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On the cover: This issue's cover features a hydrocolloid-based oleogel viewed under a polarized light microscope.

CONTENTS

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Hydrocolloid-based structuring of edible oil

Ashok Patel

Hydrophilic polymers, such as polysaccharides and proteins, are often referred to as hydrocolloids because of their ability to form viscous solutions and/or gels when dispersed in water. The presence of many hydroxyl groups, gives these macromolecules a very high affinity for binding with water molecules. Due to these water-structuring properties, hydrocolloids have a long history of use as thickening and gelling agents in processed food formulations.

- **Although the field of edible oil structuring is well over 20 years old, the possibility of using structured edible oils to develop new product formats with improved nutritional profiles has recently generated considerable interest from the scientific community [1–2].**
- **Most early work was focused on low-molecular-weight organogelators (LMOGs), but in the last six years, there has been a gradual shift toward the use of hydrophilic polymers as oil-structuring agents.**
- **This article summarizes several innovative approaches that have been developed over time (Fig. 1), and explains how polymer oleogels fit into "clean-label" positioning.**

Water acts as a strong solvent for hydrophilic agents, promoting "polymer-solvent" interactions over "polymer-polymer" interactions. Consequently, the hydrated polymeric molecules (random coils) have the freedom to acquire unfolded extended shapes that occupy distinct excluded volumes (also known as the hydrodynamic volume). Once the polymer concentration exceeds a certain concentration—C^* (also known as the overlap concentration), the hydrodynamic volumes start overlapping (i.e., the polymeric molecules start interacting weakly), and concentration beyond C^* is accompanied with a relatively higher "polymer-polymer" interaction and strong interpenetration of polymer molecules leading to the formation of semi-dilute solutions. For some polymers, a critical crosslinking concentration (also known as critical gelling concentration, C_g) may also be achieved above C^* where a three-dimensional cross-linked structure stabilized by transient junction zones is formed that results in the gelation of the solvent. Because of their large molecular sizes, the overlap and gelling concentrations of hydrocolloids are exceptionally low (as low as 0.01% weight in some cases), making them very efficient as structuring agents for aqueous solvents.

In contrast, it can be challenging to structure an oil phase using hydrocolloids, as their solubility characteristics do not match those of hydrophobic oil solvents, and they consequently do not have the required dispersion properties. To achieve a similar functionality in oil gelation, it is necessary to first pre-hydrate the hydrocolloids in the aqueous phase and arrest their hydrated conformations in a dehydrated form that enables them to be used for physical entrapment of liquid oil.

Some hydrophilic polymers, such as proteins and modified polysaccharides, are surface-active. So, conformational frameworks of these polymers can be created from their water dispersions by first promoting their adsorption to air-water or oil-water interfaces, and then "stripping-off" the water to obtain dried microstructures.

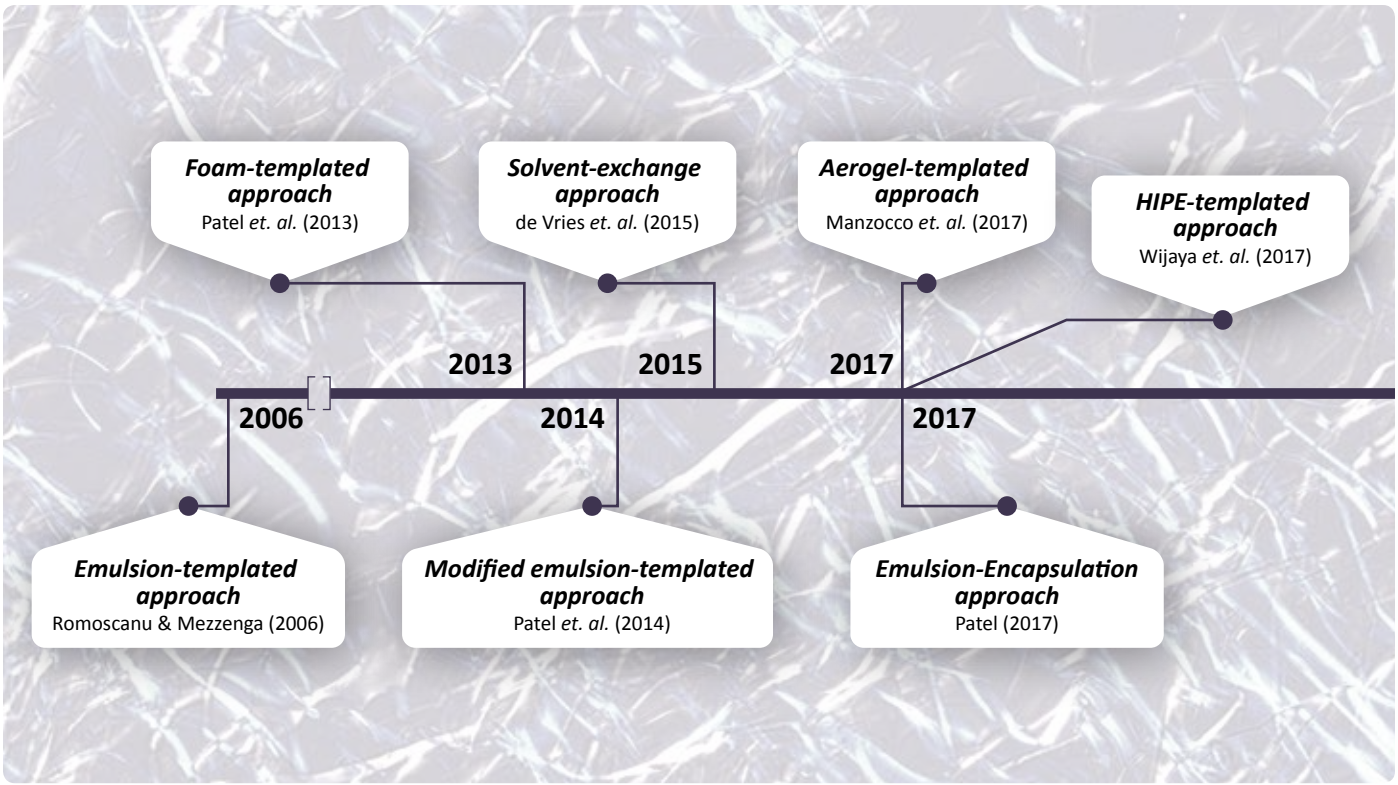

FIG. 1. Timeline of innovative approaches to structuring oils with hydrocolloids [3]

INNOVATIVE APPROACHES

Emulsion-templated approach

An indirect method for hydrocolloid-based oil structuring was first demonstrated by Romoscanu et al. at ETH Zurich in 2006 [4]. Protein (beta-lactoglobulin)-stabilized emulsions were used as templates to obtain protein-in-oil gels through freeze drying. The oil droplets packed so closely together during freezing and drying that they deformed into polyhedral cells. The gel elasticity was controlled by the Laplace pressure of the deformed droplets, which resisted any coalescence on drying. The process, however, was cumbersome and required several washing steps to remove the unabsorbed protein. Furthermore, the protein films adsorbed at the surface of the oil droplets had to be strengthened by either thermal or chemical cross-linking.

The process was modified in 2014 by Patel et al. at Ghent University. Patel's group created oil-based gels using emulsions stabilized by a combination of polysaccharides (methylcellulose as surface active component together with xanthan gum as the non-surface-active hydrocolloid) [5]. It is a well-known strategy to use such combinations of hydrocolloids to stabilize water-continuous emulsions. When combinations are used, the surface-active component is responsible for lowering the interfacial tension at the surface of dispersed oil droplets, and the non-surface-active component may provide one or several of these functions: a) interact with the surface-active component to form complexes that display a relatively higher interfacial activity; b) anchor onto the surface-active component and enhance the interfacial elasticity; and c) increase the bulk phase viscosity to decrease droplet coalescence. In this case, the combination of methylcellulose-xanthan gum resulted in an increase in the elasticity of the interfacial film, which resisted rupturing on dehydration during the drying of emulsion templates.

This approach of using a combination of hydrocolloids has been adopted successfully by several research groups. In comparison with the work of Romoscanu et al., this approach is highly reproducible and requires no crosslinking or tedious washing steps. However, it still suffers from the drawback of a long drying time, which can be detrimental to the quality of oil—especially when elevated temperature is used for drying the emulsions. The long drying time is due to the presence of unabsorbed non-surface-active hydrocolloid in the bulk phase which binds the water. Recently, Wijaya et.al. found a way to circumvent this drawback by i) using high internal phase emulsions (HIPEs) as the templates (to decrease the overall water content) and ii) using complexes of hydrocolloids as stabilizers (that accumulate at the interface so the bulk phase is relatively unstructured) [6]. HIPEs with oil volume fraction greater than 0.74 (more than the close packing of spherical oil droplets) were first formulated using pre-formed complexes of either whey protein isolate or sodium alginate with low methoxy pectin. Due to the relatively lower content of water in the bulk phase, these emulsions could be efficiently dried to obtain oil-in polymer gels.

The emulsion-templated approach has seen a gradual evolution from use of single component (crosslinked proteins) to two-components (polysaccharide combinations), and very recently, to protein-polysaccharide complexes and conjugate particles as stabilizers for emulsion templates (Fig. 2, page 8).

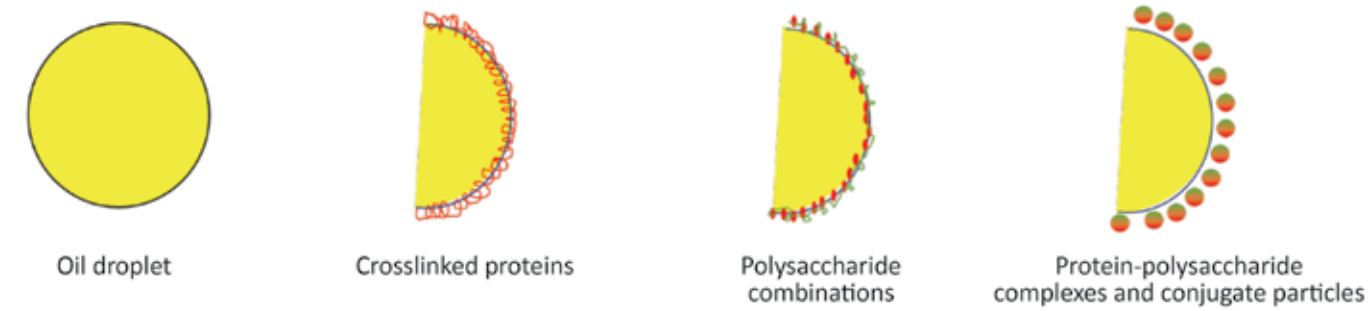

FIG. 2. Schematic representation of interfacial stabilization mechanisms in emulsion-templated approach. The stabilization of emulsion templates has gradually evolved from use of single component (crosslinked proteins), to two-components (polysaccharide combinations), to protein-polysaccharide complexes and conjugate particles. [3]

Foam-templated approach

Interface-stabilizing properties of certain hydrocolloids (hydroxy propyl methylcellulose and gelatin) were exploited by Patel et al. in 2013. The group introduced a novel foam-templated approach to create porous polymeric structures capable of absorbing large quantities of liquid oil [7]. The absorption capacity of the dried foam was quite high due to its open cellular structure. However, as the oil binding was purely physical, the oil-sorbed polymer released oil under pressure. On shearing, the oil-sorbed polymer, a self-standing biphasic colloid with deformation properties of a strong gel, was obtained. This relatively facile approach (Fig. 3) has been adapted for practical applications in structuring a range of food products, including peanut butter, sandwich cookie creams, and muffins [8–10].

Solvent-exchange approach

A more sophisticated technique involving solvent exchange has also been tried to make hydrocolloids suitable for oil structuring [11]. Typically, a hydrogel is created by dispersing the hydrophilic protein in water. It is then subjected to heat treatment to expose the hydrophobic groups of protein and create basic building blocks. Once the network is established in the aqueous solvent, a stepwise exchange from water to oil solvent is carried out using solvents with intermediate polarity to prevent any agglomeration-induced disruption of the protein network. As suggested by the authors, use of food-grade solvents such as ethanol should make this approach feasible for edible applications in commercial products.

Aerogel-templated approach

The solvent-exchange route can even be exploited to create oleogels with non-protein hydrocolloids, such as kappa-carrageenan [12]. As shown by Manzocco et.al., hydrogel of kappa-carrageenan is first converted into alcoholgel through a solvent exchange procedure. The alcoholgel is then subjected to supercritical CO_2 drying to obtain a porous aerogel that can absorb a considerable amount of liquid oil (oil absorption capacity up to 80%). The initial results are promising, and this approach could be extended to other hydrocolloids to generate aerogels that may prove to have a higher oil absorption.

Emulsion-encapsulation approach

Recently, it was demonstrated that a combination of emulsification and encapsulation can also be explored to introduce hydrated layers of hydrocolloids in the matrix of crystallizing fats [13]. This multi-step process involves emulsifying the fat hard stock (palm stearine) at high temperature in the hydrocolloid solution. The amphiphilic nature of the hydrocolloid results in the adsorption of polymer layers at the surface of dispersed fat droplets. The adsorbed layers are speculated to undergo gelation at the interface due to the unique high-temperature gelation behavior of the hydrocolloid (methylcellulose). The hot

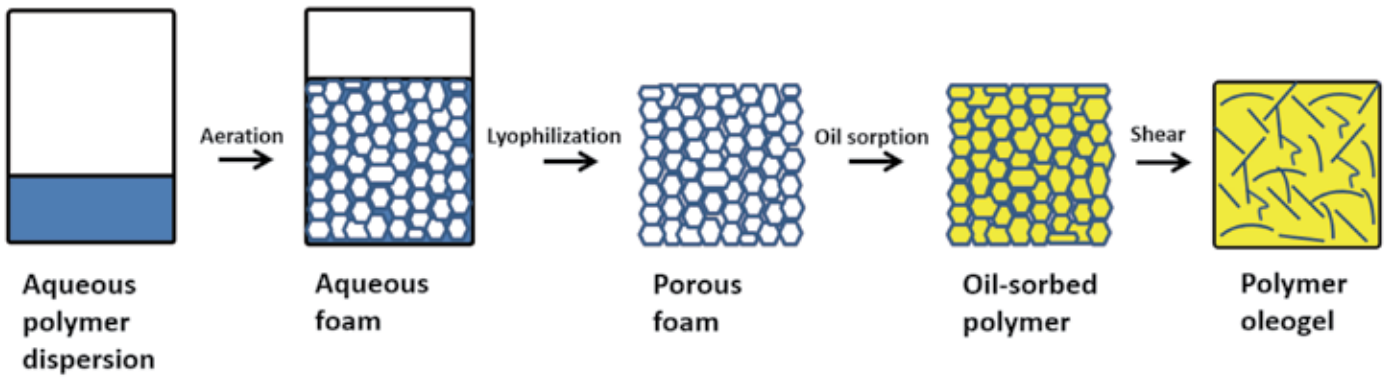

FIG. 3. Schematic representation of steps involved in formation of polymer oleogels using foam-templated approach [3]

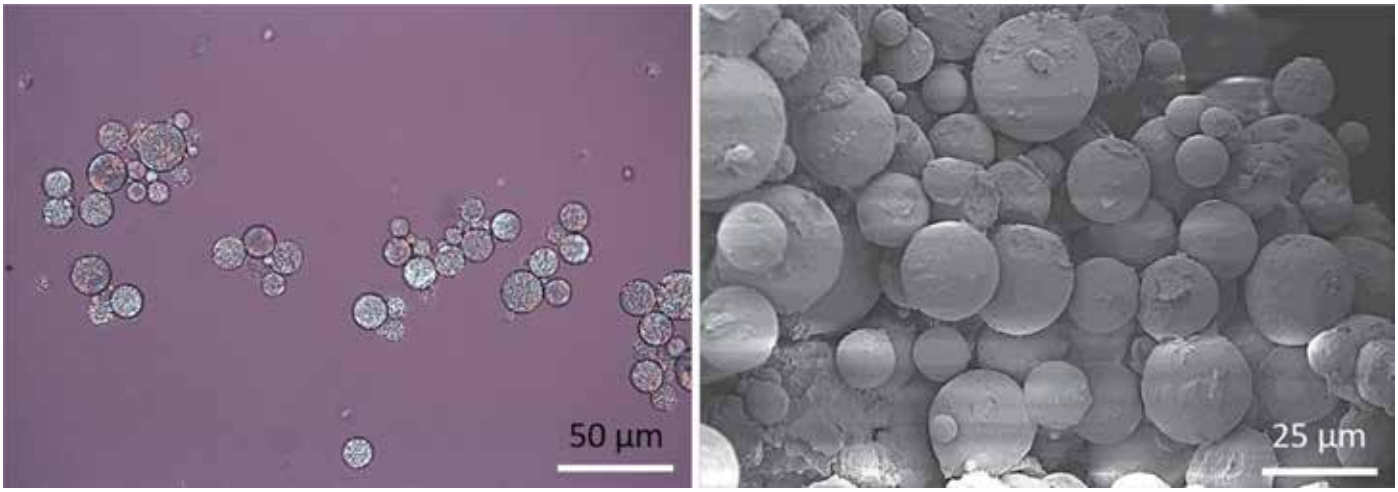

FIG. 4. Polarized light and cryo-scanning electron microscopy images of powdered hard stock (polymer coated fat capsules) prepared using emulsion-encapsulation approach [3]

emulsion is then immediately diluted in ice cold water to trigger the solidification of fat droplets into solid capsules. The large size of droplets (greater than 1 micrometer) ensures a rapid creaming of the coated capsules, which are then separated and dried. This powdered hard stock (Fig. 4) can then be used to deliver hydrated polymer strands into the liquid oil.

