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# INFORM

# TOXIC ALDEHYDES

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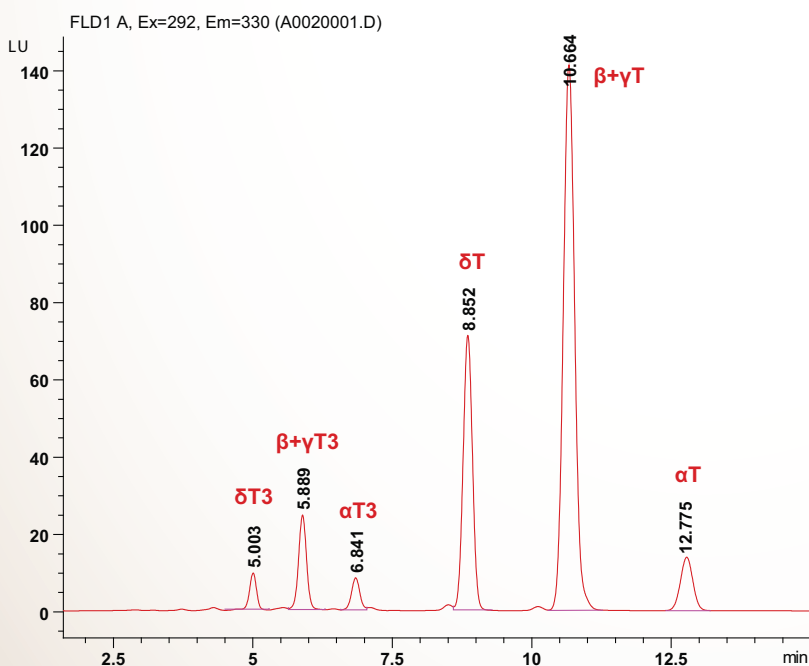
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Method: TOCOLS - HPLC Sample ID: PALM TOCOL COMPLEX

RT	Response	ECL	Peak Name	Percent	mg/100g
5.003	9732	5.607	DELTA TOCOTRIENOL	3.62	3123
5.889	24707	6.639	BETA + GAMMA TOCOTRIENOL	9.18	8124
6.841	8443	7.761	ALPHA TOCOTRIENOL	3.14	2779
8.852	71163	10.240	DELTA TOCOPHEROL	26.45	23406
10.664	141152	12.274	BETA + GAMMA TOCOPHEROL	52.46	46423
12.775	13850	14.892	ALPHA TOCOPHEROL	5.15	4557

Total Response: 269048 Percent Named: 100.00%



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# CONTENTS

## 614 **Detection, monitoring, and deleterious health effects of lipid oxidation products generated in culinary oils during thermal stressing episodes**

Trans fatty acids have received considerable attention in the media and scientific literature, but did you know that ordinary culinary oils, when heated, produce aldehydes that may be more dangerous than trans? Learn how such aldehydic fragments react *in vivo* with critical biomolecules, such as low-density lipoprotein and deoxyribonucleic acid (DNA).

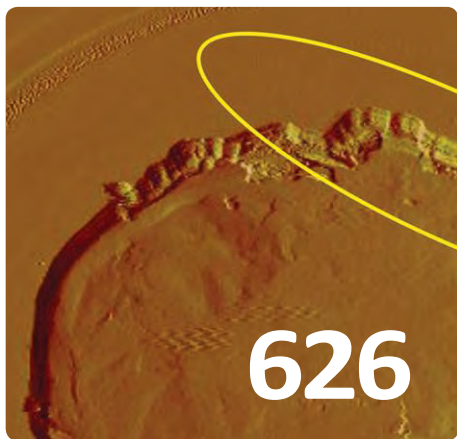
## 626 **Developing a high-performance, low-streak degreaser**

Commercial multipurpose cleaners do not offer a strong combination of heavy duty grease removal and low streaking. This article describes the development of a surfactant-based cleaner that does.

## 638 **What's the automotive aftermarket industry hiring surfactants to do?**

Learn how and why surfactants are used in such automotive applications as glass treatments, wash and wax, tire and wheel care, and under-the-hood additives for engines.





**646**

### **Lowering the boiling curve of biodiesel by metathesis**

Scientists describe how they used metathesis to lower the boiling curve of biodiesel and conducted emissions tests of the resulting fuels in three different diesel engines.

**652**

### **Approaches to mitigating esters of both 3-MCPD and glycidol**

This article describes several strategies for mitigation—in raw materials, during refining, and in fully refined oils.

**656**

### **Automatic laundry washing machine changes and impact on detergent formulation chemistry**

Learn why the reduced water volumes used in newer washing machines present new opportunities and challenges for laundry formulation chemists.

**662**

### **How optimizing your tank and tote cleaning process can improve your bottom line**

When cleaning tanks and other containers, it is important to examine the effectiveness and efficiency of the procedure. Substantial cost reductions and revenue recovery can often be made by applying the principles of cleaning and optimizing the cleaning-in-place (CIP) process.

