active voice is preferable because it is shorter and stronger. The passive voice is often necessary, but it is frequently overworked in technical writing.

Many meaningless phrases creep into scientific writing. Introductory elements such as "It is important to note that ..."

or “It should be stated that ...” add nothing and should be deleted in all instances. Notice these examples start with the pronoun “it,” but what the pronoun actually refers to is not clear. Be sure that every “it” in your document is attached unambiguously to an antecedent. The same goes for “this” when it is used as a pronoun. Similarly, many common phrases can be shortened. Instead of “a considerable number of,” substitute “many.” Consider replacing “based on the fact that,” with “because.” Many books on technical writing [2,4] include tables of weak expressions to avoid. Finally, paragraphs frequently start with wasted sentences. “The data are in Table 2,” says very little. Alternatives include “Table 2 shows that A increased proportionately with B” or consider “A increased proportionately with B (Table 2).”

## COMPARISONS

Comparisons cause a lot of difficulty in technical writing. When an introductory phase is used as part of a comparison, it should refer to the subject of the sentence, as in: “Unlike A and B, C was reactive.” Be sure that you are comparing equivalent things. For example, “The UV absorbance bands of A were sharper than *those* [meaning the absorbance bands] of B.” Do not place words in between idioms, such as “different from” or “similar to”. Note that there is a difference between “compared to,” which is used to note similarities, and “compared with,” which is used to note differences. My advice regarding comparisons is to write out the full comparison in the first draft then see what can be left out or shortened without loss of clarity during editing.

## TECHNICAL STYLE ISSUES

Although science writing should focus on clarity and is benefited by simplicity, there is a style associated with chemical publications. Journal conventions often exist for the naming of compounds, for expressing numbers and units of measure (e.g., 20 mL/sec or 20 ml/s or 20 mL s<sup>-1</sup>, etc.), for describing mixtures (e.g., methanol/acetone 10:1 v/v), for hyphenation of chemical prefixes, for the use of en dashes to combine equal elements (e.g., Michaelis–Menton), etc. These issues all form parts of the style of chemistry-based reports, but are often afterthoughts in manuscript submissions. Some guidance may be found in the journal’s Instruction for Authors but often this will not cover all issues. A good general guide for these conventions is *The ACS Style Guide* (2006) [5]. Although AOCS journals differ on a few points from the recommendations given in this book, the differences are minor. Following this guide and you will be almost perfect. The editors will be thankful and will happily worry about the rest. Admittedly, scientific writing is not an easy process. The subject matter conveyed is often complex and full of subtle distinction and trying to relate this to others can be taxing. Few writers generate concise sentences at the first draft; good writing results from editing and then more editing. A couple of strategies, however, can help. First, let early drafts sit for a period of time. Problems that

initially were invisible often become apparent. I usually cringe on reading my early drafts, and it spurs me to go back to editing. Do this as many times as necessary until you can read the report fresh and think it is perfect. Once you are at this point, give it to others to read. Find colleagues willing to be honest and yes even brutal. I’m always amazed at what others find that I should have identified. It can only help when the report is submitted to a journal for review.

Finally, help is available on many of the above points. Most journal publishers now provide on-line resources. The Springer Academy website, e.g., provides a series of writing tutorials. Additionally, many books are available on technical writing. I have cited several of these above and in the past two articles. In addition, I appreciate the book *The Craft of Scientific Writing* by Alley [6] for its chapters on being concise and forthright, and its discussions on strong and weak writing. For the importance of punctuation, consider the book *Eats, Shoots & Leaves* by Truss [7].

I’ll conclude this series by saying there are many benefits to a well-written submission. Reviewers and editors are more likely to understand your paper if the report is easy to read, the review process will move much faster, and the audience will learn more from a well-organized and well-prepared report. Finally, a certain fraction of your target audience, the knowledgeable scientific writer, will be especially impressed if the report is well written. Because your accepted report will become part of the body of scientific knowledge, your well written article will likely impress scientists many years into the future.

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*Michael K. Dowd is a chemical engineer at the US Department of Agriculture–Agricultural Research Service, and a senior associate editor for the Journal of the American Oil Chemist Society (AOCS). He can be contacted at Michael.Dowd@ARS.USDA.GOV.*

## Some resources

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# Professional Pathways

*Professional Pathways is a periodic Inform column in which AOCS members discuss their professional experiences and share advice with young professionals who are establishing their own careers in oils- and fats-related fields.*

## 1. Why did you join AOCS?

AOCS constitutes an excellent body representing the interests of the fats, oils, and detergent industries. Joining AOCS helps me to keep in touch with the latest trends and developments in these industries and also allows me to interact with professionals who attend the AOCS meetings.