HOW DO POLYMER OLEOGELS FIT INTO "CLEAN-LABEL" POSITIONING?

Earlier studies in the field of oil structuring were mainly focused on the fundamental understanding of structuring principles and identification of new structurants. In recent years, numerous reports on potential food applications of structured oil have been published. Thanks to these rapid advances in the field, we now possess enough structuring strategies and knowledge about working principles to actually implement practical applications in commercial food formulations. Some polymeric oleogels prepared using the aforementioned approaches have already been successfully evaluated for replacement of solid fats in different categories of food products, including bakery products (cakes, shortbread cookies, muffins, and cookie creams); meat products (frankfurter-style sausages); table spreads; and peanut butter.

In recent years, consumers and regulatory bodies have been majorly concerned about the impact of processed food on health, and the healthfulness of a food is often viewed as related to how natural it is. Consequently, there is a strong focus on formulating clean-label products for which the market is predicted to grow even further in the coming years. However, there is no standard (regulatory or legal) definition for "clean label," and the way consumers perceive clean label may be very different from the way food manufacturer define it. In general, the following attributes are usually associated with a clean-label product: i) natural ingredients: no artificial additives and no chemically sounding ingredients; ii) simplicity: a short list of recognizable ingredients; iii) minimal processing; and iv) transparency: sustainably sourced ingredients, gluten-free, and non-GMO.

Coming clean: about food hydrocolloids and clean labels

- Contrary to the message delivered in some popular media, food hydrocolloids are ideal ingredients for clean label products due to their natural and familiar origins.
- Most hydrocolloids come from natural sources (tree exudates, seeds, plant fragments, sea weed extracts, microbial fermentation, and so on), and most are used in their chemically unprocessed forms.
- Hydrocolloids are among the most commonly used food ingredients. Except for some hydrolyzed proteins and chemically modified polysaccharides (guar gum, cellulose, and starch), none of the hydrocolloids appear on the "unacceptable ingredients" list of major retailers (Whole Foods, Kroger, Aldi, Trader Joe's, and the like). Moreover, being multi-functional in nature, hydrocolloids can improve the clean-label image of reformulated products by simplifying ingredient lists.

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Some polymers used in the approaches described in this article, such as proteins (gelatin, whey protein isolate and soy protein isolate), are completely natural. Other hydrocolloids, such as carrageenan and pectins, are sourced from natural materials such as seaweeds and citrus fruits, respectively. Xanthan gum, which is a microbial fermentation product, should not create any issues if a non-GMO version is used (i.e., if both the bacterial strain and the feedstock used for fermentation are non-GMO). Cellulose derivatives such as methylcellulose and hydroxypropyl methylcellulose, which are chemical-sounding ingredients with long names, may not go well with clean-label positioning—even though they are derived from natural cellulose sources. However, these hydrocolloids can be easily substituted (for example, with proteins, such as gelatin and clean-label starches) without compromising functionality.

CONCLUDING THOUGHTS

Compared with other oil gelators, hydrocolloids are an attractive class of structuring agents that have the potential to be applicable in commercial settings, as most hydrocolloids are already approved for food use. Structured oils based on hydrocolloids also offer the possibility to load and deliver a high proportion of macronutrients, such as proteins and insoluble fibers, into lipid-based food products. Given current consumer trends toward healthful eating, such nutritionally enhanced products should have good market acceptance.

The versatile texturing properties of hydrocolloids have played a vital role in revolutionizing the processed food industry through the development of innovative formulations of water-based food products. However, the use of hydrocolloids in structuring lipid-continuous food products has not yet been fully explored. New advances and focused research on the indirect approaches discussed in this article may help extend the use of hydrocolloids to oil-continuous colloids (or oleocolloids). This may eventually contribute to a disruptive change in the strategies implemented for reformulation of lipid-based products, and possibly serve as a catalyst for transfiguration of the market for nutritionally enhanced food products.

Ashok Patel is an associate professor in Biotechnology and Food Engineering at Guangdong Technion Israel Institute of Technology in Shantou, China, where he is currently setting-up a state-of-the-art Food Innovation Lab. His past and current research is focused on using food-grade ingredients to create novel structured systems including oleogels, foams, colloidal particles, and complex emulsions to solve formulation issues in food product development. He can be reached at ashok.patel@gtiit.edu.cn.

This article is an extended version of a recently published opinion paper (Patel, A.R., "Structuring edible oils with hydrocolloids: Where do we stand?" Food Biophysics 13*: 113–115, 2018), and has been adapted with permission from Springer.*

Comparison of analytical methodologies for the analysis of 3-MCPD and glycidyl esters in infant formula

Jessica Beekman (Leigh)

- **Chemical contaminants 3-monochloro-1,2-propanediol (3-MCPD) esters, 2-monochloro-1,3-propanediol (2-MCPD) esters, and glycidyl esters are formed in edible oils during the deodorization step of the refining process. As they are considered potentially carcinogenic and/or genotoxic, their presence in refined oils, and foods containing these oils (particularly infant formula), may pose a potential health risk.**

- **Recent research efforts have focused on developing methodology for the extraction and quantitation of these contaminants in infant formula in an effort to estimate levels of exposure. In addition, newly proposed EU regulations have highlighted the need for accurate analytical methodologies.**

- **Because a standard reference material for MCPD and glycidyl esters in infant formula is unavailable, verifying the accuracy of analytical methods has been difficult. This article describes the comparison of various methodologies for the analysis of MCPD and glycidyl esters in infant formula to assess method performance.**

To improve consumer acceptance, the majority of edible oils undergo processing to remove undesirable flavors and odors, and to improve shelf stability and nutritional content. However, during the refining process, particularly during high-temperature deodorization of the oil, chemical changes take place that result in the formation of fatty acid ester contaminants of 3-monochloro-1,2-propanediol (3-MCPD), 2-monochloro-1,3-propanediol (2-MCPD), and glycidol. Recent toxicological data in rats have suggested that once ingested, these esterified compounds release free MCPD and glycidol, considered to be potentially carcinogenic and/or genotoxic.

3-MCPD has been classified as a carcinogen by the European Scientific Committee on Food, and the Joint Expert Committee on Food Additives (JECFA) has suggested a tolerable daily intake (TDI) of 4 microgram/kg of body weight per day. Similarly, the European Food Safety Authority (EFSA) has suggested a TDI of 2 microgram/kg of

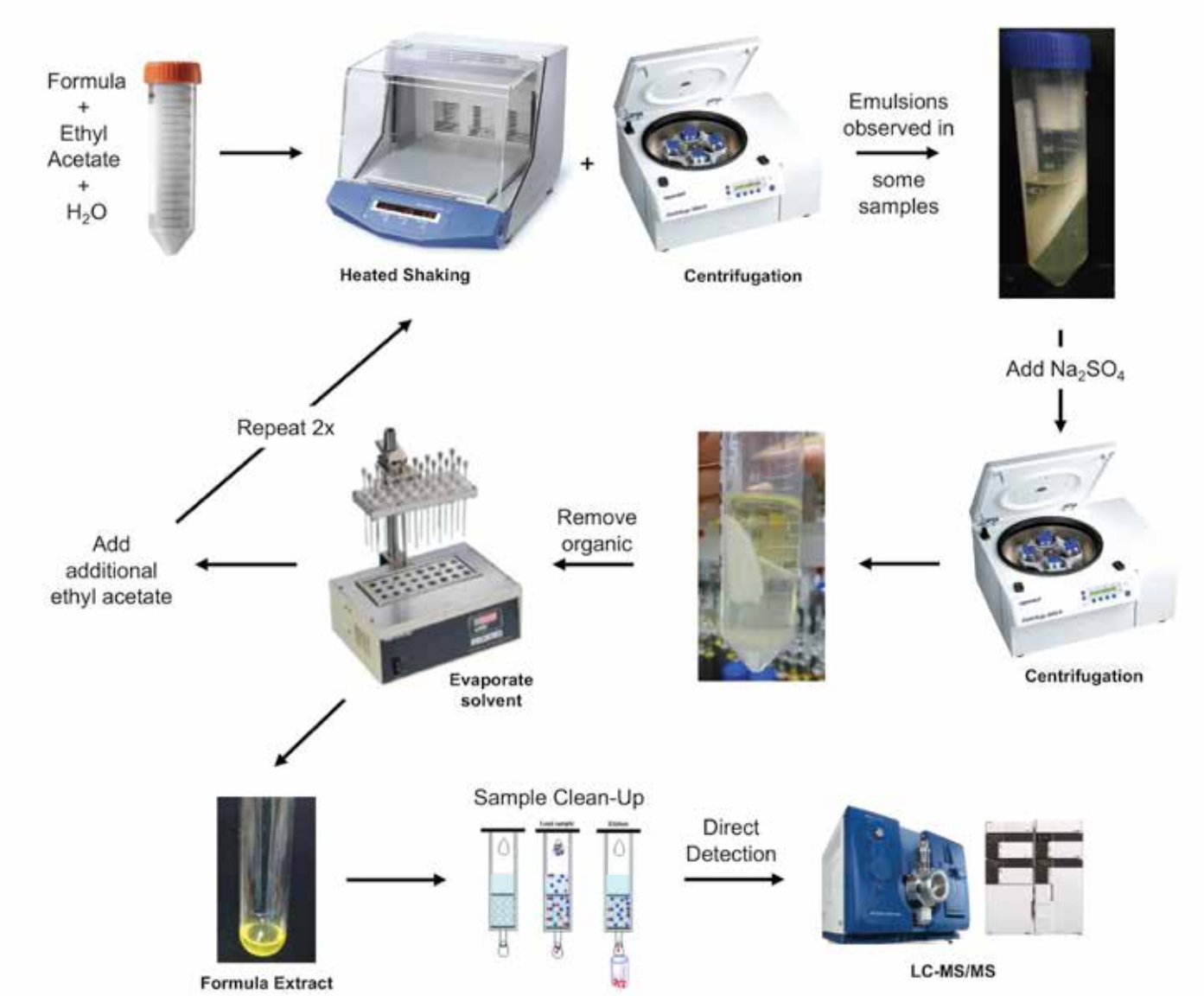

FIG. 1. US Food and Drug Administration (FDA) method for the extraction and analysis of MCPD and glycidyl esters in infant formula

body weight daily. Glycidol has been labeled a genotoxic carcinogen by the International Agency for Research on Cancer (IARC), with a suggested intake based on the ALARA (As Low As Reasonably Achievable) principle. And although there are only limited toxicological data for 2-MCPD, some unpublished studies have suggested that its toxicological properties are different than those observed for 3-MCPD.

Over the last 10 years, a number of methods have been published for the detection and quantitation of MCPD and glycidyl esters in refined oils, and these methods can be grouped into one of two categories: indirect methods and direct methods. Indirect methodologies involve a sample preparation that includes hydrolysis of the individual esters in the sample to form either 3-MCPD, 2-MCPD, or glycidol, depending on the ester backbone. The resulting free 3-MCPD, 2-MCPD, and glycidol are then derivatized and analyzed by GC-MS, allowing the ester concentrations to be expressed as a total of "bound" 3-MCPD, "bound" 2-MCPD, and "bound" glycidol. There are three AOCS official methods for indirect analysis (Cd 29a-13, Cd 29b-13, and Cd 29c-13), and they are typically used for routine analysis. Direct methods, on the other hand, involve the detection of the individual intact esters by LC-MS/MS, followed by mathematical conversion of the ester concentrations to bound MCPD and glycidol equivalents. Because the direct methods require a large number of analytical standards for quantitation of the individual esters, these types of methods have been found most suitable for research purposes.

Within the last decade, numerous publications have detailed the occurrence of MCPD and glycidyl esters in a wide array of edible oils from all over the world. For example, occurrence studies performed in our laboratory at the US Food and Drug Administration (FDA) using direct methodology have shown that although the fatty acid esters of MCPD and glycidol vary between different varieties of oils, all refined oils in the study contained detectable levels of bound MCPD and glycidol. In addition, it was also shown that, on average, palm oils (and some palm oil derivatives) typically contain higher concentrations of bound MCPD and glycidol than other refined edible oils. Similar results, using various detection methods, have also been obtained in a number of other laboratories around world.

EXTENSION OF ANALYTICAL METHODS TO INFANT FORMULA

Refined vegetable oils are also used in the manufacture of a wide variety of processed foods, and are the primary fat source in commercially available infant formulas. Due to infants' low body weights, and formula potentially being an infant's sole source of nutrition, there has been some concern about infants' levels of exposure to MCPD and glycidol. Recently, the European Union released a draft regulation for maximum limits of bound MCPD and glycidol in commercial infant formulas. Due to these factors, research efforts over the last several years have focused on developing methods to quantify MCPD and glycidyl ester content in infant formula. In recent years, these emerging extraction and detection methods have been used to determine the levels of these contaminants in infant formula and perform preliminary assessments of infants' levels of exposure. However, these methods will likely be necessary in order to identify a product's compliance with EU regulations in the near future.

Recent work carried out in our laboratory at the Center for Food Safety and Applied Nutrition (CFSAN) at the FDA has focused on developing methodology for the extraction of MCPD and glycidyl esters from infant formula. In 2016, CFSAN published a procedure for the extraction of these contaminants, and paired it with a direct detection technique that was also developed in our laboratory in 2013. The extraction method (Fig. 1), which is based on the use of ethyl acetate as an extraction solvent, yields more than or equal to 95% fat recovery from infant formula, and was shown to be applicable for all infant formula varieties on the US market.

Although the method developed in our laboratory was single-lab validated using spiked samples (based on FDA protocol for laboratory validation), it was recognized that the use of spiking solutions for a solid matrix is inherently limiting for verifying the accuracy of extracting incurred fatty acid ester compounds. To address this concern, we developed a "homemade" infant formula in our laboratory, which contained refined oils with known concentrations of MCPD and glycidyl esters. Analysis of this homemade formula using our extraction and detection methods for MCPD and glycidyl esters yielded incurred ester recoveries ranging from 85–109%, reflecting suitable method performance. However, limitations to this approach for assessing method performance were also recognized. Because commercial infant formulas are produced differently than the homemade formula developed in our laboratory, the homemade recipe may not perfectly represent a commercial infant formula reference material.

Since a certified reference material for MCPD and glycidyl ester content in infant formula is not commercially available, accurately assessing method performance has been difficult—not only in our laboratory, but for other laboratories that are also developing methods for MCPD and glycidyl ester extraction from infant formula. For this reason, we found it necessary to look for other avenues of assessing method performance.

CONFIRMING METHOD PERFORMANCE—COMPARISON OF ANALYTICAL METHODS

Over the last year, CFSAN has conducted several inter-laboratory studies involving the exchange of infant formula

samples with researchers at Health Canada, Nestle Quality Assurance Center (NQAC), and SGS Germany. In these studies, each laboratory used their own unique extraction and detection methods for the analysis of MCPD and glycidyl esters in infant formula, and the final concentration data obtained by each laboratory were compared to data obtained using CFSAN's methodology. Through this data comparison, we hoped to 1) obtain comparable concentration values for the exchanged formulas, thereby demonstrating each laboratory's method performance, or 2) identify any deviations in concentration data in order to make method improvements.

A total of approximately 50 samples were exchanged with the laboratories participating in this study. Figure 2 displays the bound MCPD and glycidol concentrations obtained in our laboratory at CFSAN vs. the data obtained using various methodologies from Health Canada, NQAC, and SGS Germany. With the exception of the six data points circled in red, the bound 3-MCPD and glycidol concentrations obtained from all collaborating laboratories show a strong 1:1 correlation with the data obtained in our own laboratory, with good reproducibility even at the lower end of the analytical range. Investigation of the "outlier" data points circled in red revealed that the corresponding formula samples all contain medium chain triglyceride (MCT) oil, which is a fraction of coconut oil containing primarily C8-C10 (or "capra") fatty acids. The direct detection method used in our laboratory at CFSAN, which requires an exact analytical standard for the quantitation of each intact ester, does not contain standards for these particular fatty acid esters. So, if C8-C10 fatty acid esters of 3-MCPD are present in these formulas, our direct detection method would not detect them, which likely accounts for the deviations observed in the bound 3-MCPD concentrations.