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## **DEPARTMENTS**

613 Index to Advertisers  
669 Classified Advertising

**MARKETPLACE**  
660 AOCS Meeting Watch  
670 Professional Pathways

**PUBLICATIONS**  
666 Patents  
668 Extracts & Distillates

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# INDEX TO ADVERTISERS

Agribusiness & Water Technology, Inc. ....	632
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*Desmet Ballestra Engineering NA .....	C2
*Desmet Ballestra S.p.A. ....	625
GEA Westfalia Separator Group .....	621
Harburg-Freudenberger Maschinenbau GmbH .....	C4
Mahle Industry GmbH .....	643
MIDI.....	609
Myers Vacuum Distillation Division .....	642
*Oil-Dri Corporation of America .....	650
Sharplex Filters (India) PVT. LTD. ....	623



# Detection, monitoring, and deleterious health effects of lipid oxidation products generated in culinary oils during thermal stressing episodes

Martin Grootveld, Victor Ruiz Rodado, and Christopher J.L. Silwood

$^1\text{H}$ , two-dimensional  $^1\text{H}$ - $^1\text{H}$ , and  $^1\text{H}$ - $^{13}\text{C}$  NMR investigations of thermally stressed PUFA-containing culinary oils have found high levels of  $\alpha,\beta$ -unsaturated aldehydes (including *trans*-2-alkenals, and *cis,trans*- and *trans,trans*-alka-2,4-dienals, the latter including the mutagen *trans,trans*-2,4-decadienal), and *n*-alkanals, together with their CHPD and hydroxydiene precursors (Fig. 2, page 618) (1–3). Indeed, samples of repeatedly used oils collected from fast-food retail outlets and restaurants have confirmed the production of aldehydic lipid oxidation products (LOPs) at levels exceeding  $10^{-2}$  moles per kilogram ( $\text{mol}\cdot\text{kg}^{-1}$ ) during “on-site” frying episodes.

As expected, the levels of total aldehydes generated increase proportionately with oil PUFA content, and over half are the more highly cytotoxic  $\alpha,\beta$ -unsaturated classes (Fig. 3a, page 620; Table 1, page 617), which include acrolein and 4-hydroxy-*trans*-2-nonenal (HNE), as well as 4-hydroperoxy-, 4-hydroxy-, and 4,5-epoxy-*trans*-2-alkenals. Total  $\alpha,\beta$ -unsaturated aldehyde concentrations in culinary oils

(heated at  $180^\circ\text{C}$  for 30–90 minutes or longer) are often higher than  $20\text{ mmol}\cdot\text{kg}^{-1}$  and can sometimes approach  $50\text{ mmol}\cdot\text{kg}^{-1}$ . Furthermore, relatively low concentrations of  $^1\text{H}$  NMR-detectable aldehydes and their CHPD precursors are even found in newly purchased unheated culinary oils. Key resonances for CHPD isomers are also readily detectable within the 5.5–6.7 ppm (parts per million) regions of high-resolution  $^1\text{H}$  NMR spectra. We can also employ these NMR techniques to monitor the corresponding degradation of culinary oil PUFAs and monounsaturated fatty acids (MUFAs) during heating/frying (Fig. 2b, 619).

Portable bench-top NMR instruments clearly have potential for monitoring PUFA peroxidation and LOP generation in cooking oils at industrial sites or in restaurants.

Acrylamide (which can exert toxic effects on the nervous system and fertility, and may also be carcinogenic) can also arise from an acrolein source when asparagine-rich foods are deep-fried in PUFA-rich oils (4). The levels of acrylamide generated in foods during high-temperature cooking/frying processes are substantially lower than those recorded for aldehydes formed in PUFA-rich culinary oils during frying episodes (to date, the very highest reported levels are only *ca.* 4 ppm, equivalent to  $56\text{ }\mu\text{mol}\cdot\text{kg}^{-1}$ ). Should we not also have major concerns about the much higher concentrations of cytotoxic LOPs