## 2. Please describe your career path.

After obtaining my master's degree in plant biochemistry from GB Pant University in India, I came to Australia, where I obtained my Ph.D. in plant biochemistry from the University of Adelaide in 1985. My Ph.D. research was focused on the role plant lipids play in hormone perception, and I had a desire to skill up in molecular biology. This led me to move to the University of Queensland, where I was a postdoctoral fellow in molecular oncology for five years. In 1990, I joined CSIRO Agriculture in Canberra where I initiated research in the molecular biology of plant fatty acid synthesis. I now head up a 30-strong Plant Oil Engineering Group applying molecular biology of fatty acid synthesis and metabolic engineering to manipulate fatty acid composition to produce novel edible and industrial plant oils.

## 3. What do you love about your job?

My passion is to excel in translational science. I like to use science to deliver practical impacts. I am very fortunate that my group is on track to deliver two new oilseed crops, super-high-oleic safflower and long-chain omega-3 canola to the market with traits that will help to create new industries and work for the betterment of human health and the environment.

## 4. How do you see biotechnology research changing in the next five years?

I see the two emerging technologies of gene editing and synthetic biology having a profound influence on my field of plant biotechnology. Both these technologies are disruptive in nature and have the potential to create crops that will be able to meet the challenge of feeding, fueling, and clothing burgeoning populations in the face of global climate change that may not be conducive to current crops.

## 5. Nature Biotechnology (NB) recently named you as being among the most active translational scientists for patenting. Do you have any advice for others regarding the research-to-patent process?

Patenting is a key driver of innovation and translational research. Research and patenting can coexist. In fact, publication of research in peer-reviewed journals can underpin a patenting strategy. This is evidenced by the very high H-indices of translational researchers represented in the NB list of top 20 translational researchers for 2014. [The H index attempts to measure both the productivity and citation impact of the published body of work of a scientist or scholar.]

## 6. Please describe memorable job experiences.

When you grapple with a sticky problem for days on end or you get an experimental result that does not make sense, the solution can present itself at the most unexpected moments such as in the middle of watching a movie. These eureka moments are my most memorable experiences in my job.

## 7. Please describe a course, seminar, book, mentor, or speaker that has inspired you in ways that have helped you advance your career.

Two great mentors in my life have been my boss, Dr. Allan Green, who hired me to set up the Plant Oil Engineering Group in CSIRO, and a Swedish lipid biochemistry guru. Both of them have instilled a great love for lipid science in me—something that has occupied me for the last 25 years.

## 8. Do you have any advice for young professionals who are trying to develop an effective network of other professionals?

Be part of professional societies. Attend and present talks/posters at conferences. Both of these activities provide great access to researchers at the top of their game. Seek out mentors, as they can open up the doors to networks for you.

## 9. What are the opportunities for advancement in your career/field and how can someone qualify for such advancements?

Bring your passion for science to your workplace. The path for innovation is not linear so don't be afraid to fail as some of the biggest success in science and industry are built on failures.

## 10. What is your opinion about the value of obtaining or possessing a graduate degree given a challenging global economy?

In this post-"omics" era, a graduate degree—preferably a multi-disciplinary one—is a must if you want to pursue your ideas and do translational science leading to practical impacts.

Surinder Singh was recently, named by Nature Biotechnology (NB) as No. 10 on that journal's list of the top 20 translational researchers of 2014. The ranking is based on an examination of 2014's most active scientists for patenting, according to patent analytics firm IP Checkups.



Singh is based at CSIRO (Commonwealth Scientific and Industrial Research Organisation) Agriculture Canberra, where he is group leader of the CSIRO Plant Oil Engineering Group. His group, which is comprised of 30 research scientists, technicians, and doctoral students, uses "omics," lipid biochemistry, and metabolic engineering to study the molecular biology of fatty acid and oil biosynthesis in plants as well as to apply this knowledge to improve and diversify oil-producing crops through metabolic engineering of oil composition and yield. Singh has also received the CSIRO Medal for Research Achievement for the development of DHA canola, the CSIRO Newton Turner Award, and the CSIRO "Go for Growth" Award for the development of the seed oils research group, among other awards.