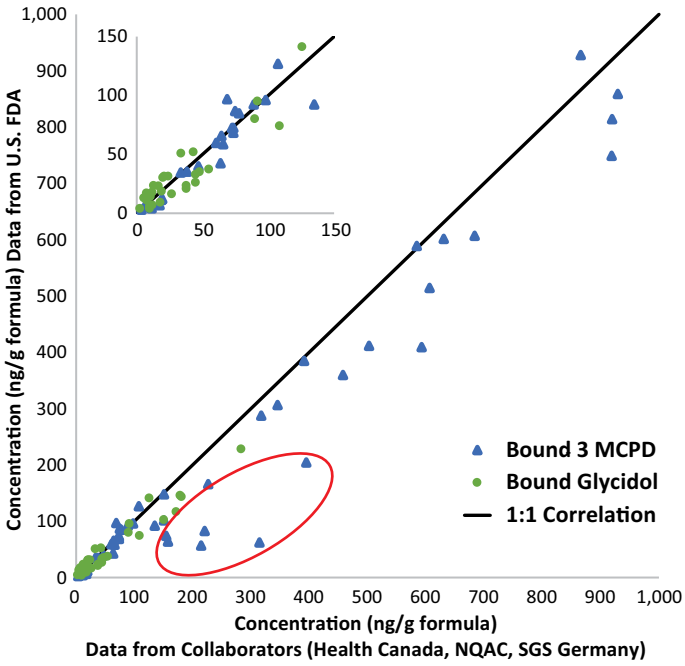

FIG. 2. Comparison of bound 3-MCPD and glycidol concentrations obtained using US FDA methods and methods used by collaborators

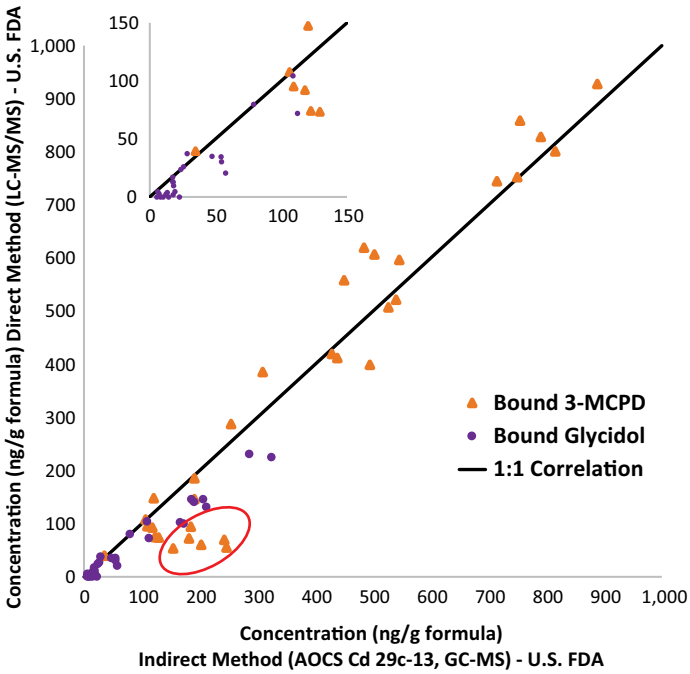

FIG. 3. Comparison of bound 3-MCPD and glycidol concentrations obtained using the US FDA extraction method with direct and indirect methodologies

In addition to the inter-laboratory study, approximately 20 unique formula samples were analyzed in our laboratory using two different detection techniques—the direct method developed in our laboratory and an indirect AOCS official method (Cd 29c-13)—to determine if the detection method impacts observed 3-MCPD and glycidol concentrations. Figure 3, which displays the bound 3-MCPD and glycidol concentrations obtained using the direct and indirect methods, shows small deviations in the bound 3-MCPD concentrations for the data points circled in red, but otherwise excellent 1:1 correlation for the bound 3-MCPD data. As described in the previous paragraph, the formulas corresponding to these outlier data points all contain MCT oil, which likely contains 3-MCPD fatty acid esters that are not used in the direct method. However, these esters would be accounted for when using the indirect method (since all esters are hydrolyzed to their free form), thereby resulting in a higher observed 3-MCPD concentration in comparison to data obtained using the direct method. With respect to bound glycidol, it does appear that data obtained using the direct method results in glycidol concentrations with a slight low bias in comparison to concentrations obtained using the indirect method. Although some further investigation is needed to determine the cause of this bias, the deviation observed is minimal and, in general, would likely not have a significant impact on quantitation results.

FUTURE PERSPECTIVES

Overall, the results of our inter-laboratory studies have shown that all methods investigated appear to perform similarly, producing generally comparable quantitative results for

bound 3-MCPD and glycidol concentrations in infant formula. Although a commercial reference material is unavailable to further assess method performance, the results of these studies have allowed a greater level of confidence in the accuracy of the results obtained using these new methodologies. Particularly with the introduction of draft regulations in the EU for maximum MCPD and glycidol levels in infant formula, the availability of accurate methodologies has become even more important. Future steps in the analysis of bound MCPD and glycidol in infant formula should involve the identification of "official," multi-laboratory-validated methods for use in enforcement of these new EU regulations.

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The author acknowledges colleague Shaun MacMahon of the US FDA for the development of the direct methodology used in our laboratory, and for helpful review of the study data. The author also acknowledges Jan Kuhlmann (SGS Germany), Adam Becalski (Health Canada), Greg Jaudzems (NQAC), and Fabien Robert (NQAC) for participation in this study.

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Leigh, J. and S. MacMahon, "Occurrence of 3-monochloropropanediol esters and glycidyl esters in commercial infant formulas in the United States," *Food Addit. Contam.: Part A*. *34*: 356–370, 2017.

The icing on the cake

Frank Flider

The US Food and Drug Administration's (FDA) determination that partially hydrogenated oils (PHOs) are no longer "generally recognized as safe" (GRAS) became effective on June 18, 2018. PHOs may no longer be used in foods sold in the United States without food additive approval. Eliminating this trans fatty acid source from the diet is certainly good news for the public's health, but it represents a number of challenges for food producers. In particular, foods that depend upon the function of trans fatty acids for structure, texture, mouthfeel, and eating qualities must be reformulated using non-trans fatty acid alternatives.

- **Partially hydrogenated oil (PHO) functionality is driven by trans (eg., elaidic acid).**
- **High-oleic soybean oil provides an element of elaidic acid functionality.**
- **A multi-year collaborative research effort between Qualisoy and Stratas Foods, LLC has determined that enzymatically interesterified high-oleic soybean oil shortenings are a suitable drop-in alternative to partially hydrogenated oils (PHOs).**

In the baking industry, one of the more challenging issues is finding a functional PHO replacement for icing and frosting, particularly the high-fat creamy buttercream-style icing. Buttercream icing is a blend of sugar, shortening, milk solids, salt, water, and flavorings. The shortening provides much of the texture, stability, and eating qualities of the icing. The high levels of trans fatty acids in PHO shortenings provided the ideal melting properties desired for high-quality icings.

A baking shortening is primarily comprised of oils and fats. The main difference between the two components is their physical form at room temperature; oils are liquid while fats are solid.

Traditionally, a baking and icing shortening was formulated with a partially hydrogenated soybean oil to which a fully hydrogenated soybean and/or cottonseed oil was added. The resultant product was comprised of triacylglycerols with melting points which ranged from a low

of -24.2°C for trilinolenic to a high of 73.1°C for tristearic, functionally held together by the trans form of trioleic, denoted as trielaidic with a mid-range melt point of 42.0°C [Daniel Swern (ed.), *Bailey's Industrial Oil and Fat Products* (1979). John Wiley & Sons, Hoboken, New Jersey, USA, pp. 194–195.]

This combination of low-melting-point and high-melting-point triacylglycerides functionally held together by trans isomers like trielaidic is no longer available without PHOs. Food scientists, required to utilize non-PHO alternatives, are under pressure to achieve non-PHO based approaches that deliver comparable consumer experience for the end use products as it relates to taste and quality.

Among the first PHO alternatives evaluated were palm oil-based shortenings and blends of hard fats and liquid oils. Challenges with these alternatives may include grainy mouthfeel and texture as well as a darker or yellow color. In order to use these early alternatives, process modifications such as changing mix times and speeds, batter temperature, and ratios of key ingredients may be required. In short, it may be necessary to reformulate the product in order to replace the PHO shortening with a palm-based shortening, and therefore, it is not a true drop-in solution.

Blends of palm or another hardstock, such as fully hydrogenated soybean oil, with liquid oil tend to result in shortenings that are higher in polyunsaturates than typically observed with a PHO shortening with a given solid fat content (SFC) profile. The presence of these polyunsaturates has a negative effect on the icing structure as well as mouthfeel, viscosity and stability.

A newer alternative is enzymatically interesterified (EIE) high-oleic soybean oil shortening. EIE high oleic soybean shortening has a composition similar to high-trans PHO shortenings. Because of this compositional similarity, functional properties of PHOs can be more closely approximated than is possible with other alternatives.

Specifically, two components are responsible; utilization of an oleic-acid-rich vegetable oil like high-oleic soybean oil, coupled with the process of enzymatic interesterification.

The oleic fatty acid in the trioleic form, as the cis alternative to trielaidic, has a published melt point value of 5.0°C. While this is not as high in magnitude as the trielaidic melting point of 42.0°C, it is a positive step forward to achieving the functionality of PHOs without contributing trans fat. This is due to utilizing trioleic as a mid-melting point material between -24.2°C for trilinolenic to 73.1°C for tristearic.

The process of enzymatic interesterification is a lipase enzyme catalyzed fatty acid randomization reaction at triacylcglyceride positions 1 and 3 within and between triacylcglycerides. The outcome of this process results in modification of melting point and functional crystallization properties without changing the overall fatty acid composition of the base oil blend (Figs. 1 and 2). Specifically, expansion of the fatty acid combination types, made possible by the lipase enzyme catalyzed randomization at positions 1 and 3, results in a greater differentiated array of triacyclglycerides than what is achieved via only physically blending.

The combination of base oil inputs, specifically high oleic soybean oil, subjected to the process of enzymatic interesterification, results in shortening bases featuring comparable melting characteristics to the PHO shortening bases they were designed to replace.

In a study conducted by Qualisoy and Stratas Foods, EIE high-oleic soybean shortening was compared to PHO shortening, EIE conventional soybean oil and a palm-soy blend in the production of a buttercream icing. EIE high oleic soybean oil shortening produced the most similar viscosity to the PHO

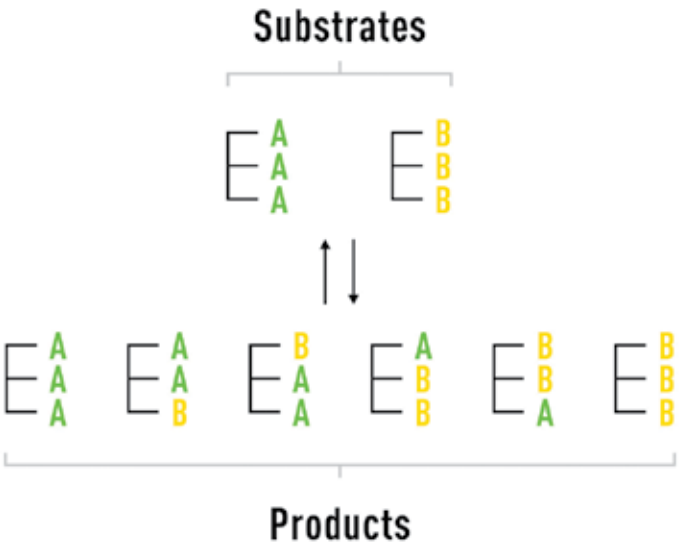

FIG. 1. Stylized triacyclglyceride fatty acid combination types expansion from oil denoted as A and fat denoted as B subjected to EIE

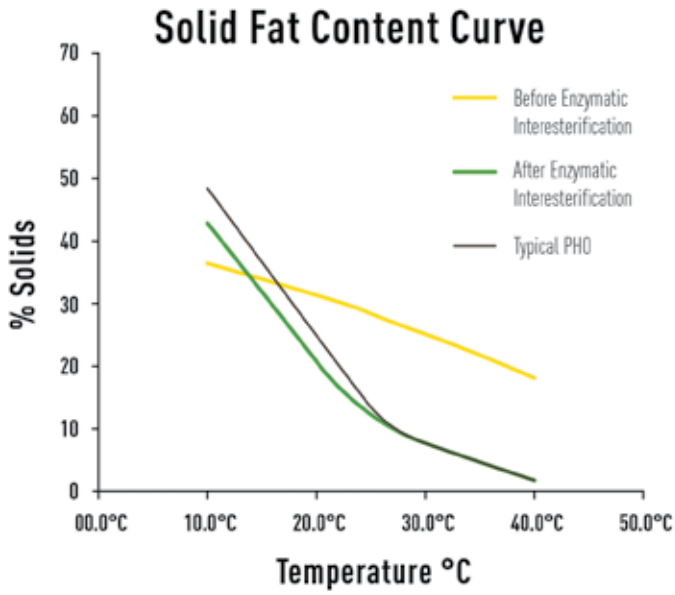

FIG. 2. Solid fat content (SFC) for various oil blends

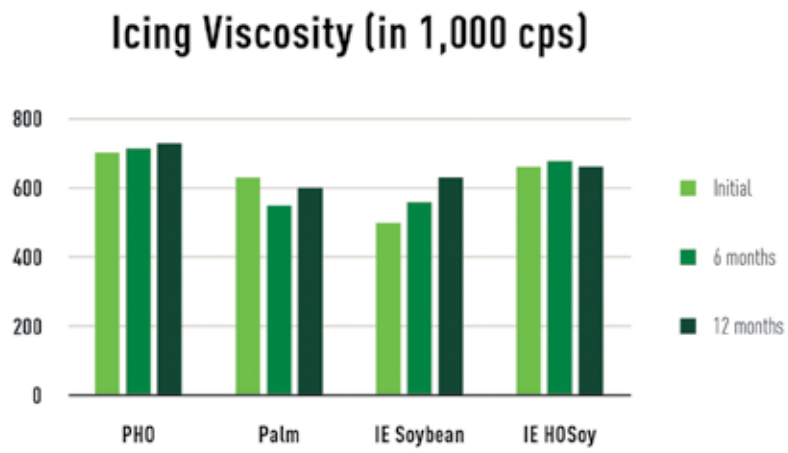

FIG. 3. Buttercream icing viscosity variances observed when utilizing icing shortening options at time 0, 6, and 12 months

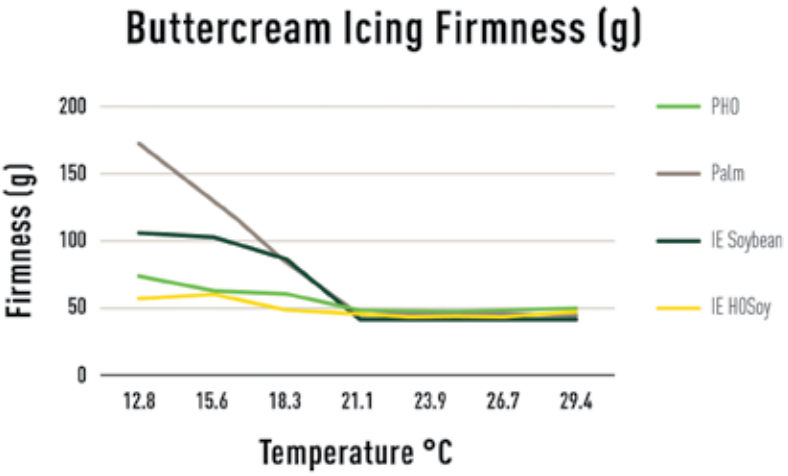

FIG. 4. TA-X2 firmness values for buttercream icing prepared with various icing shortenings

shortening (Fig. 3). When using a texture analyzer, the EIE high oleic soybean shortening outperformed all other shortenings, including PHO, showing the least change in firmness over a range of temperatures (Fig. 4). Icing made with palm showed the most change, especially at colder temperatures, indicating possible difficulties in spreadability, mouthfeel, and overall functionality.

Out of all available PHO alternatives, EIE high-oleic soybean shortening just may be the icing on the cake.

Frank Flider, Qualisoy consultant, is active in the Institute of Food Technologists and American Oils Chemists' Society. He serves on the editorial advisory committee for Inform *and the editorial board for the* Journal of the American Oil Chemists' Society. *Flider is the author of numerous technical papers and holds several US patents. He brings more than 40 years of technical, managerial, and marketing experience in the oilseed and agricultural biotech industries to Qualisoy. He can be contacted at fflider@mac.com.*

Roger Daniels, vice president research, development, and innovation, for Stratas Foods, LLC, is an inventor, author, and edible oils industry expert with numerous patents in edible oil products and processing. Prior to joining Stratas, Daniels led the R&D team for Bunge Oils, a division of Bunge North America, and held positions of increasing responsibility with Campbell Soup Company, ConAgra Foods, and ABITEC Corporation. He can be contacted at roger.daniels@stratasfoods.com.

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The AOCS Laboratory Proficiency Program (LPP), formerly known as the Smalley Check Sample Program, is the world's most extensive and respected collaborative proficiency testing program for oil- and fat-related commodities, oilseeds, oilseed meals, and edible fats. More than 500 chemists participate to verify their lab's quality control. Participants use AOCS or similar methods for sample analysis and then compare their results with those from a large cross section of other laboratories using the same methods and samples. For more information, contact Dawn Shepard at AOCS Technical Services (phone: +1 217-693-4810; fax: +1 217-693-4855; email: dawns@aocs.org).

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SERAGRO
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AFLATOXIN IN PEANUT PASTE TEST KIT

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Honorable Mention
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Honorable Mention
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AFLATOXIN IN PISTACHIO AND ALMOND

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Honorable Mention
Alina Hernandez, Jacqueline Monroy, Tou Thao
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Honorable Mention
William Spence
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Honorable Mention
George Hicks
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FEED MICROSCOPY

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

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

April 26–29, 2020. AOCS Annual Meeting & Expo, Palais des congrès de Montréal, Montréal, Québec, Canada.