CONTINUED ON PAGE 617





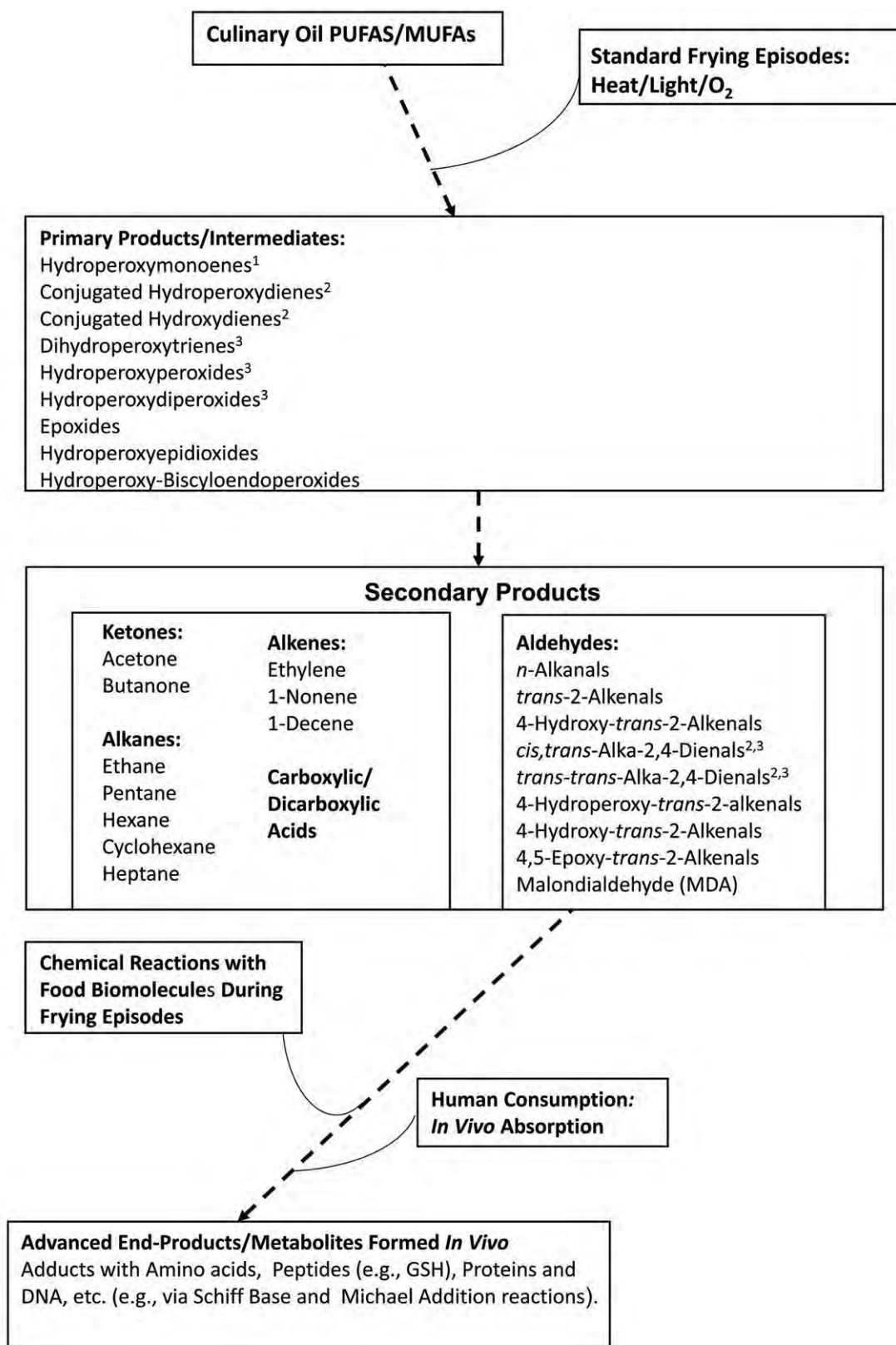


- Trans fatty acids have received considerable attention in the media and the scientific literature (together with their putative atherogenic and carcinogenic actions). The far more toxic aldehydes generated in thermally stressed culinary oils, however, have received little or none.

- The most important chemical reaction involved is the self-sustaining, free radical-mediated oxidative deterioration of polyunsaturated fatty acids (PUFAs), which occurs during the heating of culinary oils (Fig. 1, page 616) and, to a much diminished level, saturated fatty acid (SFA)-rich fats. This oxidative degradation process can generate extremely toxic conjugat-

ed lipid hydroperoxydienes (CHPDs). These are unstable at standard frying temperatures (*ca.* 180°C) and are degraded to a broad range of secondary products, particularly saturated and unsaturated aldehydes, together with di- and epoxyaldehydes.

- Such aldehydic fragments also have toxicological properties in humans owing to their high reactivity with critical biomolecules *in vivo* (proteins such as low-density lipoprotein, amino acids, thiols such as glutathione, DNA, etc.). Despite their reactivities, high levels of CHPDs can remain in PUFA-rich oils which have been subjected to routine frying practices.



**FIG. 1.** Schematic representation of the peroxidation of polyunsaturated fatty acids (PUFAs; i.e., linoleoyl- and linolenoylglycerols) and, to a much lesser extent, monounsaturated fatty acids (MUFAs; i.e., oleoylglycerols) occurring during standard frying practices; primary and secondary lipid oxidation products (LOPs) arising from this process, together with advanced end-products, are indicated. Also shown is the fate of aldehydic LOPs, specifically the generation of advanced end-products from their possible reactions with biomolecules in foods during frying processes, and also those potentially arising in humans following *in vivo* absorption of these aldehydes from the gut into the systemic circulation subsequent to dietary consumption. These advanced end-products include mammalian metabolites, such as mercapturate conjugates derived from the metabolic transformation of *trans*-2-alkenals (see ref. 5). Primary and secondary products arising from <sup>1</sup>monoene, <sup>2</sup>diene and <sup>3</sup>triene lipids are designated. Abbreviation: GSH, glutathione.

available for consumption by humans (see sidebar on toxicological actions)?

The concentrations of aldehydes generated in culinary oils during episodes of heating at 180°C represent only what remains in the oil: Owing to their low boiling points, many of the aldehydes generated are volatilized at standard frying temperatures. These represent inhalation health hazards, in view of their inhalation by humans, especially workers in inadequately ventilated fast-food retail outlets.

Figure 3b (page 620) shows a colorimetric (TBARS test) illustration of aldehyde production in a sample of corn oil subjected to heating for 60 minutes.

IN VIVO ABSORPTION OF DIETARY LOPs

Except for direct damage to the gastrointestinal epithelium, the toxicological actions exerted by LOPs depend on their rate and extent of absorption from the gut into the systemic circulation where they may cause damage to essential organs, tissues, and cells. Experiments in rats have demonstrated that *trans*-2-alkenals, which are generated in PUFA-containing culinary oils during thermal stressing episodes, are absorbed (5). Following absorption, these cytotoxic agents are metabolized by a process involving the primary addition (Michael addition reaction) of glutathione across their electrophilic carbon-carbon double bonds and finally excreted in the urine as C-3 mercapturate derivatives.