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# Career advice: how to find and work with a headhunter

Patrick Ropella

Working with a headhunter can be the single most life-changing decision you make for the future of your career. During the 30-plus years I've worked in the executive search industry, I've seen families brought closer together, watched a single new hire create innovations that changed the world forever, and forged relationships that stood the test of time. That being said, if you don't quite fit the laser-focused qualifications the headhunters are seeking right now, they may ignore your call. Perhaps they want someone in oleogels while your expertise lies elsewhere. Headhunters work for corporate clients, not for you. So, you may face a whole lot of direct rejection when you reach out to them. Still, there's no better way to land a great job than when a top-quality recruiter contact and qualifies you. This article offers some basic guidelines that will increase your odds of success should you choose to work with one.

Working with a headhunter can be the single most life-changing decision you make for the future of your career. During the 30-plus years I've worked in the executive search industry, I've seen families brought closer together, watched a single new hire create innovations that changed the world forever, and forged relationships that stood the test of time. That being said, if you don't quite fit the laser-focused qualifications the headhunters are seeking right now, they may ignore your call. Perhaps they want someone in oleogels while your expertise lies elsewhere. Headhunters work for corporate clients, not for you. So, you may face a whole lot of direct rejection when you reach out to them. Still, there's no better way to land a great job than when a top-quality recruiter contact and qualifies you. This article offers some basic guidelines that will increase your odds of success should you choose to work with one.

## HONESTY PAYS

Be real about your skills, experience and knowledge. Headhunters get paid to find people who bring specific skills and experience to the job. They don't get paid to try out this man or that woman. If you lack the exact experience the headhunter is seeking, tell the truth. If you don't, your shortcoming will inevitably become obvious, and you will never hear from that recruiter again.

A headhunter's fees are substantial, because he is expected to find and develop a slate of highly qualified, well-screened candidates. His clients can clearly tell if a candidate has the specific experience sought, fits the compensation range, and is motivated by the right reasons. Be ethical.

If a headhunter tells you about a job in soybean processing that another headhunter already mentioned to you, you must tell the second one, "Thanks, but another headhunter already told me about that opportunity." Same thing goes if you have already directly submitted your résumé for that same job to the company.

- Working with a headhunter can give you special access to confidential positions and opportunities.
- Recruiters will go to bat for you, but you must present the honest facts upfront.
- Working with a headhunter could mean facing constant rejection, but there's no better way to land your dream job.



## HOW TO CHOOSE THE RIGHT HEADHUNTER

- Any recruiter can source candidates from the Internet, but the best headhunters use the Sourcing, Marketing, Assessing, Recruiting, and Training (SMART) search system and provide it to their clients.
- Study headhunters' websites and biographies. Ask yourself, "Will this relationship be a good fit based on personality and the positions and the companies the recruiter works with?"
- Be open to exploratory interviews.
- Follow the headhunter's lead if she says, "Although you're not a perfect fit for this job, the client is open to people with your background in palm oils and edible fats."
- Respond promptly if a headhunter requests more details. This makes his job easier and shows that you're professional. (Your recruiter will go to bat for you, but he needs solid information to keep the client interested. The more specific the information, the better he can make you appear.)
- Listen carefully and ask questions when the recruiter offers interview preparation.
- Ask your headhunter about the process. How many phone or face-to-face interviews will there be? Are presentations required? What are the expectations and goals? The more details you have, the better equipped you are.
- Call the recruiter immediately after the interview to discuss the next step. Make your headhunter a partner in the process and you'll gain more insight and improve your odds of success.
- Provide all computer database information so the headhunter will easily remember you when he gets new clients. Keywords should spell out your skills, abilities and experiences, products, industries, roles, languages, and so on.
- If you specialize in a specific industry or in a specific role, find headhunters who specialize in your area. Ask them what positions they recently filled.
- Generally, retained headhunters are more polished and respected than contingency recruiters; industry or functional specialists are preferred over generalists.

CONTINUED ON NEXT PAGE



- You want a recruiter who is seasoned in the business of executive search. Anyone with fewer than five years of experience can be risky. Anyone with more than 10 years of experience is most likely a professional. The few with 25 years of experience will stand out. You will recognize their expertise when you visit their websites, speak to them on the phone, or meet them face-to-face. Request references for both the recruiter and the client. It's a red flag if they lack references.