May 2–5, 2021. AOCS Annual Meeting & Expo, Oregon Convention Center, Portland, Oregon, USA.

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Salting out versus pervaporation in IPA recovery system

Sayeh Sinichi and Levente L. Diosady

- An integrated process for the production of high-quality protein products and biodiesel from dehulled yellow mustard flour was developed [1, 2, 3, 4].

- This summary of a paper, originally published in the *Journal of the American Oil Chemists' Society* (*JAOCS*), describes how isopropyl alcohol (IPA) recovery and recycling techniques were used to complete an integrated process for biodiesel production directly from yellow mustard oil +IPA miscella [5].

The overall objective of this investigation was to develop a rapid industrial method for production of biodiesel from yellow mustard. We have already reported on a process that combined isopropyl alcohol (IPA)-based solvent extraction with transesterification. The next step was to complete the process cycle by selecting and designing a viable IPA recovery and recycling technique. The final process design consists of four-stage extraction using an IPA/flour ratio of 1.5:1 (volume:weight) followed by alcohol transesterification using 1.2% potassium hydroxide as catalyst, which resulted in high (89%) ester yield. Miscella treatment with a water-acid mixture as well as the addition of an acid transesterification step followed by a base-catalyzed transesterfication will ensure maximum ester yield (99%). IPA was used in this study both as an extracting solvent for oil from mustard flour, and as a reactant and/or a co-solvent in the transesterification process [6]. When the water content of the extraction solvent reaches 5%, the removal of water becomes a necessary step for the successful recovery and reuse of the IPA. To achieve IPA recovery, salting out was investigated (Fig. 1).

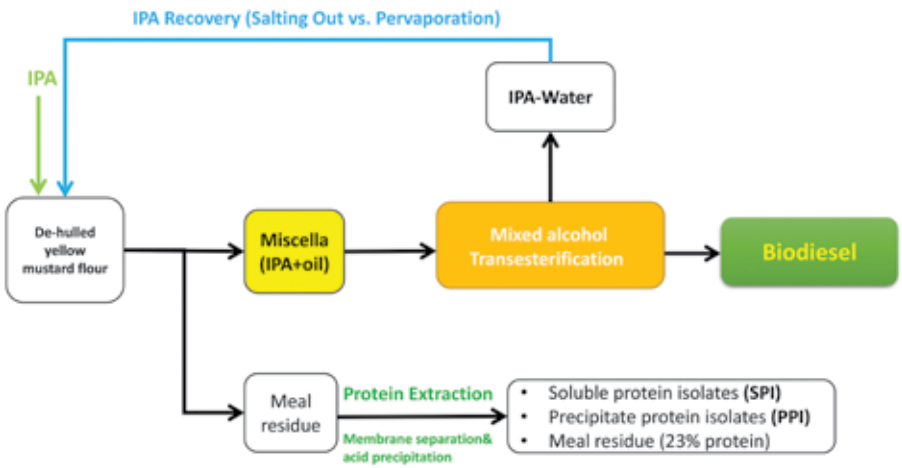

FIG. 1. Simultaneous protein and biodiesel production from yellow mustard

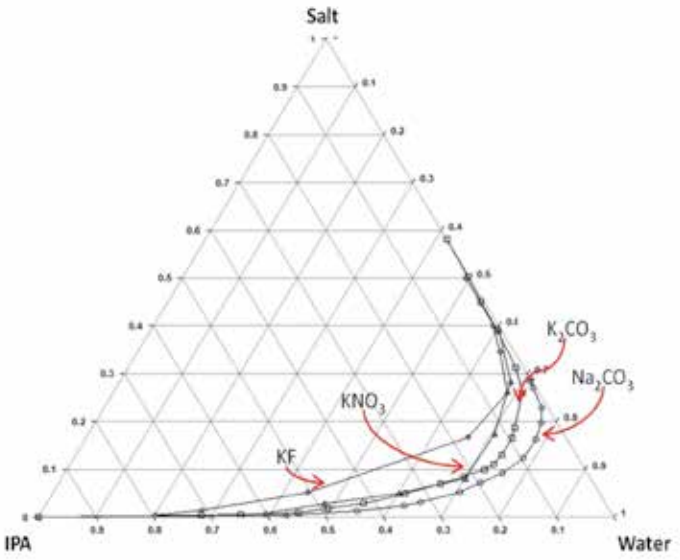

FIG. 2. Solubility curves of sodium carbonate, potassium carbonate, sodium fluoride, and potassium nitrate

The purpose of using salts was to break the molecular bonds (hydrogen bonding) between IPA and water. This allows the IPA to partition into a separate light phase with reduced water content of less than 1% for recovering and reusing IPA from the oil extraction process. To select suitable salts for IPA dehydration, several compounds were tested. Ternary phase diagrams of the solubility of the IPA/salt/water system were prepared for the four short-listed salts (Fig. 2). According to these solubility curves, sodium carbonate had the smallest mono-phasic area, which means that it can induce phase separation with a lower amount of salt when a solution has a higher water concentration. Sodium carbonate and potassium carbonate (which had the second smallest monophasic area) were selected due to their ability to induce phase separation (Fig. 3). Sodium carbonate and potassium carbonate are soluble in water and have negligible solubility in IPA. Pure sodium carbonate and potassium carbonate are white and form strongly alkaline water solutions. Both salts can be dissolved in water until saturation is reached; beyond saturation sodium carbonate converts to a monohydrate ($Na_2CO_3 \cdot H_2O$) which crystallizes at room temperature. Heptahydrates ($Na_2CO_3 \cdot 7H_2O$) and deca-

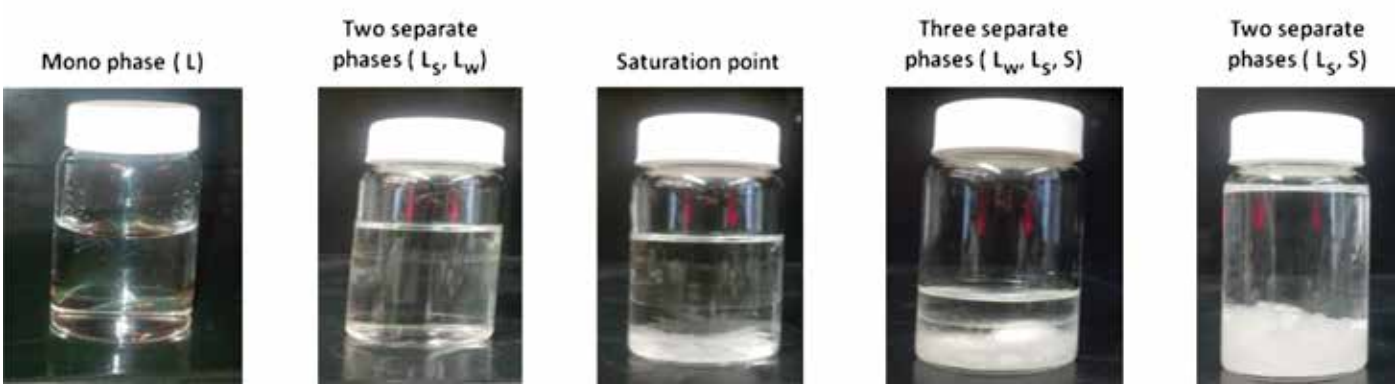

FIG. 3. IPA/salt /water solutions in different concentrations representing system behavior in various area of the ternary phase diagram. L; Mono phase, L_s; IPA-rich phase, L_w; Water rich phase, S; Solid phase.

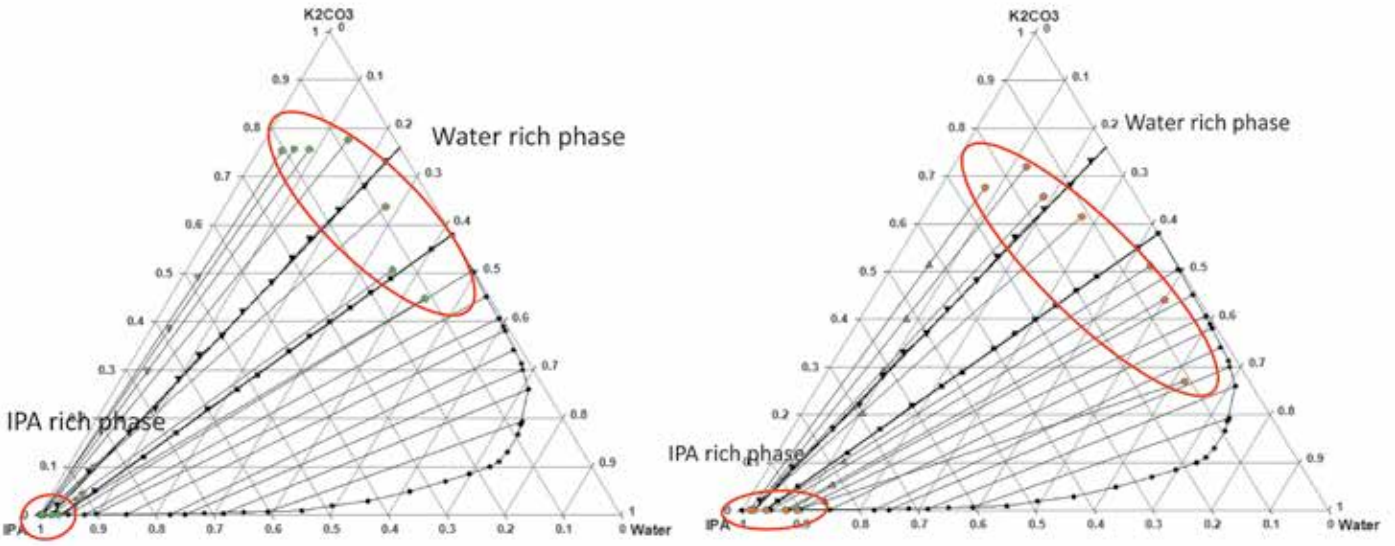

FIG. 4. Phase separation in bulk solutions with water content of 5% and 13%, with potassium carbonate content varying from 2% to 50% shown on ternary phase diagram. The red circles indicate the composition of IPA, water, and salt in the IPA-rich phase, and the water-rich phase after the addition of salt to the bulk solutions.

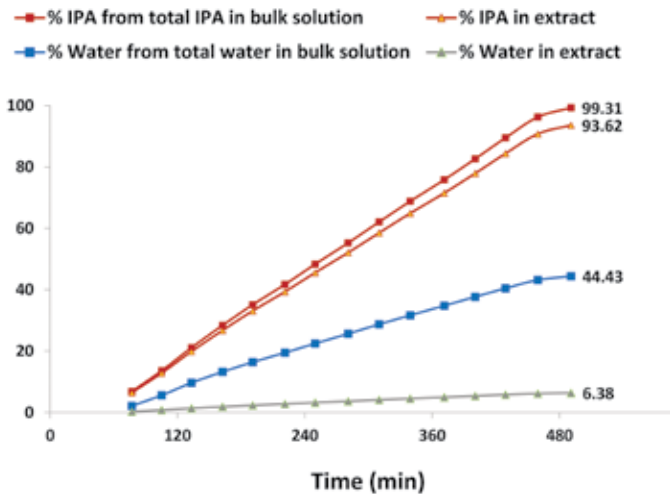

FIG. 5. Extraction data collected from azeotropic distillation using 10% potassium carbonate in the mixture of IPA/water azeotrope

hydrates ($Na_2CO_3 \cdot 10H_2O$) of sodium carbonate are also formed and tend to precipitate from solution. These higher hydrates are more difficult to re-dissolve than monohydrates.

IPA/water mixtures with water contents at 5% and 13% (weight:weight) were considered for salting out by high concentrations of salts. The bulk solutions were prepared and the salt content in both 5% and 13% mixtures were increased from 2% to 50%. Ternary phase diagram (Fig. 4) represents the compositions of IPA, water and salt in each IPA-rich phase, and the water-rich phase (solid salt) which were separated after salt addition.

For IPA recovery and recycling both of the salts (potassium carbonate and sodium carbonate) were used for the salting out process. Both salts were effective in modifying the liquid-liquid equilibrium of the IPA/water system in favor of IPA extraction from IPA/water mixtures. Approximately 95% and 96% of the IPA was recovered with 99% purity from bulk solutions with 5% and 13% water content, respectively, using 20% potassium carbonate (weight:weight). Sodium carbonate salted out IPA with more than 99% purity when the water content in the bulk solution was lower than 5% using 30% salt.

Tests were performed on azeotropic distillation of the IPA/water mixture at the azeotropic concentration with 10% potassium carbonate or sodium carbonate (Fig. 5). Potassium carbonate was more effective in modifying the liquid-liquid equilibrium of the IPA/water system in favor of IPA extraction from the bulk solution (about 99% IPA extraction, about 94% IPA content in the extract).

The next approach to separating IPA from water was pervaporation, which eliminates the use of reagents, such as salt, from the system. A pervaporation unit was prepared by modifying a SEPA CFII membrane separation unit available in our lab (Fig. 6).

The operating conditions for the SEPA CF II and the selected membrane filtration system were the following:

- Motor frequency (10 Hz)
- Typical flow rate (2.22 L/min.)
- System temperature (60°C)
- Backpressure for TFC-HR membrane (160 psi)
- Membrane pH range (4–11 at 25°C)
- Membrane temperature range (75°C)

The system was tested with different samples of pure IPA and water as well as a 50% IPA/water (volume:volume) mixture. Polyamid AK, thin film composite HR membranes were selected for the pervaporation tests. The TFC-HR membrane responded well. Only water passed through the membrane and the permeate did not smell of IPA. The GC analysis for trace IPA in the permeate was impractical.

The concentration of water in the feed at the end of the process was determined using the Karl Fischer titration method. In a batch process the starting feed stock (500g) containing 25g of water produced an average of 13g of permeate. The average concentration of the feed for 5 hours of extraction was 2.46% in the second run. The average flux for the run was calculated to be 200 mL/m²h or 0.12 gal/ft²d. The