ESTIMATING HUMAN DIETARY INTAKE OF LOPs

Estimates of dietary LOP intake should be ideally (but not exclusively) targeted at foods subjected to high-temperature frying/cooking. Indeed, the composition and content of hazardous LOPs available in fried foods depend on the identity of the frying/cooking oil and its PUFA content, the frying conditions employed, the length of the frying process, exposure of the frying medium to atmospheric O<sub>2</sub>, the reactivities of these agents with a range of other biomolecules (e.g., amino acids and proteins), and, to a limited extent, the antioxidant content of the frying matrix. Our experiments have shown that shallow frying gives rise to much higher levels of LOPs than deep frying under the same conditions

GLOSSARY

- CHPD conjugated lipid hydroperoxydienes
- HNE 4-hydroxy-*trans*-2-nonenal
- LDL low density lipoprotein
- LOP lipid oxidation product
- MDA malondialdehyde
- MUFA monounsaturated fatty acid
- NIOSH US National Institute for Occupational Safety and Health
- NMR nuclear magnetic resonance
- OSHA US Occupational Safety and Health Administration
- PEL permissible exposure level
- PUFA polyunsaturated fatty acid
- SFA saturated fatty acid
- TBARS thiobarbituric acid reactive-substances
- TOH α-tocopherol

(reflecting the influence of the surface area of the frying medium, its exposure to atmospheric O<sub>2</sub>, and the subsequent dilution of LOPs generated into the bulk medium).

Although the lipid (predominantly triacylglycerol, or TAG) content of deep-fried French fries is dependent on the nature of the frying process, its length, and temperature, these values generally range from 7% to 35% (w/w), of which SFAs are 28–46%, MUFAs 25–43%, and PUFAs 2–45%. On this basis, a 300-g serving of French fries (chips) with a fat content of 11.5% (w/w) contains 35 g of LOP-containing lipids per serving. If we estimate that the total level of aldehydic LOPs present in a typical thermally stressed PUFA-rich culinary oil is 10 millimoles per kilogram (mmol·kg<sup>-1</sup>) and assume they are not significantly consumed by reaction with food biomolecules, the aldehyde content of this serving of French fries will be *ca.* 0.35 mmol, of which over half are the extremely toxic α,β-unsaturated ones (Fig. 3a, page 620).

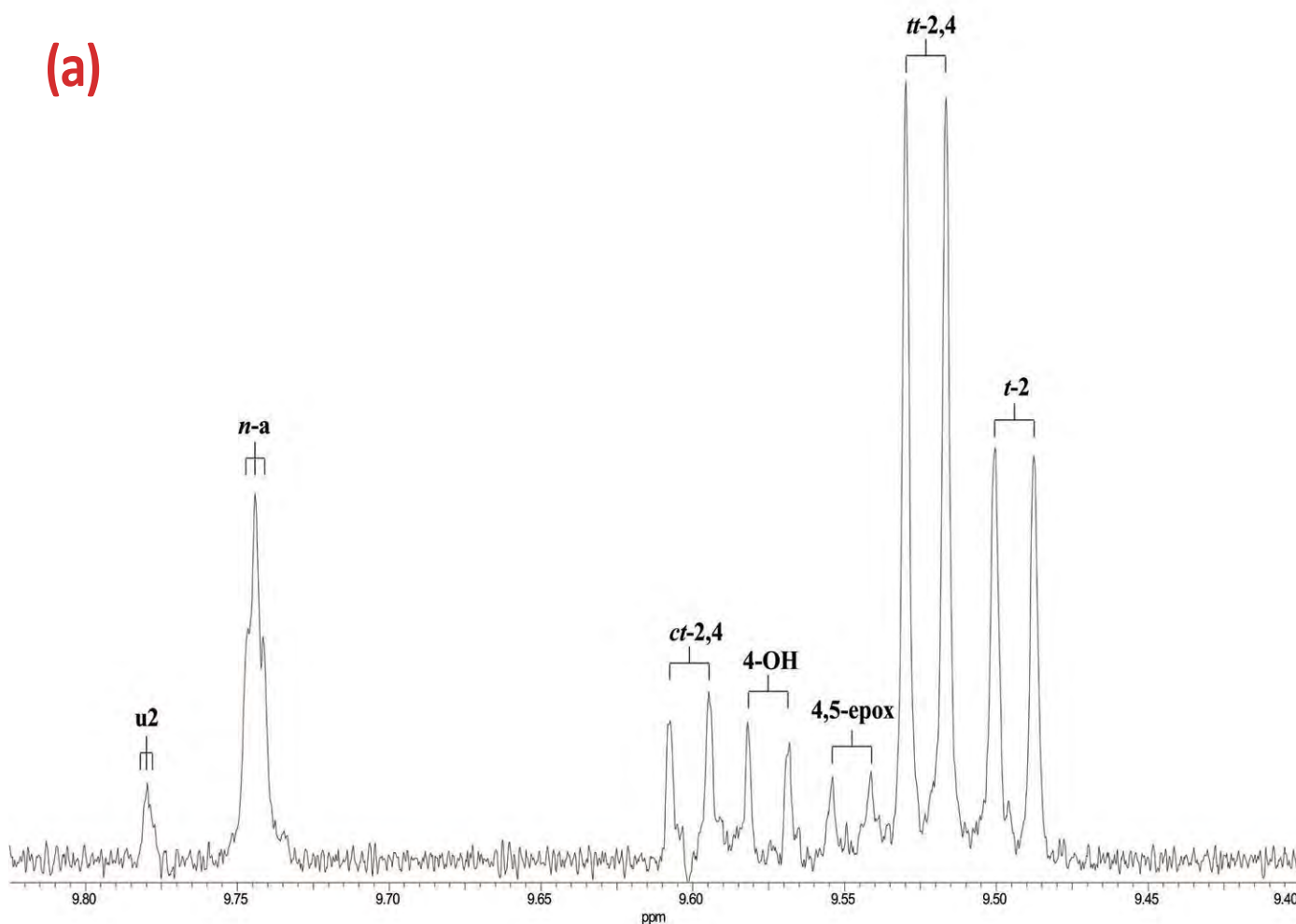
The New Zealand Heart Foundation estimated that a meal consisting of two pieces of battered fish plus an average serving of fries represents 95 g of fat. With an assumption