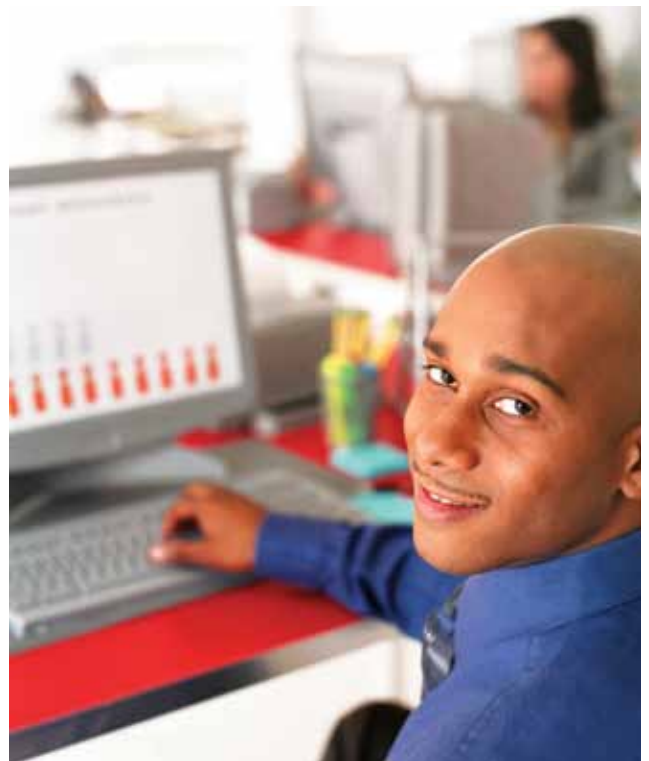
## THE RIGHT FIT, THE RIGHT JOB

Always show headhunters that you are most interested in the right fit and that you value job satisfaction and career development. The right fit is the alignment of skills, attitude, cultural fit, experience, and other requirements of the position. This mature approach will make the headhunter respect you and want to help you more. Here are more specific tactics.

- Tell her where you will and won't relocate.
- In setting salary expectations, realize that your current or past compensation, benefits, and experience may not apply to the position offered. Use current and past details as benchmarks, not as requirements.
- If you get a raise, more power to you! Still, consider the whole package: the role, the relationships, the location, and other factors. I often see people take less money just to get into more satisfying circumstances. Focus first on the right fit, and the total compensation will take care of itself.
- Help your headhunter by providing clear expectations. If you believe this position is the right fit for you, tell him 1. what salary you would reject, 2. what salary you would consider, and 3. what salary he can accept on your behalf. Don't try to control the negotiations between the head-

hunter and your potential employer. Your headhunter knows the employer's compensation and benefits flexibility and the breaking point, as well as other candidate statuses that you don't know. Partner with your headhunter, but let her drive the car.

- Consistently update your headhunter whenever you improve your resume, gain new experiences or skills, or change your contact information.
- The more a recruiter shows you what's in line with your goals and aspirations, the more effort you should put into developing a long-term relationship.
- Good headhunters help you sharpen your technique for winning the job. Use the tools they give you for better results the next time you go for an interview.





## PROS AND CONS OF WORKING WITH A HEADHUNTER

### Advantages

- When a headhunter presents you to a company, you're much more likely to be noticed.
- If the hiring authority says, "I don't see a perfect fit here," your recruiter will address questions and sell you to get you an interview.
- If you're on your own, you wouldn't even know why you were rejected.
- A company willing to pay for a headhunter takes hiring seriously and wants to make the right hire. It shows that the company is financially strong and committed to making this hire succeed.
- Often, headhunters help fill openings that are confidential or high priority; these openings are rarely found on job boards or in other recruiting venues.
- Headhunters can guide you through résumé and information presentations, interview preparation, and negotiations.
- They do this over and over; they're adept at improving the odds of your success.

### Disadvantages

- As in most service industries, 80% of the people in the recruiting business are apprentices who are learning and growing through the school of hard knocks. About 20% are masters with time-tested systems of excellence; they do 80% of the business, leaving 20% for the apprentices.
- You will need to spend considerable time to ensure that the headhunter understands your successes, talents and qualities. This time may seem wasted if you don't get the job you wanted. But the truth is you probably would never have gotten as close to the job without them.