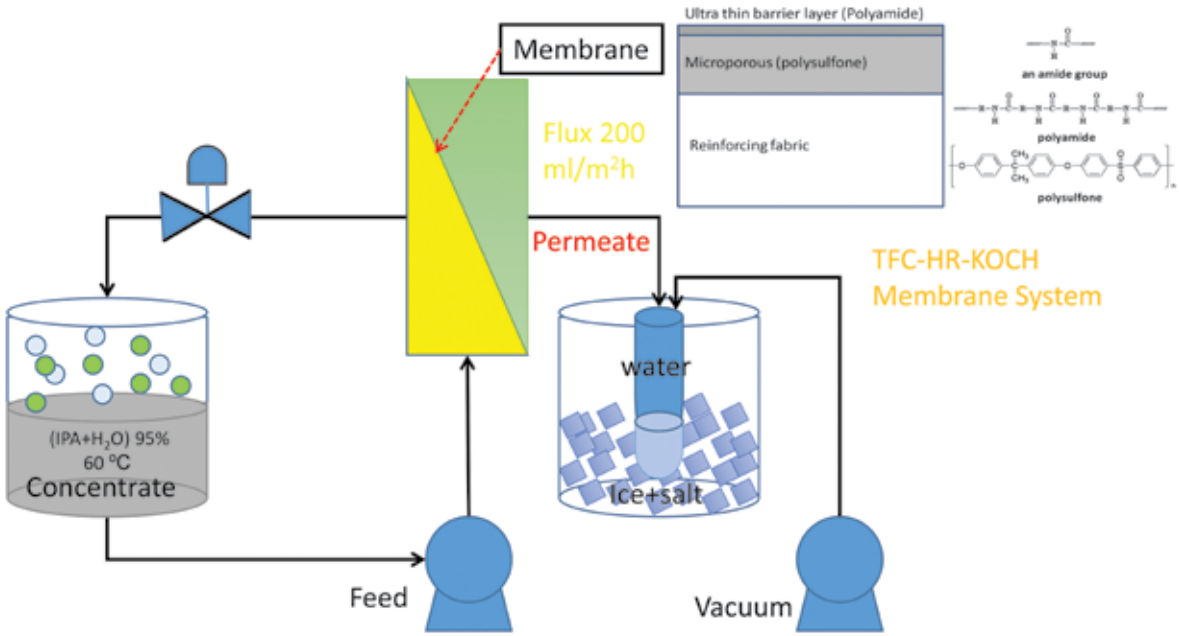

FIG. 6. Pervaporation apparatus experimental design

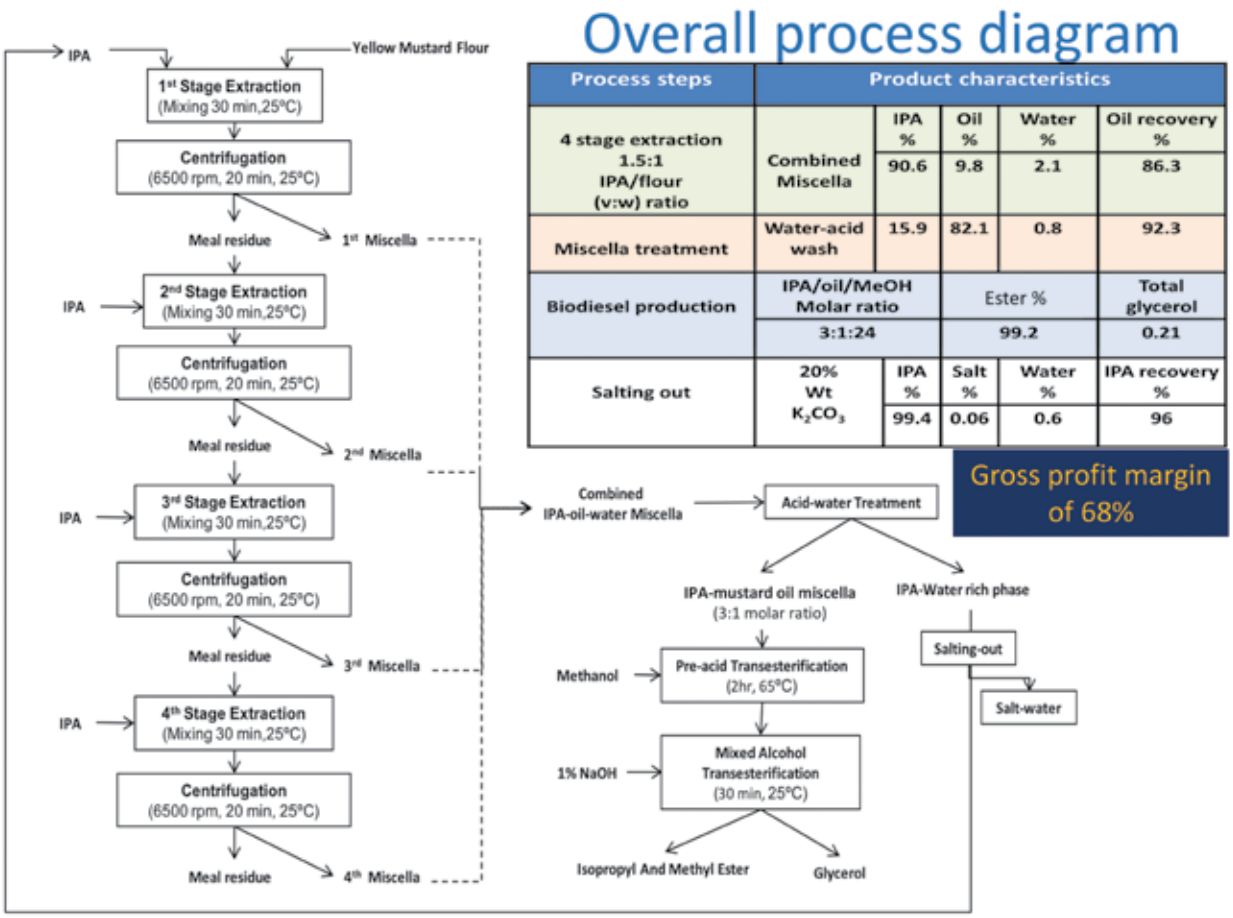

FIG. 7. Biodiesel production steps from yellow mustard flour

percentage of water removed in the batch was 48%. Water separation from IPA was possible through the TFC-HR membrane, however the process was very slow and the separation factor was lower than expected.

WHAT WAS LEARNED

A four-stage extraction process 1.5:1 IPA to flour (volume:-weight) ratio followed by mix alcohol transesterification using 1.2% potassium hydroxide resulted in a high (89%) ester yield. Miscella treatment with a water-acid mixture as well as the addition of a pre-acid step transesterification followed by a base catalyst transesterfication increased the ester yield to 99%. Most of the IPA (95%) can be recovered through application of a potassium carbonate salting out process at water concentration up to 13%. This is a highly attractive and economically viable process since an underutilized Canadian crop (yellow mustard seed) is used to produce food-grade protein products as well as high-grade biodiesel. Final investigations into the recovery and recycling of IPA refined this process and thereby made it even more economically attractive (Fig. 7).

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Something in the air

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

Rosa Richards

People in Europe and the United States spend an average of 90% of their time indoors, exposed to a variety of hazardous chemicals and other pollutants in the air and at risk of major and minor illnesses. Some workers, such as painters, are particularly exposed.

Volatile organic compounds (VOCs) originate from many building materials, including coatings, adhesives, and sealants. Some contain hazardous substances, such as toluene, benzene, chlorinated paraffins, and short-chain hydrocarbons. Others come from products used by the occupants, such as personal care and cleaning products containing odorants and fragrances, and dry cleaning solvents.

According to the US Environmental Protection Agency (EPA), VOC levels are typically up to 10 times higher indoors than outside. High levels are also found in indoor air samples in the United Kingdom, with most exceeding the Building Research Establishment Environmental Assessment Method (Breeam) limit for total VOCs (TVOCs), according to Tim Robinson, director of Waverton Analytics.

Waverton found the most common level of TVOCs to be 1,000 ug/m3 or more, in thousands of indoor air quality (IAQ) measurements taken from homes and buildings in the United Kingdom. These results are in line with tens of thousands of similar samples analyzed by the Prism Analytical Technologies laboratory in the United States.

The World Health Organization (WHO) has set evidence-based guidance limits for individual hazardous pollutants (HAPs) in indoor air. Although international green building certification schemes, like Breeam and WELL, set limits for TVOCs, there is no WHO-recommended limit, because different compounds have different toxicological profiles, so overall effects cannot easily be determined.

"TVOC gives you an indication of how problematic your air quality might be," says Alice Delia, lab director of Prism. "Almost all indoor air samples share the same basic set of sources. This is true of both homes and commercial spaces, such as offices.

"Although the levels of individual substances may vary between samples, the source signatures are almost always present and sometimes can constitute the majority of them. Most of ours are from buildings in the US but samples from other countries haven't been that different," Delia says.

Waverton singles out 17 HAPs that are commonly detected in homes and known or suspected to cause cancer or other serious health effects. The US National Institute for Occupational Safety and Health (Niosh) has developed exposure limits for most of these and two are on the REACH substances of very high concern (SVHCs) list: 1,2 dichloroethane and trichloroethene.

The most effective methods of minimizing air pollution include source avoidance, good ventilation, and air filtration. Using greener products and natural building materials with fewer hazardous chemicals can help to reduce the sources of pollution. Designing in more efficient ventilation systems ensures that filtered fresh air circulates.

Driven by tighter building regulations, clients demanding high-performance buildings and an increasing awareness of

the pervasiveness of poor IAQ and its effects, there is increasing interest in designing eco-friendly, healthy environments for living and working.

IAQ is therefore a hot topic for architects. "The energy efficiency of buildings has improved, but ventilation also needs to be addressed—usually mechanically," says Tom Kyle, associate at Sheppard Robson, an architectural practice which specializes in sustainable design.

Ventilation is often inadequate between the completion or renovation of buildings and their occupation. The systems may not be up and running until long after finishes have dried. This can lead to VOCs that are emitted by drying paint and adhesives being trapped in the building, according to Clare Perkins, a materials scientist at Arup.

"Once the building is conditioned, the VOC concentration in air will be removed pretty quickly," Perkins adds. "Off-gassing is faster from thinner finishes like paint or varnish, so levels approach acceptable faster than from thicker materials like floor finishes."

When specifying building materials and products, architects rely to some extent on market regulations. Polychlorinated biphenyls (PCBs), lead, asbestos and mercury are generally banned from new builds, although they may be historically present in refurbishments. There are also regional restrictions on hazardous substances, like formaldehyde in timber board products and VOCs in paints and coatings.

"In Canada, big interior surface materials are for the most part low-VOC," says Simon Richards, principal architect at Cornerstone Architecture in Vancouver.

"I avoid specifying vinyl and use linoleum instead which has some off-gassing, but most clients find that acceptable. Most products are well regulated, but my engineering contacts say that one major scrutiny gap is in the adhesives used in building construction."

Sustainable building certification schemes like WELL go beyond regulatory requirements and give optional credits for toxic material reduction and precautionary materials selection. The WELL standard bans isocyanate-based polyurethane products in interior finishes and sets recommended limits for:

- perfluorinated chemicals (PFCs) in furniture and furnishings;
- flame retardants in building materials, textiles and furniture;
- phthalates in flooring, wall coverings, blinds and furniture; and
- urea formaldehyde in furniture, composite wood products, laminating adhesives and resins, and thermal insulation.

Further optional credits are given if at least 25% of furnishings, built-in furniture, interior finishes, and finish materials are accredited with certain ecolabels, such as Living Building Challenge Compliant or Certified Cradle to Cradle Material Health, or by other criteria.

Reduction or management plans for other chemicals, such as pesticides and cleaning products that are used during occupation of the building, are also prerequisites.

"Large companies which offer private healthcare packages are driving the adoption of the WELL building certification scheme in the UK, as they have an incentive to increase employee well-being and reduce health care bills," Kyle says. "Institutional clients take a long-term view of the design of their buildings. House builders, on the other hand, just aren't that interested."

In Canada, like the United Kingdom, sustainable building design has been largely driven by building regulations—the federal building codes. Vancouver has energy efficiency targets to make buildings net zero by 2032. These are strongly based on the Passivhaus building standard.

Richards says that the government is driving industry. "My practice has moved from the LEED building standard, which tries to be all-encompassing and is too burdensome, to the Passivhaus standard, which has simpler objectives, is therefore more effective and has spin-off benefits, including better ventilation," he adds.

Passivhaus results in the design of ultra-low energy buildings that require little energy for space heating or cooling. The benefits are environmental: comfort, IAQ, noise containment, and energy reduction. Continuous mechanical or natural ventilation is currently mandatory in small buildings in Vancouver, but the next building code will make it so for all buildings.

Richards recognizes a drawback in moving towards the Passivhaus standard, though. "In moving to Passivhaus, we have lost post-construction testing of IAQ, which verified whether the specification and construction standards were appropriate or successfully executed under LEED. WELL is increasingly referred to in Canada and could be a good complement to Passivhaus," he says.

Some sustainable building certification schemes are stricter than others on minimizing the sources of hazardous chemicals in indoor air. In general, WELL addresses indoor air pollution more comprehensively, with a 33 microgram/m³ limit on formaldehyde in ambient air.

There are also optional credits for toxic materials reduction; precautionary materials selection; strategies for pesticide elimination; and cleaning methods during occupation.

Breeam has a tighter limit on TVOCs (300 microgram/m³) than WELL (500 microgram/m³). Under the scheme, air quality testing takes place post-completion and before occupation, when the level of VOCs is still high.

Testing for WELL is undertaken up to one year after occupation, by which time VOC levels are likely to be lower. Under LEED, optional IAQ testing takes place before occupancy to ascertain whether recommended limits have been met.

Thus, under LEED and Breeam, there is less incentive to reduce indoor air pollution. Optional credits can be gained by taking measures to address it, but clients may prefer to install water and energy efficiency measures to gain credits, meet or exceed building regulations and make savings on utility bills.

LEED has slightly more lenient standards in terms of IAQ. Although it requires ventilation and a minimum air flow, other measures, like flush-out before occupation and phased flush out or IAQ testing to demonstrate compliance with limits, are part of a voluntary credit.

Under LEED, voluntary credits can be earned for using low VOC materials in set categories. Other chemicals used by occupants, like air fresheners and personal care products, some of which have endocrine disrupting properties, are detected in indoor air but are outside the scope of any of these building schemes.

Domestic customers demand and expect "safe" building materials and products when decorating their homes. Occasional scare stories, such as a recent US study where PCBs were detected in air and traced back to kitchen cabinets containing polymer resin, can make customers wary.

Consumers can use ranking systems, like "Mind the Store," to choose the retailer with the best overall score for toxic chemicals. Retailers also have a reputational risk if they sell products containing hazardous substances. Domestic DIY retailers and builders' merchants have consequently developed policies on sustainable products and safer chemicals.

The Kingfisher Group, which owns do-it-yourself (DIY) retailers in many countries, has a stated "commitment to the responsible use of chemicals in our products and supply chain and by promoting healthy homes and gardens for our customers." This takes a proactive approach, going beyond regulatory requirements and undertaking innovation in sustainable chemistry.

Ikea, which Mind the Store ranked second among furniture and home goods retailers last year, aims to "refrain from the use of chemicals and substances that can be harmful to people and the environment" and takes a "precautionary approach" in line with REACH. If legislation on chemical use is tightened in one region, the company adopts this as a minimum in other countries where it does business.

Many actors in the supply chain keep track of hazardous chemicals in building materials and products. Legislation has reduced the use of the most hazardous of these in construction products.

Engineering firms stay informed to advise clients on specification of materials. Green building certification schemes are updated with newly classified hazardous substances in order to control their use and to set lists of pre-approved products.

Manufacturers and retailers of building materials also monitor chemical regulation in order to keep their products compliant or to go beyond requirements and reduce reputational risk.

IAQ argues for a precautionary approach from the specification and installation of materials, to the use of products, to ventilation and other measures to ensure a healthy environment when a building is in use. WELL, an aspirational evidence-based building standard, is increasingly being adopted around the world to achieve this.

Rosa Richards is a journalist with Chemical Watch.

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What impacts are UK SMEs experiencing in their supply chains in the run-up to Brexit?

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

Elaine Burridge

As things stand, the UK will cease to be a member of the EU on March 29, 2019. At the time this issue of *Inform* went to press, there were dire warnings about what might happen and news of secret government contingency plans. How have companies been dealing with the uncertainty?

Executives at small and medium enterprises (SMEs) are often unwilling to speak on controversial matters or prefer to do so anonymously, but among those who were interviewed, some common themes emerged. To date, the direct impacts on the supply chain have been minimal, but UK companies remain highly concerned, above all because of the continuing uncertainty over what form Brexit will ultimately take.

Geoff Mackrill, managing director of marine coatings manufacturer Teal & Mackrill, and Lynda Platts, managing director of ACP Solutions, a maker of insect repellents, agree that the biggest impact they have felt so far has been on costs. These have risen as a result of the decline in sterling, which is itself attributed in large measure to the Brexit vote and resulting uncertainty.

ACP has seen quite a few raw material price rises, Platts says. For example, the active ingredient used in one of its products is made in Germany and its price has increased by 10% since the vote. Notifications from suppliers have pointed to the decline in the value of the pound as the key reason for the increases and she fears that there is probably more to come.

ACP's biggest market is continental Europe, where it sells around 70% of its products. Platts notes that the company operates in a low-price, very price-sensitive market, with many manufacturers in Europe selling similar products. As a result, prices may go up to the point where its product is no longer affordable for its European distributors.

"We offer low-value goods with strong EU competition. The falling pound, rising costs, and potential tariffs will make us uncompetitive. We will certainly have to take the company offshore. I do not see how we can survive otherwise. With the impact on raw materials and duties, I do not see how we can remain competitive," she says.

Some companies are seeing other effects already. The technical director of an SME that formulates industrial cleaning detergents and paint removers notes that, since the Brexit vote, deliveries have been slightly slower and UK distributors are holding less stock of European-made materials. Whether

this will remain a long-term issue, he says, depends mainly on what kind of Brexit there will be.

Meanwhile, a senior manager at a distributor of DIY chemicals says that the company has been asking up the supply chain about product availability. People are "hedging their bets" and waiting on developments, while customers are doing more due diligence work because of Brexit. After Brexit, the company will move to become an importer. This, he says, is a dramatic shift of responsibility and the infrastructure required for it cannot be put in place in a short timeframe.

A director at a specialty chemicals and ingredients supplier says that not knowing what is happening with regards to Brexit is the biggest problem for him at the moment. The company expects that customers will start moving their business away from UK manufacturers and suppliers and that European companies will turn to local firms instead.

This company does not have the financial capacity and international footprint to move assets around but those which do are looking into it. Platts and her husband, who were looking toward selling up and retiring to Italy, have brought their plans forward and will seek to acquire residency and move the business there too. "There is nothing we cannot do in Italy that we do in the UK. We can protect our market in the EU from Italy," she says.

The director at the detergents and paint removers company says it will have to shift some production to contract manufacture and is now in talks with potential contract manufacturers. "We can move quite quickly in a hard Brexit scenario," he says but in his view avoiding this should be government's top priority.

The do-it-yourself (DIY) products distributor has not made any firm business decisions as a result of Brexit yet, but it is looking at new materials suppliers and has invested in software for compliance, tracking, and volumes. "We need a timeline to get answers.

Do we have three months, six months or two weeks? Give us an indication of where you are," the director urges the authorities.

Because any company with a biocide will have to have a host company in the EU, Teal & Mackrill will be opening a subsidiary in a member state, although the uncertainty has made investment decisions more risky. Mackrill adds that the company is also looking at forming partnerships in Europe, possibly for manufacturing but maybe just administrative.

The SMEs who were interviewed all agree that the UK should stay within the EU regulatory framework, including some form of associate membership of Echa. A particular concern is that Echa and other agencies are not communicating with UK firms until there is clarity on what form Brexit will take.

There will be a long-term problem without Echa membership Mackrill believes. "We need a level playing field across Europe," he says. "Companies that export are going to have to comply with EU regulations. We just need to know if that is going to happen or not, and we need a commitment from the government."

The director at the specialty chemicals firm, however, warns that associate membership could come with strings attached. "We will find the direction to go and sell internationally thwarted. We could end up with the best of both worlds or the worst. I think it will be the worst. There is no way of coming out of this without some considerable restrictions."

Interviewees cite many other short- and long-term worries. The potential for immediate loss of customers is massive. There will also be new government computer systems and unknown tariff codes, with companies needing to adjust their internal stock codes, major long-term effects on taxation and tax legislation, and the direct cost of tariffs.