CONTINUED ON NEXT PAGE

TABLE. Aldehyde levels detectable in a sample of sunflower oil subjected to thermal stressing at a standard frying temperature of 180°C in the presence of atmospheric O<sub>2</sub> (0-90 minutes). Typical results are shown. Abbreviations: nd, none detectable.

Length of Thermal Stressing Episode	<i>trans</i> -2-Alkenals (mmol.kg <sup>-1</sup> )	<i>trans,trans</i> -Alka-2,4-Dienals (mmol.kg <sup>-1</sup> )	<i>cis,trans</i> -Alka-2,4-Dienals (mmol.kg <sup>-1</sup> )	4-Hydroxy- <i>trans</i> -2-Alkenals (mmol.kg <sup>-1</sup> )	<i>n</i> -Alkanals (mmol.kg <sup>-1</sup> )	Further Unassigned Aldehydes (total: mmol.kg <sup>-1</sup> )
Unheated (0 min.)	nd	nd	nd	nd	nd	nd
30 min.	3.90	6.42	1.12	nd	2.91	0.05
60 min.	7.16	10.63	1.79	nd	5.22	0.03
90 min.	10.56	13.77	2.71	0.87	6.62	0.03





**FIG. 2. ABOVE:** (a) Aldehydic-CHO (9.4–9.9 ppm) region of the 600 MHz  $^1\text{H}$  NMR spectrum of a typical thermally stressed PUFA-rich culinary oil (corn oil heated at a temperature of  $180^\circ\text{C}$  for a 60 minute period). This typical spectrum was acquired on a 3 $\times$  dilution of the sampled oil in deuteriochloroform ( $\text{C}^2\text{HCl}_3$ ) at an operating frequency of 600.13 MHz. Abbreviations: *t*-2, *trans*-2-alkenals; *ct*- and *tt*-2,4, *cis,trans*- and *trans,trans*-alka-2,4-dienals respectively; 4-OH, 4-hydroxy-*trans*-2-alkenals; 4,5-epox, 4,5-epoxyaldehydes; *n*-a, *n*-alkanals; *u*2, unknown saturated aldehyde resonance (triplet) tentatively assignable to a core aldehyde. **AT RIGHT:** (b) 60 MHz  $^1\text{H}$  NMR spectrum of a sample of sunflower oil acquired prior and subsequent to its employment for the frying of French fries in a restaurant, and showing clear decreases in the intensity of the *bis*-allylic- $\text{CH}_2$  function resonance of PUFAs ( $-\text{CH}=\text{CH}-\text{CH}_2-\text{CH}=\text{CH}-$ ) therein ( $\delta = 2.76$  ppm), which reflects their peroxidation. (c) As for (b), but for a sample of olive oil heated at a temperature of  $180^\circ\text{C}$  for 8 hours and showing clear aldehydic-CHO proton signals located at  $\delta = 9.48$ , 9.53, and 9.75 ppm (assignable to *trans*-2-alkenals, *trans,trans*-alka-2,4-dienals, and *n*-alkanals respectively). Spectra shown in (b) and (c) were acquired on an Oxford Instruments Pulsar<sup>TM</sup> portable bench top NMR instrument incorporating a 1.4T rare earth permanent magnet. For this analysis, oil samples were diluted 7:3 (v/v) with  $\text{C}^2\text{HCl}_3$ , and spectral acquisition parameters were: a filter file of 4,000 Hz; 30 scans with a repetition delay of 15 seconds; and 32 K receiver data-points.