*Patrick Ropella is Chairman and CEO of the Ropella Group, [www.Ropella.com](http://www.Ropella.com), an international executive search, leadership transformation, and corporate consulting firm. He authored the book and web-based training program, The Right Hire—Mastering the Art of SMART Talent Management, and his content has been featured in many trade magazines, business publications, and industry journals. Patrick regularly speaks at webinars, career fairs, and conferences, and can be contacted at +1 850-983-4777.*



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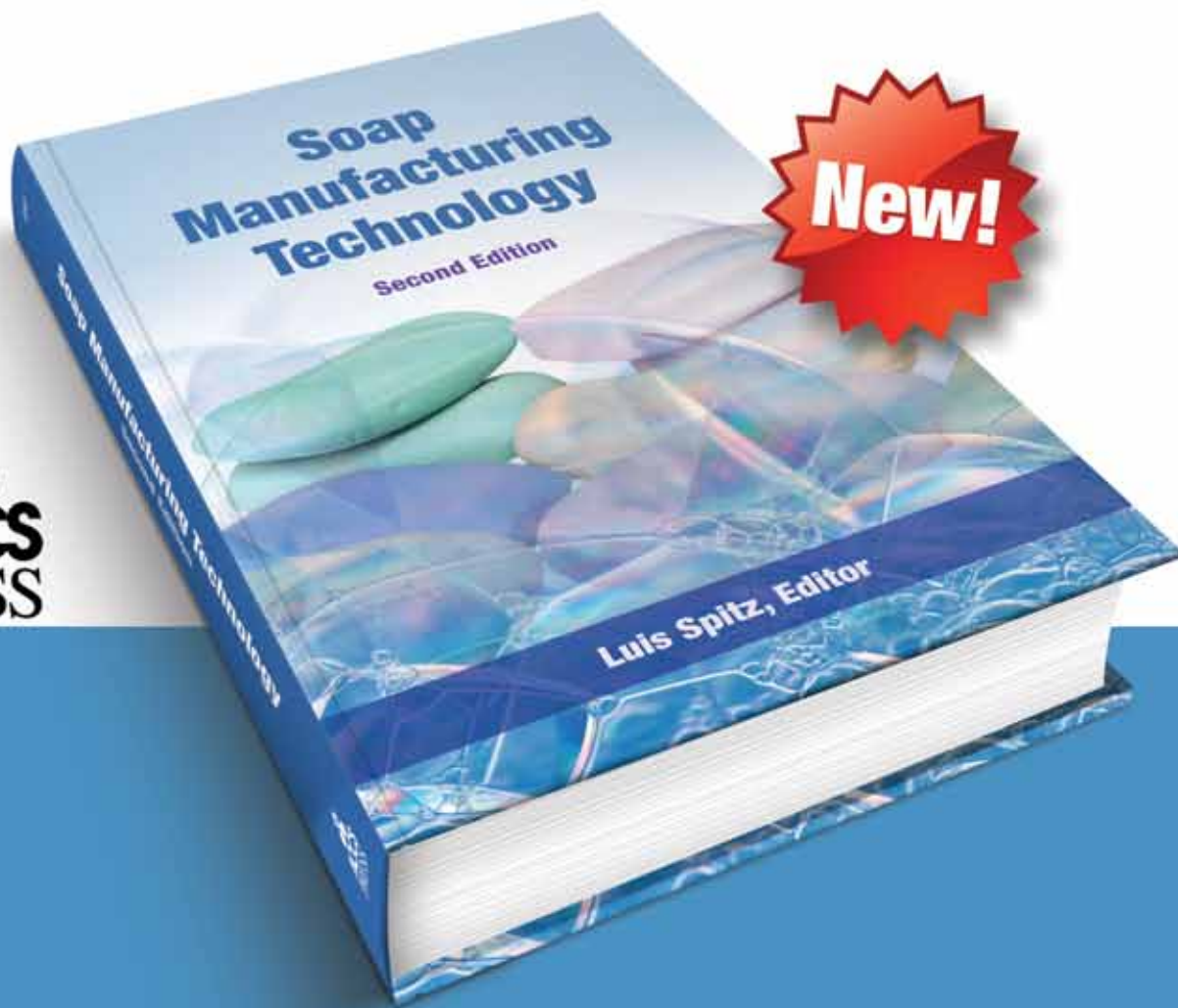


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