In addition, some fear that exporting costs will increase significantly, as will transport times, because Customs & Revenue does not have enough staff to cope. Symptomatic, perhaps, of how some are having to go looking for information, one mentions a "fairly sensible" source saying that Customs & Revenue has cleared an area in Belgium to hold trucks in the expectation of delays resulting from handling the paperwork.

Another potential impact is where products hitherto covered by EU legislation will need a licence and documents for export. The Home Office has not prepared a UK license yet, and more support will be necessary for an increased number of license applications.

Without a trade deal, says Mackrill, Teal & Mackrill may have to help its distributors, if their costs increase as a result of extra administration and tariffs. In this scenario, EU27 companies will find it easier to do business with each other, although UK companies can still trade if they have a good product. "We should be able to export, but there will be cost implications," he says.

Other concerns center around REACH and whether UK REACH registrations will be legitimate if there is a hard Brexit, as well as the implications for other major UK industries, like pharmaceuticals, food, and personal care. Even if there are potential advantages from Brexit, chemical SMEs doubt that the government has the political strength or understanding of global supply chains to capture them, particularly with international trade wars a real threat.

"If we leave the EU, just when a trade war starts, we could be very vulnerable. It would be the perfect storm," says the director of the specialty chemicals company. "There has been no political decision made and the longer that goes on, the closer to the cliff edge we are."

Platts adds: "I do not believe that we are going to get the deal with the US that we hoped for. Where can we take this without killing the UK economy?" She believes there should be a second referendum, stating: "The country is allowed to change its mind if it chooses."

Elaine Burridge is a reporter at Chemical Watch.



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LATAM: Dairy alternatives

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

Leslie Kleiner

In some world regions, dairy alternatives have been growing at a fast pace. As an R&D Product Developer myself, I am currently working on dairy alternatives based on the use of pea protein. My day-to-day activities made me curious about the role of dairy alternatives in LATAM. To learn more, I consulted a recently published "Category Insider: Dairy Alternatives: Global report" (June 2018) from Innova Market Insights (www.innovadatabase.com) [1]. Below is what I learned, in Q&A format.

Q: What is the dairy alternative consumption (milks, yogurts, ice cream, cheese, and so on) in leading countries for the category, and what is the consumer's perspective on these products?

Despite rapid growth in the United States and United Kingdom, Asia is the most important region for this category, with China being the largest single market overall (38% of global retail value) compared to 19% for the United States. Global sales of dairy alternative drinks in 2017 were US $13.4 billion at retail level.

In the United Kingdom and United States, a consumer research study of 500 consumers (ages 18–55) indicated that two-thirds of consumers occasionally include plant-based dairy in their diets. Of these consumers, approximately 10% consider the products to be daily staples, and 30% are increasing their intake. Half of the consumers view dairy alternatives to be more healthful than cow's milk and products derived from cow's milk. The most preferred plant sources are almond and coconut, due to their palatability.

Figures 1 and 2 show data from a consumer study in the United States and United Kingdom that illustrates consumers' perceptions of dairy alternatives.

Q: What is the role of soy in dairy alternatives?

Although soy continues to be the most important plant-base for dairy alternatives, it has lost considerable share due to more sustainable and palatable alternatives. It is now common to see almond, oat, rice, and other sources in new product development (NPD).

Outside of Innova reports, it is worth mentioning that pea protein is quickly gaining market share due to its image with respect to sustainability, protein quality, versatility during processing, and not being a major allergen, such as soy and other nuts.

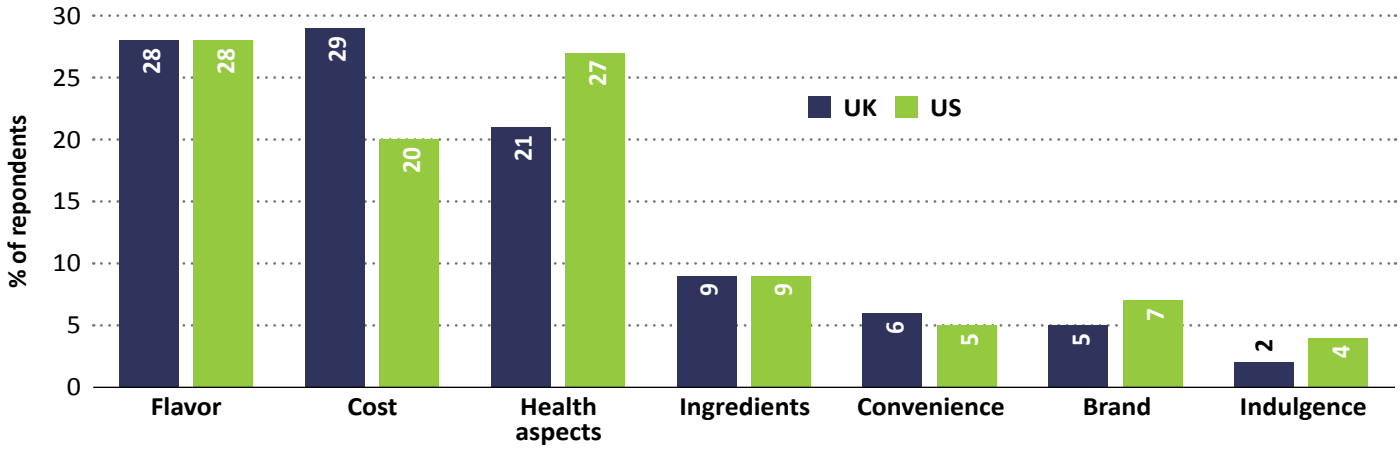

FIG. 1. Factors that consumers consider important when purchasing dairy alternatives (UK and US, 2017). Source: Innova Market Insights

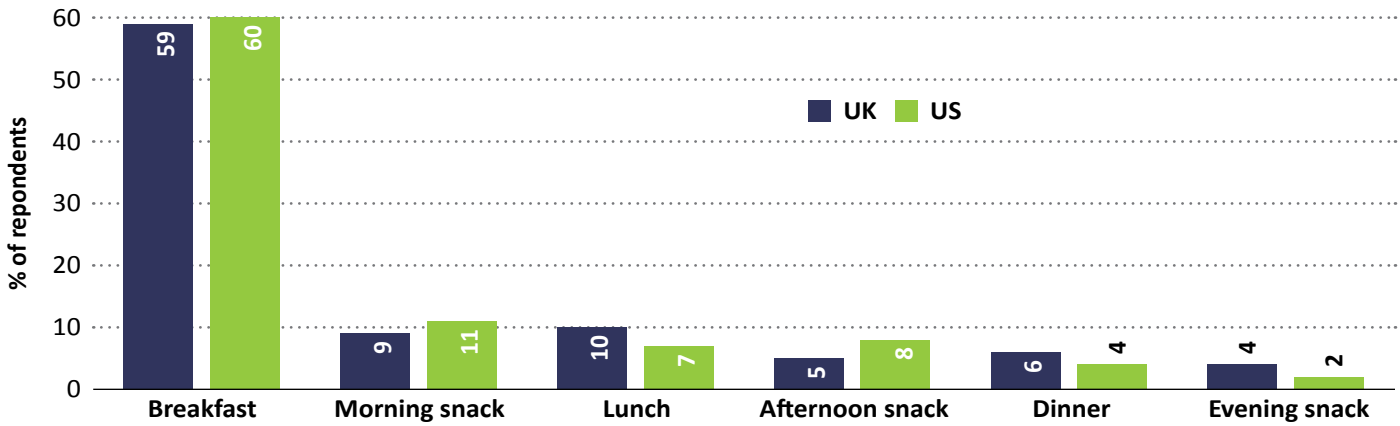

FIG. 2. Circumstances for consumption of dairy alternatives (UK and US, 2017) Source: Innova Market Insights

Q: How are dairy alternatives placed in the LATAM region? What trends are representative of new NPD in this field?

LATAM accounts for 5.3% of the 2017 global dairy alternative drinks market value by region (% retail sales). Brazil is one of the top 10 countries within this group (following 2017 retail sales in China, the United States, Japan, Canada, Thailand, Spain, the United Kingdom, and France, and preceding Germany at the end of the list). However, neither Brazil nor any other LATAM country made the list of fastest-growing countries by value for the 2010–2022 forecasted period, which includes China, Canada, Serbia, the United States, Ukraine, the United Kingdom, Thailand, Vietnam, Romania, and Indonesia.

In Brazil, NPD includes dairy alternatives to drinks such as milk and flavored shakes, as well as powdered drinks. Many of the ingredients used are local, such as quinoa and cashew nut. There have also been some introductions of non-dairy yogurt, ice cream, and cheese.

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.

Five review articles headline *JAOCS* special issue focused on plant and alternative proteins

The August issue of the *Journal of the American Oil Chemists' Society* (*JAOCS*) includes 18 articles focused on the sources, quality, processing, modification, and applications of plant and other alternative proteins.

Wanasundara

Hojilla-Evangelista

This special issue is the result of a fruitful collaboration between *JAOCS* and the AOCS Protein and Co-Products (PCP) Division (https://tinyurl.com/yazeazod). More than 15 members from the PCP Division contributed to the issue organized by guest editors Janitha P.D. Wanasundara and Mila P. Hojilla-Evangelista, who had the vision and commitment to see this project through to completion. The papers presented provide a snapshot of currently available research and information in the growing interest area of plant proteins and alternative proteins, which according to the guest editors, is quickly becoming its own industry.

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JAOCS, VOLUME *95*(8)

Commentary: Achieving your goals by helping others achieve their goals
Kenar, J. A.

Editorial: Satisfying protein demand with plant and alternative proteins
Wanasundara, J.P.D. and M.P. Hojilla-Evangelista

Review: Protein Solubilization
Sathe, S.K., V.D. Zaffran, S. Gupta, and T. Li

Technoeconomic prospects for commercialization of *Brassica* (cruciferous) plant proteins
Mupondwa, E, X. Li, and J.P.D. Wanasundara

Changes in corn protein content during storage and their relationships to dry grind ethanol production
Ramchandran, D., *et al.*

Review: Grain thin stillage protein utilization: a review
Ratanapariyanuch, K., Y.Y. Shim, D.J. Wiens, and M.J.T. Reaney

Review: Rice endosperm and bran proteins. a review
Al-Doury, M.K.W., N.S. Hettiarachchy, and R. Horax

Composition and phosphorous profile of high-protein rice flour and broken rice, and effects of further dry and wet processing
Liu, K.

Review: Making kafirin, the sorghum prolamin, into a viable alternative protein source
Taylor, J., and J.R.N. Taylor

Structural characterization and functional properties of proteins from oat milling fractions
Walters, M.E., C.C. Udenigwe, and A. Tsopmo

Composition and functional properties of saline-soluble protein concentrates prepared from four common dry beans (*Phaseolus vulgaris* L.)
Hojilla-Evangelista, M.P., *et al*.

Stability and bioavailability of curcumin in mixed sodium caseinate and pea protein isolate nanoemulsions
Yerramilli, M., N. Longmore, and S. Ghosh

Application of barley and lentil protein concentrates in the production of protein-enriched doughnuts
Eckert, E., *et al*.

Yogurt fortification with chickpea (*Cicer arietinum*) flour: physicochemical and sensory effects
Chen, X., M. Singh, K. Bhargava, and R. Ramanathan

Emerging camelina protein: extraction, modification and structural/functional characterization
Boyle, C., L. Hansen, C. Hinnenkamp, and P.B. Ismail

Review: Plant RuBisCo: an underutilized protein for food applications
Di Stefano, E., D. Agyei, E.N. Njoku, and C.C. Udenigwe

Review: Canola Protein—A promising protein source for delivery, adhesive, and material applications
Bandara, N., A. Akbari, Y. Esparza, and J. Wu

Preparation and properties of solution cast films from pennycress protein isolate
Selling, G.W., *et al*.

Antioxidant properties of flaxseed protein hydrolysates: influence of hydrolytic enzyme concentration and peptide size
Nwachukwu, I.D. and R.E. Aluko

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Identification of phytyl vaccinate as a major component of wax ester fraction of extra virgin olive oil

Mariani, C., *et al. Eur. J. Lipid Sci. Technol. 120*: 1800154, 2018, https://doi.org/10.1002/ejlt.201800154.

Despite the great efforts devoted to the study of the chemical composition of olive oil, the identification of minor components is still incomplete. Within this context, the characterization of esters of fatty alcohols with fatty acids is far from complete. The objective of this study is to investigate phytyl wax esters profile of extra-virgin olive oils to provide an update on the current knowledge of the chemical composition of this fraction. Extra-virgin olive oil waxes are transesterificated with HCl/isobutanol and isobutyl esters are analyzed by GC-FID and GC-MS using two capillary columns with different polarity. A comparison of the GC retention times with analytical standards together with the acquired mass spectra allow to identify and confirm, for the first time, phytyl vaccinate as the relevant unknown peak detected in the wax esters fraction of extra-virgin olive oils. Additionally, the analysis of the composition of aliphatic alcohols obtained by hydrolysis of waxes reveals that the preponderant compound is C24 alcohol derived from lignoceryl palmitate, which is practically the only wax really present, while the low level of C28 alcohol confirms that extra-virgin olive oils of good quality contain negligible amounts of long-chain waxes which are derived from C28 alcohol.

Effect of conjugated linoleic acid on blood inflammatory markers: a systematic review and meta-analysis on randomized controlled trials

Haghighatdoost, F. and B. Fatemeh Nobakht M. Gh, *Eur. J. Clin. Nutr. 72*: 1071–1082, 2018, https://doi.org/10.1038/s41430-017-0048-z.

Conjugated linoleic acid (CLA) is a polyunsaturated fatty acid with attractive biological activities. Numerous studies have been conducted on the inflammation-lowering effects of CLA in *in vitro* and in animal models. However, the effects of CLA treatment on the inflammatory markers in humans are controversial. Therefore, the objective of this study was to perform a systematic review and meta-analysis on controlled clinical trials (RCT) assessing the effects of CLA supplementation on circulating inflammatory markers, including C-reactive protein (CRP), interleukin-6 (IL-6), and tumor necrosis factor-alpha (TNF-alpha). The literature search of RCTs was performed using Pubmed/Medline, Scopus, ScienceDirect, Web of science, Cochrane, and Google Scholar databases from inception to March 2017. Weighted mean differences were estimated, and the pooled effect size was calculated by a random effects model. Of the 427 identified studies, eleven RCTs, including 420 subjects were included in the statistical analysis. Findings suggested that CLA supplementation increased blood levels of CRP by 0.89 mg/l (95% CI: 0.11, 1.68; $P = 0.025$) and TNF-α levels by 0.39 pg/ml (95% CI: 0.23, 0.55; $P < 0.0001$). However, blood IL-6 levels were marginally decreased by 0.32 pg/ml (95% CI: −0.71, 0.07; $P = 0.11$) following CLA supplementation. There was a significant heterogeneity for the impact of CLA on CRP and IL-6, but not TNF-alpha. This meta-analysis showed that CLA supplementation may increase inflammatory markers (CRP and TNF-alpha). There are concerns about using CLA supplementation as an anti-obesity agent among the obese population for at least a short duration.

Fatty-acid derivative acts as a sea lamprey migratory pheromone

Li, K., *et al., Proc. Nat. Acad. Sci. 115*: 8603–8608, 2018, https://doi.org/10.1073/pnas.1803169115.

Olfactory cues provide critical information for spatial orientation of fish, especially in the context of anadromous migrations. Born in freshwater, juveniles of anadromous fish descend to the ocean, where they grow into adults before migrating back into freshwater to spawn. The reproductive migrants, therefore, are under selective pressures to locate streams optimal for offspring survival. Many anadromous fish use olfactory cues to orient toward suitable streams. However, no behaviorally active compounds have been identified as migratory cues. Extensive studies have shown that the migratory adult sea lampreys (*Petromyzon marinus*), a jawless fish, track a pheromone emitted by their stream-dwelling larvae, and, consequently, enter streams with abundant larvae. We fractionated extracts of larval sea lamprey washings with guidance from a bioassay that measures in-stream migratory behaviors of adults and identified four dihydroxylated tetrahydrofuran fatty acids, of which (+)-(2S,3S,5R)-tetrahydro-3-hydroxy-5-[(1R)-1-hydroxyhexyl]-2-furanoctanoic acid was shown as a migratory pheromone. The chemical structure was elucidated by spectroscopies and confirmed by chemical synthesis and X-ray crystallography. The four fatty acids were isomer-specific and enantiomer-specific in their olfactory and behavioral activities. A synthetic copy of the identified pheromone was a potent stimulant of the adult olfactory epithelium, and, at 5 × 10−13 M, replicated the extracts of larval washings in biasing adults into a tributary stream. Our results reveal a pheromone that bridges two distinct life stages and guides orientation over a large space that spans two different habitats. The identified molecule may be useful for control of the sea lamprey.

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Impact of reduced dietary levels of eicosapentaenoic acid and docosahexaenoic acid on the composition of skin membrane lipids in Atlantic salmon (*Salmo salar* L.)

Cheng, K., *et al., J. Agric. Food Chem. 66*: 8876–8884, 2018, https://doi.org/10.1021/acs.jafc.8b02886.