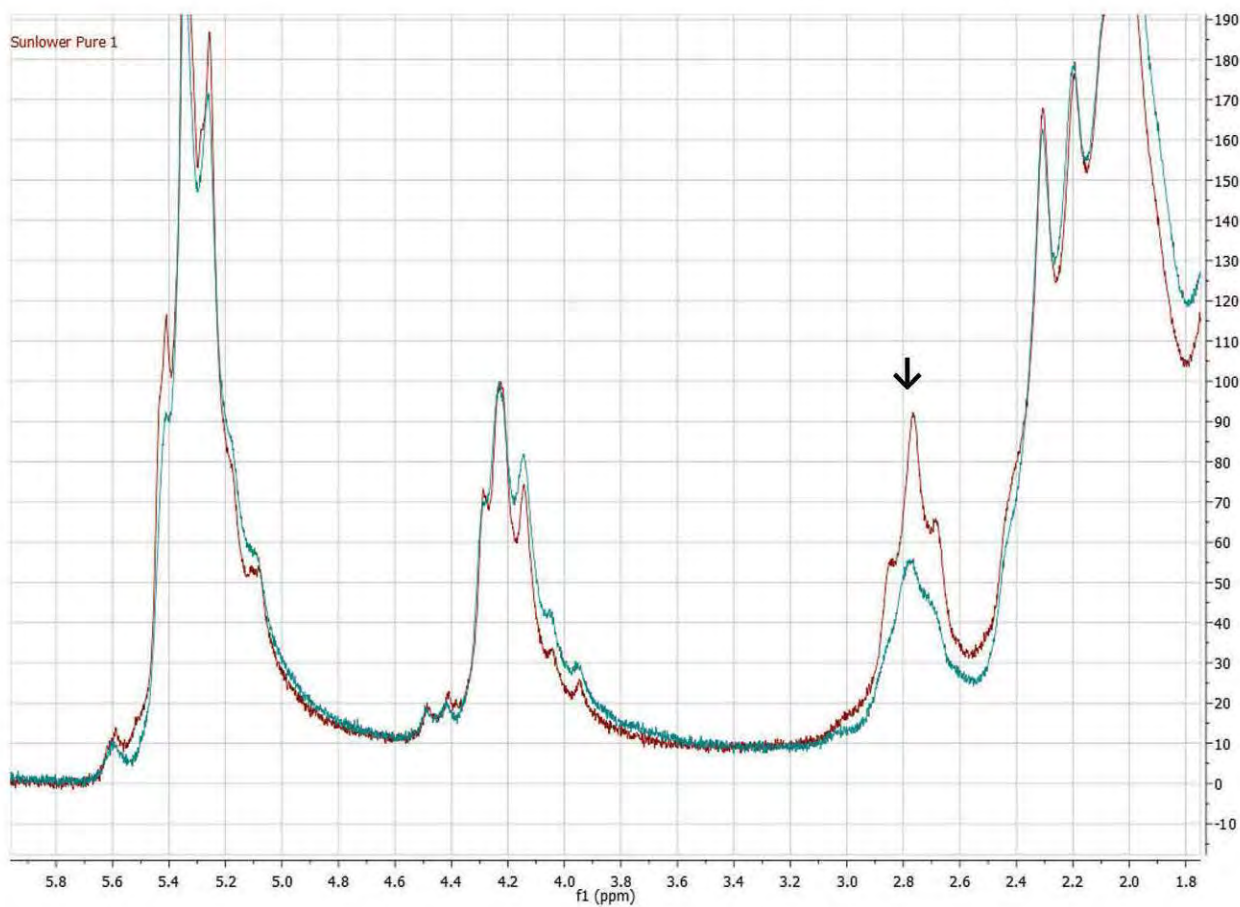
of only  $10 \text{ mmol}\cdot\text{kg}^{-1}$  total aldehydes in thermally stressed culinary oils used to fry such foods, there would be 0.95 mmol of such LOPs in an average fried meal—a not insignificant figure in view of the potent toxicological properties of such agents.

Acrolein represents the simplest and perhaps one of the more toxic  $\alpha,\beta$ -unsaturated aldehydes generated from the lipid peroxidation process, and the WHO's 'tolerable' intake level of this agent is only  $7.5 \mu\text{g}$  ( $0.13 \mu\text{mol}$ ) per day per kg of body weight, and therefore this would be equivalent to  $525 \mu\text{g}$  ( $9.4 \mu\text{mol}$ ) per day for an assumed (average)

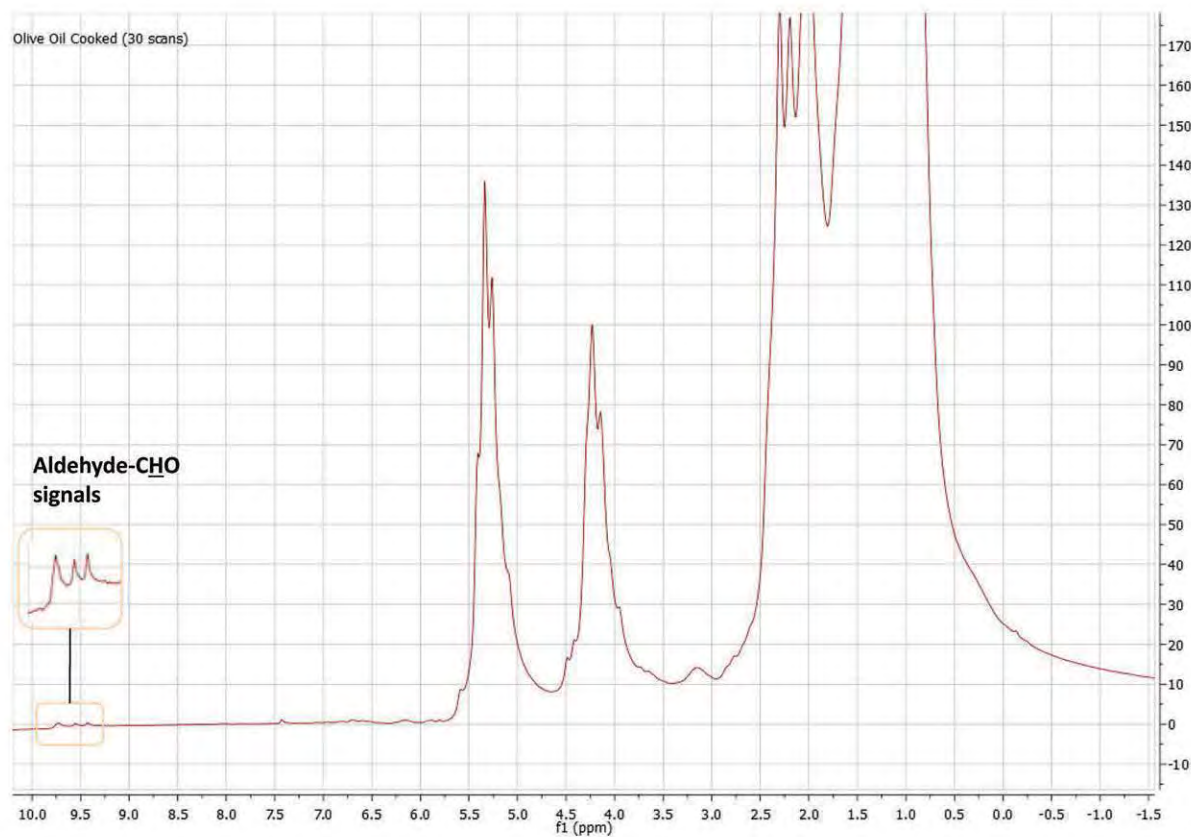
human body weight of 70 kg, a value which is substantially less than that estimated above for total aldehydes in typical fried food portions. If all of the above estimated 0.35 and 0.95 mmol of total food portion-associated aldehyde was acrolein, it would be equivalent to a content of 430 ppm in the food matrix to be consumed.