Membrane lipids, including sphingolipids and glycerol-phospholipids, are essential in maintaining the skin's barrier function in mammals, but their composition in fish skin and their response to diets have not been evaluated. This study investigated the impacts of reducing dietary eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) on membrane lipids in the skin of Atlantic salmon through a 26-week feeding regime supplying different levels (0–2.0% of dry mass) of EPA/DHA. Ceramide, glucosylceramide, sphingomyelin, sphingosine, and sphinganine in salmon skin were analyzed for the first time. Higher concentrations of glucosylceramide and sphingomyelin and higher ratios of glucosylceramide/ceramide and sphingomyelin/ceramide were detected in the deficient group, indicating interruptions in sphingolipidomics. Changes in the glycerol-phospholipid profile in fish skin caused by reducing dietary EPA and DHA were observed. There were no dietary impacts on epidermal thickness and mucus-cell density, but the changes in the phospholipid profile suggest that low dietary EPA and DHA may interrupt the barrier function of fish skin.

Hexane- and ethanol-extracted seed oils and leaf essential compositions from two castor plant (*Ricinus communis* L.) varieties

H.M. Sbihi, *et al., Ind. Crop. Prod. 122*: 174–181, 2018, https://doi.org/10.1016/j.indcrop.2018.05.072.

The choice of organic solvent plays an important role in the degree and nature of lipid extraction. This work aimed to first evaluate the effect of solvents (hexane an ethanol) used for the extraction of castor oils on their physicochemical properties and compositions. Second, the chemical compositions of the essential oils obtained from fresh and dried leaves of castor plants were studied by gas chromatography–mass spectroscopy analysis. Two castor plant varieties grown in Saudi Arabia (*Ricinus communis zanzibariensis* and *Ricinus communis impala*) were used in this study. The results showed that the tocol and sterol contents were significantly affected by the type and variety of the solvent. Moreover, the presence of minor fatty acids was related to the type of solvent. The ricinoleic acid and oil yields were not affected by the type of solvent but were affected by the variety. The chemical compositions of the essential oils obtained from fresh and dried leaves were characterized by high concentrations of fatty acid methyl esters (38.42%) in *Ricinus communis zanzibariensis* and ketone (29.67%) and aldehyde (24.77%) components in *Ricinus communis impala*. This work has proved that the quality of castor oils is related to the type of solvent extraction and the variety.

Cloning and functional characterization of long-chain acyl-CoA synthetase 1 from the mesocarp of African oil palm (*Elaeis guineensis* Jacq.)

Zheng, Y.-S., *et al., Ind. Crop. Prod. 122*: 252–260, 2018, https://doi.org/10.1016/j.indcrop.2018.06.003.

African oil palm (*Elaeis guineensis* Jacq.) is the highest-yielding oil crop in the world. Recent studies have shown the importance of long-chain acyl-CoA synthetases (LACSs) in lipid biosynthesis and degradation, but few studies have focused on oil palm. In this work, we found that transcription of EgLACS1was positively correlated with lipid accumulation in the oil palm mesocarp. To clarify the role of EgLACS1 on lipid metabolism, three variants of the EgLACS1 gene were cloned from oil palm and designated EgLACS1-V1, EgLACS1-V2, and EgLACS1-V3. Sequence analysis revealed that the three variant proteins contained several highly conserved motifs, shared important sequence similarities with other known LACS1s, and demonstrated that EgLACS1-V1, EgLACS1-V2, and EgLACS1-V3 were 666-, 664-, and 601-amino acid proteins, respectively. All of the three variant genes encoding-EgLACS1 were expressed and tested for their functionality in an acyl-coenzyme-A synthetase- (ACS-) deficient mutant yeast strain YB525. Only EgLACS1-V1 and EgLACS1-V2 could rescue the growth deficiency (an inability to grow on a fatty acid auxotrophic medium) of YB525 in complementation tests. Using a fluorescent fatty acid analogue confirmed that EgLACS1-V1 and EgLACS1-V2 facilitated exogenous fatty acid uptake. EgLACS1 overexpression in yeast reduced fatty acid content. All these results suggested that not only did both the EgLACS1-V1 and EgLACS1-V2 enzymes exhibit acyl-coenzyme-A activities, but that they were also involved in the transfer of fatty acid-coenzyme A. They may also take part in the storage of lipid during fruit development in oil palm. The characterization of EgLACS1 will provide a molecular basis for the study of acyl-coenzyme A synthetase-mediated lipid synthesis and degradation in oil palm.

Lipid Oxidation/Antioxidants

Overview on mitigation of acrylamide in starchy fried and baked foods

Baskar, G. and R. Aiswarya, J. Sci. Food Agric. 98: 4385–4394, 2018, https://doi.org/10.1002/jsfa.9013

Acrylamide in fried and baked foods has the potential to cause toxic effects in animals and humans. A major challenge lies in developing an effective strategy for acrylamide mitigation in foods without altering their basic properties. Food scientists around the world have developed various methods to mitigate the presence of acrylamide in fried food products. Mitigation techniques using additives such as salts, amino acids, cations, and organic acids along with blanching of foods have reduced the concentration of acrylamide. The use of secondary metabolites, such as polyphenols, also reduces acrylamide concentration in fried food products. Other

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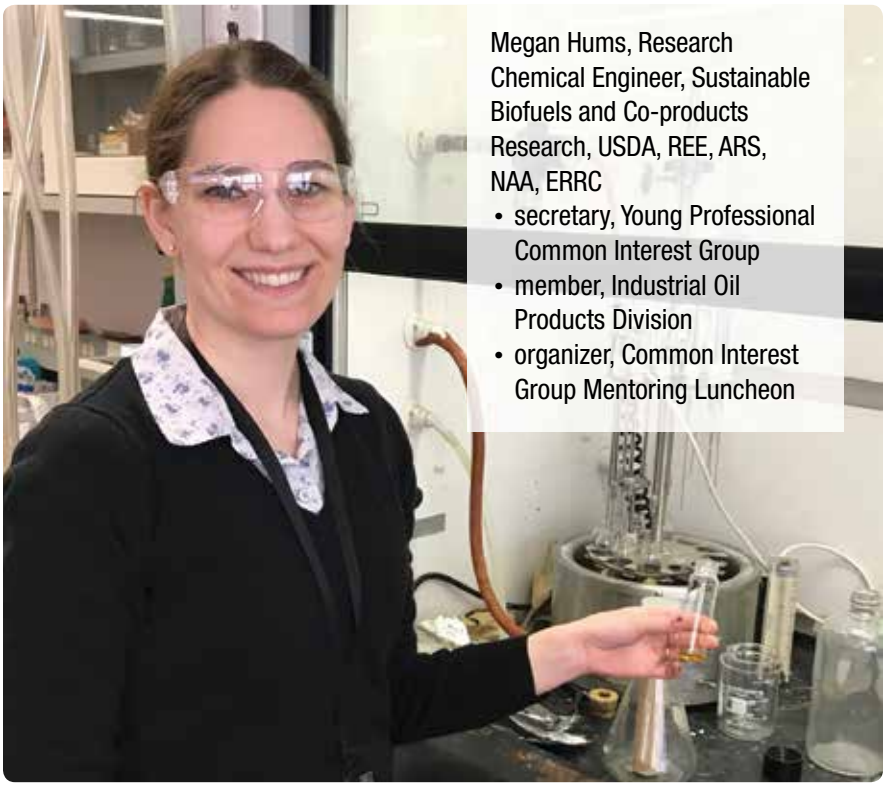

Megan Hums, Research Chemical Engineer, Sustainable Biofuels and Co-products Research, USDA, REE, ARS, NAA, ERRC

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mitigation techniques, such as asparaginase pre-treatment and low-temperature air frying with chitosan, have been effective in mitigating the concentration of acrylamide. The combined pre-treatment process along with the use of additives is the latest trend in acrylamide mitigation.

Phytochemical profile and antioxidant activity of caper berries (*Capparis spinosa* L.): evaluation of the influence of the fermentation process

Jiménez-López, J., *et al*., *Food Chem. 50*: 54–59, 2018, https://doi.org/10.1016/j.foodchem.2018.01.010.

In this work, we report the phytochemical profile and antioxidant activity of caper berries (*Capparis spinosa* L.) before and after a fermentation process. The phytochemical profiles were evaluated by high-performance liquid chromatography with UV and electrospray ionization mass spectrometry detection (HPLC-DAD-ESI-MSn). Twenty-one compounds were characterized, and seven of them quantified. The main component of non-fermented berries was glucocapparin, which was degraded upon the fermentation process. Most of the compounds were quercetin and kaempferol glycosides, epicatechin, and proanthocyanidins. The main differences observed upon the fermentation process were a decrease in epicatechin concentration, the hydrolysis of quercetin glycosides, and the degradation of glucosinolates. Total phenolic and flavonoid contents, as well as the antioxidant activities by the *in vitro* antioxidant assays DPPH and ABTS+, were determined, observing that the values were slightly higher after the fermentation process.

Comparison of the oxidative stability of soybean and sunflower oils enriched with herbal plant extracts

Kozłowska, M. and E. Gruczyńska, *Chem. Papers 72*: 2607–2615, 2018, https://doi.org/10.1007/s11696-018-0516-5.

The present study was conducted to determine and compare the oxidative stability of soybean and sunflower oils using differential scanning calorimetry (DSC). These edible oils were enriched with marjoram (*Origanum majorana* L.), thyme (*Thymus vulgaris* L.), and oregano (*Origanum vulgare* L.) extracts at three different concentrations and synthetic antioxidant (BHA). The fatty acid composition of studied oils was determined by gas chromatography mass spectrometry to evaluate the content of unsaturated fatty acids that are sensitive to oxidation. Oil samples were heated in the DSC at different heating rates (4.0, 7.5, 10.0, 12.5, and 15.0°C min^{-1}) and oxidation kinetic parameters (activation energy, pre-exponential factor, and oxidation rate constant) were calculated. The results showed that the oxidative stability of sunflower oil samples enriched with oregano extracts and soybean oil supplemented with thyme extracts was improved compared to samples without the addition of herbal plant extracts and the synthetic antioxidant.

Optimization of preparation process of beta-cyclodextrin inclusion compound of clove essential oil and evaluation of heat stability and antioxidant activities *in vitro*

Ma, S., *et al*., J. *Food Measurement and Characterization 12*: 2057–2067, 2018,https://doi.org/10.1007/s11694-018-9820-6.

To investigate the inclusion process of clove essential oil (CEO), saturated solution method was performed and beta-cyclodextrin (beta-CD) was used as wall materials. Based on the experimental results of single-factor experiments, response surface methodology (RSM) was employed to optimize the inclusion process. Ultraviolet spectrophotometry (US) was then used to verify the existence of inclusion complexes. Then, heat stability and antioxidant activities under different temperature were determined to estimate the inclusion process. 2,2′-diphenyl-1-picryhydrazyl (DPPH) scavenging capacity, 2,2′-azino-bis(3- ethylbenzothiazoline-6-sulphonicacid) (ABTS) scavenging capacity, anti-lipid peroxidation capacity, and ferric- reducing antioxidant power were used to determine the antioxidant capacities. The results showed that the optimum inclusion process of CEO was as follows: temperature, 43°C, time, 2.5 h and ratio of beta-CD to CEO, 9:1. Besides, inclusion process could strongly protect CEO from volatilization. This paper provided useful information to utilize the resource of Chinese herbal medicine and testified the great antioxidant activities of beta-CD inclusion compound of CEO, and the optimized inclusion conditions of CEO from clove could be used in industrial production of health food.

A comprehensive study of polyphenols contents and antioxidant potential of 39 widely used spices and food condiments

Assefa, A.D., *et al*., *J. Food Measurement and Characterization 12*: 1548–1555, 2018, https://doi.org/10.1007/s11694-018-9770-z.

Spices and condiments are rich sources of potent antioxidants. In the present investigation, total equivalent antioxidant capacities (TEAC) of 39 spices were studied using 1,1-diphenyl-2-picrylhydrazyl (DPPH) scavenging, 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) scavenging, and cupric reducing antioxidant capacity (CUPRAC) assays. Their total phenolic contents (TPC) and total flavonoid contents (TFC) were also determined. TEAC, TPC, and TFC varied significantly ($P < 0.05$) among these spices. ABTS, DPPH, and CUPRAC assay values of studied spices ranged from 1.42 to 112.94 mg ascorbic acid equivalents (AAE)/g, 1.14 to 91.09 mg Trolox equivalents (TE)/g, and 0.52 to 54.47 mg TE/g, respectively (dry weight basis; DW). Based on Folin–Ciocalteu assay, TPC ranged from 2.93 to 160.55 mg of gallic acid equivalents (GAE)/g DW. Strong correlations between TPC and TEAC values ($R = 0.966$, 0.825, and 0.954 for ABTS, DPPH, and CUPRAC, respectively) were found. This indicates

that phenolic compounds are potent antioxidants in these spices. Principal component analysis (PCA) indicated that cloves (flower buds of *Syzygium aromaticum*) had the most distinct and potent antioxidant capacity, followed by allspice (fruits of *Pimenta dioica*) and cinnamon (bark of *Cinnamomum verum*). Results of the present study provide adequate evidence that polyphenols are responsible for their compelling antioxidant capacities of studied spices. Thus, consumption of antioxidant-rich spices such as cloves, allspice, and cinnamon can significantly prevent oxidative stress in the human body.

Impact of interfacial composition on co-oxidation of lipids and proteins in oil-in-water emulsions: competitive displacement of casein by surfactants

Yi, J., *et al*., *Food Hydrocoll. 87*: 20–28, 2019, https://doi.org/10.1016/j.foodhyd.2018.07.025.

In this study, the interfacial composition of protein-stabilized emulsions was manipulated by adding a small molecule surfactant to induce displacement of the adsorbed protein. The rate and extent of lipid and protein oxidation was then measured for emulsions with different interfacial compositions. Lipid and protein co-oxidation were studied in walnut oil-in-water (O/W) emulsions (5% v/v oil, 1.0% w/v protein, 0–1.0% w/v Tween 20, pH 7). The interfacial tension, protein surface load, surface potential, and mean particle size of the emulsions decreased as the surfactant concentration increased, suggesting that caseinate was displaced by Tween 20. Emulsions stabilized solely by caseinate exhibited relatively slow lipid oxidation when incubated in the dark at 45°C for up to 8 days, as determined by lipid hydroperoxides and 2-thiobarbituric acid-reactive substances (TBARS). In contrast, the caseinate itself was rapidly oxidized, as shown by carbonyl formation, intrinsic fluorescence loss, and electrophoresis measurements. Competitive displacement of adsorbed caseinate by Tween 20 reduced protein oxidation but promoted lipid oxidation, indicating that adsorbed proteins were more sensitive to oxidation themselves but also more efficient at protecting lipids from oxidation than non-adsorbed ones. These results demonstrate the important role that interfacial composition plays on the oxidative stability of food emulsions containing mixtures of emulsifiers.

Oxidative stability of microencapsulated fish oil with rosemary, thyme, and laurel extracts: a kinetic assessment

Yeşilsu, A.F. and G. Özyurt, J. *Food Eng. 240*: 171–182, 2019, https://doi.org/10.1016/j.jfoodeng.2018.07.021.

Antioxidant activities of rosemary (R), thyme (T), and laurel (L) extracts were evaluated for protection of fish oil during microencapsulation and heat-induced degradation. For this purpose, oil stability was optimized by comparing oxidation levels of fish oils which are exposed to high temperatures, i.e., 23°C, 40°C, and 60°C in the presence of natural and commercial antioxidants, i.e., 1000 (1) 1500 (2) ppm extracts and two control groups without and with BHT (250 ppm) (C1, C2). Then, peroxide formation kinetics of the microcapsules were also determined. Peroxide values (PV) of microencapsulated fish oil in the presence of 1500 ppm rosemary (R2) were lower (3.08 mEq O_2/kg oil) than the control group with commercial antioxidant (C2, 4.25 mEq O_2/kg oil). Thiobarbituric acids (TBA) of R2, R1, and L2 (0.36 mmol MDA/kg oil, 0.56 mmol MDA/kg oil, 0.59 mmol MDA/kg oil respectively) were also lower than C2 (0.64 mmol MDA/kg oil). Besides, R2 and R1 (31.62 kJ/mol, and 30.82 kJ/mol) had higher activation energies than C2 (30.46 kJ/mol). As a result, it is revealed that rosemary and laurel extracts can successfully be applied to anchovy oil for improving the oxidative stability in microencapsulation by spray drying.

Industrial Applications

Use of sunflower seed fried oil as an ecofriendly plasticizer for starch and application of this thermoplastic starch as a filler for PLA

Volpe, V., *et al.*, Ind. Crop. Prod. *122*, 15: 545–552, 2018, https://doi.org/10.1016/j.indcrop.2018.06.014.

The high cost of the poly(lactic) acid when compared to its durable competitors is a significant drawback that inhibits its diffusion for many industrial applications. A common solution is the compounding with other polymers that preserve biodegradability, and thermoplastic starch (TPS) is a conventional choice. The innovation proposed in this work is to replace into the starch plasticization process part of the glycerol with a sunflower seed oil resulting from the frying process in a fast food. The so plasticized TPS was compared to the TPS plasticized with only glycerol and then mixed to the PLA. The replacement of a certain percentage of glycerol with fried edible sunflower oil as plasticizer for starch results in an improvement in material properties of the TPS and does not change the properties of the PLA/TPS blends. Plasticization of starch with fried oil has been proven to be the most environmentally sound solution also from a life cycle assessment.