Even if almost 50% or so of these aldehydes were the less chemically-reactive and toxic saturated ones, this is still very worrying. We should expect at least some reaction of

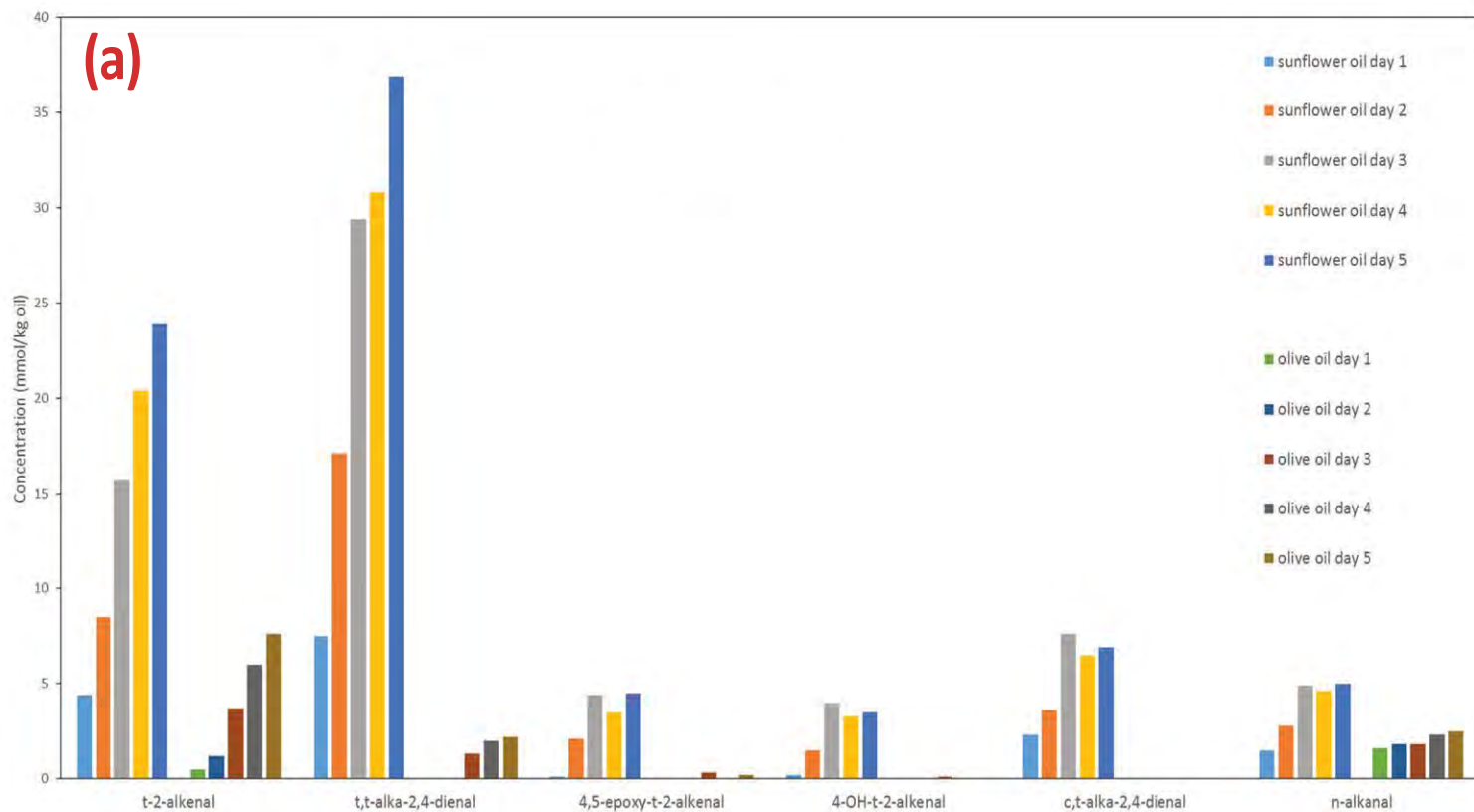
CONTINUED ON PAGE 621



(b)



(c)



(b)