Bio-based cationic waterborne polyurethanes dispersions prepared from different vegetable oils

Liang, H., *et al. Ind. Crop. Prod. 122*: 448–455, 2018, https://doi.org/10.1016/j.indcrop.2018.06.006.

In this study, a series of bio-based polyols were prepared from olive, castor, corn, canola, rice bran, grape seed, and linseed oil by thiol-ene photo-click reaction. The relationship between carbon-carbon double bonds in the backbone of vegetable oil fatty acid chains and the functionalities of the polyols was elucidated. The advantage and disadvantage between thiol-ene photo-click reaction and traditional methods for vegetable oil based polyols are summarized and compared. These bio-based polyols were used to prepare cationic waterborne polyurethane dispersions. With the increase of the vegetable oil based polyols' hydroxyl values, the tensile strength, Young's modulus, Tg, water contact angle of the waterborne polyurethane films increase from 1 to 11 MPa, 10 to 395 MPa, 23 to 50°C and 38 to 46°C, respectively, but the elongation at break and thermal stability of them decrease. Thiol-ene photo-click reaction offers a bio-based platform to create a variety of waterborne polyurethanes that promises economic and environmental benefits.

Medical/Pharmaceutical Applications

Tuna oil alleviates d-galactose-induced aging in mice accompanied by modulating gut microbiota and brain protein expression

Zhang, D., *et al., J. Agric. Food Chem. 66*: 5510–5520, 2018, https://doi.org/10.1021/acs.jafc.8b00446.

This research addresses a huge area of emerging research focused on the gut-brain axis. Tuna oil has been associated with healthy benefits, but identifying a scientific basis is another milestone. This research may propel similar research with natural botanical ingredients.

To discern whether tuna oil modulates the expression of brain proteins and gut microbiota structure during aging induced by d-galactose, we generated an aging mouse model with d-galactose treatment, and the mice showed aging and memory deterioration symptoms according to physiological and biochemical indices. Treatment with different doses of tuna oil alleviated the symptoms; the high dose showed a better effect. Subsequently, brain proteomic analysis showed the differentially expressed proteins were involved in damaged synaptic system repair and signal transduction system enhancement. In addition, tuna oil treatment restored the diversity of gut microbiota—27 key operational taxonomic units, which were identified using a redundancy analysis and were significantly correlated with at least one physiological index and three proteins or genes. These findings suggest that the combination of proteomics and gut microbiota is an effective strategy to gain novel insights regarding the effect of tuna oil treatment on the microbiota–gut–brain axis.

Prebiotic mannan-oligosaccharides augment the hypoglycemic effects of metformin in correlation with modulating gut microbiota

Zheng, J., *et al., J. Agric. Food Chem. 66*: 5821–5831, 2018, https://doi.org/10.1021/acs.jafc.8b00829.

A pre-biotic when used in combination with a drug augmented the drug action. More research will follow this path, helping to reduce intake of drugs for the same effects when used in combination with select food containing natural active compounds.
Type 2 diabetes (T2D) induced by obesity and high-fat diet is significantly associated with gut microbiota dysbacteriosis. Because the first line clinical medicine of metformin has several intestinal drawbacks, combination usage of metformin with a prebiotic of konjac mannan-oligosaccharides (MOS) was conceived and implemented aiming to investigate whether there were some intestinal synergetic effects and how MOS would function. Composite

Chemists find AOCS proficiency program and Approved Chemist status build customer confidence

Quantifying customer confidence can be as challenging as quantifying materials in a sample. Customers look to certifications and awards as measures of a laboratory's quality.

That was how Claire Traynor, Head of Mylnefield Lipid Analysis of James Hutton Limited, and her lab became involved with the AOCS Laboratory Proficiency Program (LPP). Traynor originally enrolled her lab in the GOED Nutraceutical Oils Series at the request of a customer. Participating in the LPP series has allowed them to build customer confidence and better understand how their results compare to their peers.

"Taking part in the program helps us to determine how our laboratory compares to other laboratories, and we were especially pleased to receive First Place in the 2016–17 LPP. This was a great boost for my analysts, especially those performing the analysis, but also confirms the quality of the work that we undertake for our customers is to a very high standard," according to Traynor.

Each year, AOCS publishes a list of LPP Award Winners as part of the Society's commitment to recognize the expertise and dedication to quality of chemists in the program. The list consists of analysts who have scored in the top 10% of each series. The recognition is important both inside and outside of the lab for Traynor and her analysts.

"Taking part in such programs is important as part of our commitment to quality, and it gives our customers extra confidence in their results."

In addition, AOCS recognizes the analytical excellence of individual chemists with Approved Chemist status. Earning Approved Chemist status allows chemists to use the Approved Chemist logo to advertise their expertise. To earn it, chemists must achieve a precise score in four consecutive quarters as an LPP participant (July 1, October 1, January 1 and April 1), return results for all samples, report results for all required constituents and be an AOCS member.

Rudy Fulawka, a Seed Chemist with Bayer CropScience, has been an AOCS Approved Chemist in gas chromatography for 17 years. He uses AOCS methods to identify and quantify tocopherols and glucosinolates in canola seeds and to determine the fatty acid profile of the oil.

According to Fulawka, he is a competitive person, but lab work doesn't offer many opportunities to compete. Earning Approved Chemist status gives him a standard to shoot for and an outlet for his competitive nature. It looks good on his curriculum vitae and to customers.

"AOCS Approved Chemist status gives clients confidence in my results."

Full-year LPP participants are eligible to apply for the Approved Chemist program. **AOCS Approved Chemists** are in high demand, and are highly respected throughout the industry. Use your status as an AOCS Approved Chemist to promote your technical expertise and attract new business – apply today!

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treatment of metformin and MOS demonstrated synergistic effects on ameliorating insulin resistance and glucose tolerance, also on repairing islet and hepatic histology. In addition, MF+MOS altered the gut community composition and structure by decreasing the relative abundances of family *Rikenellaceae* and order Clostridiales while increasing an unnamed OTU05945 of family S24–7, *Akkermansia muciniphila*, and *Bifidobacterium pseudolongum*. The present study suggested that usage of MOS could augment the hypoglycemic effects of metformin in association with gut microbiota modulation, which could provide references for further medication.

Roasted barley extract (mugi-cha) containing cyclo(d-phe-l-pro) prevents lowering of the cutaneous blood flow and skin temperature under air conditioning: a randomized, double-blind, placebo-controlled, crossover study

Ashigai, H., *et al.*, *J. Agric. Food Chem. 66*: 5901–5906, 2018, https://doi.org/10.1021/acs.jafc.8b02485.

The folks sitting in air conditioned rooms with jackets need to drink barley juice instead and enjoy summer indoor as well as outdoor.

Roasted barley extract (RBE), also known as mugi-cha, is a well-known healthy non-caffeinated beverage, and its health functionality has been widely reported. Our previous clinical study showed that RBE affects the cutaneous blood flow and skin temperature after cold-water immersion and that cyclo(d-Phe-l-Pro) is responsible for its effect. In this study, we investigated whether cyclo(d-Phe-l-Pro)-containing RBE prevents the decrease in the cutaneous blood flow and skin temperature. Subjects remained in the air-conditioned room while ingesting RBE or a placebo. We measured the cutaneous blood flow and skin temperature. We evaluated the effect of RBE administration by two-way repeated measures analysis of variance. A total of 15 subjects were enrolled. The change in cutaneous blood flow in the RBE and placebo groups was −0.79 ± 0.38 and −2.03 ± 0.35 mL min−1 100 g−1, respectively (p value of 0.041). The change in the skin temperature in the RBE and placebo groups was −1.85 ± 0.35 and −3.02 ± 0.30°C, respectively (p value of less than 0.001). We also did subclass analysis with cold-feeling subjects. For the seven subjects who had cold sensation, the change in the cutaneous blood flow in the RBE and placebo groups was −0.48 ± 0.58 and −2.56 ± 0.48 mL min−1 100 g−1, respectively (p value of 0.008). The change in the skin temperature in the RBE and placebo groups was −1.46 ± 0.74 and −2.89 ± 0.39 °C, respectively (p value of 0.009). Thus, RBE containing cyclo(d-Phe-l-Pro) prevents the decrease in the cutaneous blood flow and skin temperature under air conditioning.

Chicory roots for prebiotics and appetite regulation: a pilot study in mice

Fouré, M., *et al.*, *J. Agric. Food Chem. 66*: 6439–6449, 2018, https://doi.org/10.1021/acs.jafc.8b01055.

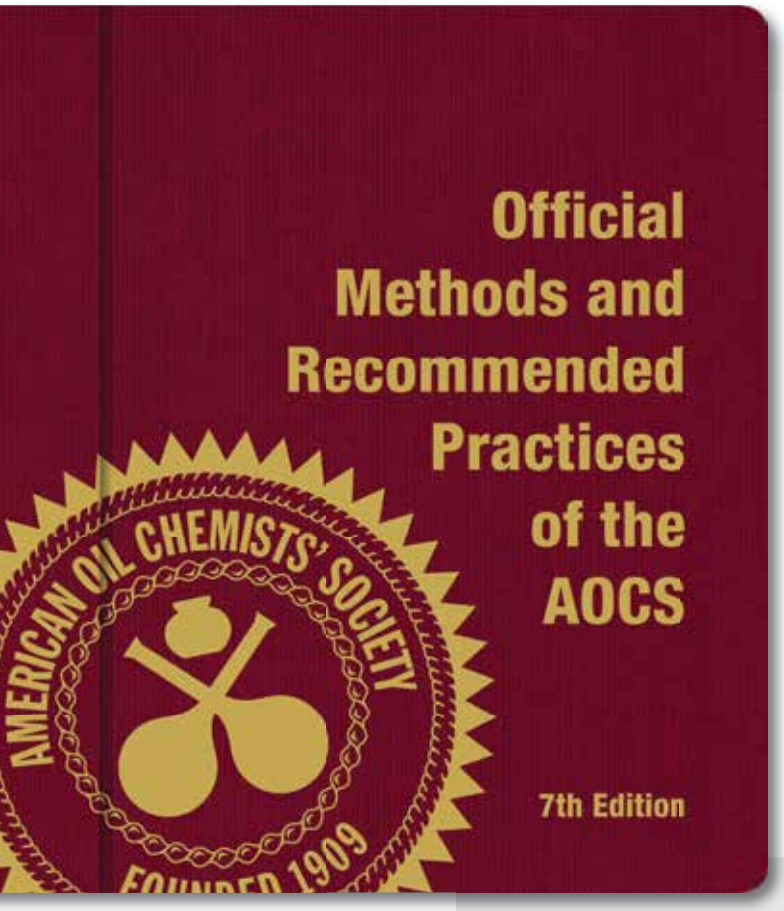
The objectives of this work are to address the prebiotic effects of chicory (*Cichorium intybus*) together with its possible role in appetite control. We compared nine chicory genotypes in order to determine if variations in the content of metabolites in the roasted roots would lead to modifications in release of satiety hormones and in composition of gut microbiota. To this aim, a 5-week dietary-intervention study was achieved using mice fed with distinct chicory-based preparations. A 16S rRNA gene-based metagenetic analysis of fecal microbiota was performed. In vitro gastrointestinal digestions were performed in order to study the effect of chicory intestinal digests on gut hormone regulation in enteroendocrine cells. Firmicutes/Bacteroidetes ratio and gut bacterial groups, such as *Alloprevotella*, *Blautia*, *Alistipes*, and *Oscillibacter*, were found to be modulated by chicory. On the other hand, CCK and GLP-1 satiety hormones were demonstrated to be significantly increased by chicory *in vitro*.

Phenolic constituents isolated from the twigs of *Cinnamomum cassia* and their potential neuroprotective effects

Liu, X., *et al.*, *J. Nat. Prod. 81*: 1333–1342, 2018, https://doi.org/10.1021/acs.jnatprod.7b00924.

Traditional use of spices and condiments have long been suspected to impart cognitive and neuroprotective benefits. The identification of neuroprotective agents in cinnamon support the use of natural and traditional herbs and confirm some traditional beliefs about the benefit of naturals.

Seven new alpha, beta-diphenyl-gamma-butyrolactones (1–7), three new lignans (8–10), five new neolignans (11–15), two new 1,3-biphenylpropanoids (16 and 17), and a new flavonol galactoside-lignan ester (18), together with 43 known compounds (19–61), were isolated from the twigs of *Cinnamomum cassia*. Their structures were elucidated by spectroscopic data analysis as well as chemical methods. The alpha, beta-diphenyl-gamma-butyrolactones are a class of unique natural compounds that have only been isolated from *C. cassia*. Compounds 11 and 12 are rare examples of neolignans possessing a 1,2-dioxetane moiety. Compound 13 is a new oxyneolignan possessing a unique C-9–O–C-9′ linkage between the benzopyran and cinnamyl alcohol moieties. Compound 15 is the first example of a natural neolignan possessing a 2-styryl-3-phenyltetrahydrofuran skeleton. The isolated compounds were evaluated for their neuroprotective activities against tunicamycin-induced cytotoxicity in SH-SY5Y cells. Compounds 3, 5, 10, 11, 12, 20, 36, and 56 showed statistically significant neuroprotective activity with EC50 values ranging between 21 and 75 micrometers.



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Fabrication of stable zein nanoparticles coated with soluble soybean polysaccharide for encapsulation of quercetin

Li, H., *et al.*, *Food Hydrocoll.* *87*: 342–351, 2019, https://doi.org/10.1016/j.foodhyd.2018.08.002.

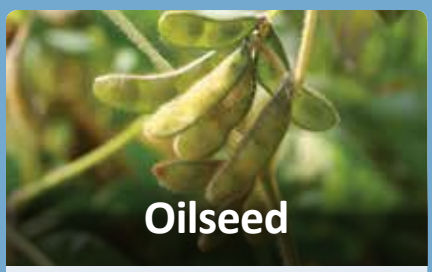
The present work aims to prepare and stabilize zein colloidal nanoparticles using soluble soybean polysaccharide (SSPS) as a stabilizer. A simple antisolvent precipitation method was used to fabricate the zein/SSPS composite nanoparticles at pH 4.0. The results showed that the SSPS-stabilized zein nanoparticles (size around 200 nm, PDI below 0.2) did not aggregate or precipitate at pH 2.0–8.0. And they were also relatively stable for elevated ionic strength and high temperature. Affected by the SSPS coating, the surface hydrophobicity of the zein nanoparticles decreased over a wide pH range. Conditions of forming zein/SSPS nanoparticles were successfully used to encapsulate hydrophobic quercetin (Q). The encapsulation efficiency was greatly improved to 82.5% after SSPS coating, compared to 59.2% for the zein without coating. Besides, the photochemical stability and $ABTS^{+}$ scavenging ability of Q in zein/SSPS composite nanoparticles were significantly enhanced. This study demonstrates that these composite nanoparticles can be used as all-natural delivery systems for bioactive molecules in food and pharmaceutical formulations.

Roles of spicy foods and their bioactive compounds in management of hypercholesterolemia

Zhao, Y. and Z,-Y. Chen, J. Agric. Food Chem. 66: 8662–8671, https://doi.org/10.1021/acs.jafc.8b02975.

Hypercholesterolemia, as one of the major risk factors in development of cardiovascular diseases, is of mounting prevalence worldwide in recent years. Many nutraceuticals and phytochemical supplements serve as a promising complementary therapy in the management of hypercholesterolemia. Among them, spicy foods have attracted special attention. Plasma lipid-lowering activity of garlic, ginger, and turmeric have been well-studied in both humans and animals. Consumption of either 3 g/day of ginger or 2 g/day of curcumin for over 4 weeks effectively reduced blood cholesterol in hypercholesterolemia subjects. However, effects of chili and black peppers on blood cholesterol are studied little clinically. The present review is to summarize the findings of recent studies on the efficacy and mechanism of spicy foods and their primary bioactive components in management of hypercholesterolemia from preclinical studies to clinical trials.

Worldwide production of oilseeds, vegetable oils, and protein meals MMT*



	2016/17	2017/18	2018/19
Copra	5.51	5.73	5.83
Cottonseed	39.1	44.7	44.37
Palm kernel	17.4	18.6	19.21
Peanut	44.9	45.6	44.58
Rapeseeed	71.3	74.3	72.15
Soybean	350.8	336.7	367.1
Sunflower	48	47.3	49.88
Total	**577**	**572.7**	**603.12**

	2016/17	2017/18	2018/19
Coconut	3.4	3.5	3.6
Cottonseed	4.4	5.1	5.2
Olive	2.5	3.3	3.2
Palm	65.3	69.7	72.6
Palm kernel	7.6	8.2	8.4
Peanut	5.9	6	6.1
Rapeseed	28.2	28.8	29.6
Soybean	53.8	55.8	58.4
Sunflower	18.2	18.4	19.3
Total	**189.3**	**198.7**	**206.3**

	2016/17	2017/18	2018/19
Copra	1.8	1.9	1.93
Cottonseed	13.4	15.5	15.84
Fish	4.9	4.6	4.71
Palm kernel	9.9	9.6	9.92
Peanut	7.3	7.4	7.48
Rapeseed	39.9	40.6	40.34
Soybean	225.9	235.7	242.71
Sunflower	19.3	19.7	20.51
Total	**321.5**	**335.1**	**343.43**

*MMT, million metric tons.

Source of Data: USDA/FAS Market Report August 2018.

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