**FIG. 3.** (a) Aldehyde concentrations generated in sunflower and refined olive oils during 6-hour daily repeated thermal stressing episodes performed throughout a total 5-day period. Samples of oil (400 mL) were heated at 180°C in the presence of atmospheric O<sub>2</sub> in a 1.0 L glass beaker (internal surface area 25 cm<sup>2</sup>) on a thermostatically controlled electronic hotplate. The much lower aldehyde concentrations (especially those of alka-2,4-dienals) produced in the olive oil sample reflect the much smaller PUFA content of this material. (b) Simple colorimetric demonstration [via the thiobarbituric acid-reactive substances (TBARS) test] of aldehyde generation in a commercial corn oil sample that was thermally-stressed at a temperature of 180°C for 60 minutes. TBA was added to the oil at a concentration of 1 mg mL<sup>-1</sup>, which was then either heated or unheated in this manner. The bright red aldehyde-dependent coloration was observed only in the thermally-stressed sample.



these aldehydes with food biomolecules (amino acids, etc.) during the frying process (and presumably much less subsequently at ambient or lower temperatures). Nevertheless, culinary oil aldehyde and further LOP concentrations—which we monitor by high-resolution NMR analysis—are only those that **remain** in the oil matrix after frying/heating episodes.

Furthermore, with regard to the risk of inhalation of these aldehydes volatilised during frying practices by humans, we should consider the maximum US Occupational Safety and Health (OSHA) permissible exposure limit (PEL) for acrolein, which is an (atmospheric) level of 0.1 ppm (equivalent to only  $1.8 \mu\text{mol}\cdot\text{kg}^{-1}$  in our fried food model) for a time-weighted long-term (8 hour) exposure, and 0.3 ppm ( $5.4 \mu\text{mol}\cdot\text{kg}^{-1}$ ) for a short-term (15 minute) one. This 15-minute exposure time can be considered to be less than the time taken to consume a typical fried meal!

Hence, we believe that many researchers and food scientists involved in this area should re-think their recommendations of the “health-promoting” properties of linoleoyl- and linolenoylglycerols in shallow and deep frying processes.

Acrolein is just one of the  $\alpha,\beta$ -unsaturated aldehydes generated in thermally stressed PUFA-rich oils: Many others generated in this manner have comparable toxicological properties. The foregoing considerations exclude possible toxicological properties of their isomeric CHPD precursors (also present in the high millimolar range in thermally stressed oils) in a typical fried food meal. Indeed, in one early investigation, a single intravenous dose of methyl linoleate hydroperoxide ( $20 \text{ mg}\cdot\text{kg}^{-1}$ ) administered to rats gave rise to a high mortality within 24 hours (animals dying from lung damage), although a higher dose given orally was without effect. This observation may reflect the limited *in vivo* absorption of these particular aldehyde precursors, in contrast to the known absorption of aldehydes.

## TOXICOLOGICAL AND PATHOGENIC PROPERTIES OF DIETARY LOPs

*Potential influence of dietary LOPs on metabolic pathways.* As a consequence of their absorption from the gut into the systemic circulation, LOPs may penetrate cellular membranes, allowing their entry into particular intracellular sites/organelles where many critical metabolic processes occur. Literature evidence indicates that feeding thermally stressed or repeatedly used culinary oils to experimental animals induces significant modifications to key liver microsomal pathways and to the mitochondrial respiratory chain, for example. These effects are likely to occur via reactions of LOPs with key enzymes (and more especially their active sites), for

CONTINUED ON NEXT PAGE

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## TOXICOLOGICAL ACTIONS OF ALDEHYDES (AND OTHER LIPID OXIDATION PRODUCTS)

- Atherogenicity
- Mutagenicity and carcinogenicity
- Congenital malformations (causal)
- Gastropathic actions
- Pro-inflammatory effects
- Teratogenicity
- Hypertensivity

example, the oxidation of active methioninyl and cysteinyl residues by CHPDs, or alteration of critical side-chain amino acid amine or thiol groups with aldehydes via Schiff base or Michael addition reactions.

**Atherosclerosis.** Investigations have revealed that dietary derived LOPs can accelerate all three stages of the development of atherosclerosis (i.e., endothelial injury, accumulation of plaque, and thrombosis). Animal studies have shown that diets containing thermally stressed, PUFA-laden (and hence LOP-rich) oils exhibit a greater atherogenicity than those containing unheated ones (6). Because cytotoxic aldehydes can be absorbed, they have the capacity to attack and structurally alter the apolipoprotein B component of low density lipoproteins (LDLs) (5). This mechanism can engender uptake of lipid-loaded LDLs by macrophages, which, in turn, transforms them to foam cells, the accumulation of which is responsible for the development of aortic fatty streaks, a hallmark of the aetiology of atherosclerosis and its pathological sequelae. More recently, our co-investigators found that aldehydic LOPs elevated the expression of the CD36 scavenger receptor of macrophages, a phenomenon that also promotes this process (7).

**Mutagenic and carcinogenic properties.** Since they are powerful electrophilic alkylating agents,  $\alpha,\beta$ -unsaturated aldehydes can covalently modify DNA base units via a mechanistically complex process that may involve their prior epoxidation *in vivo*. Such chemically altered bases may therefore be of mutagenic potential. Additionally, these LOPs can inactivate DNA replicating systems, a process that can, at least in principle, elevate the extent of DNA damage. Hence, following cellular uptake, such aldehydes have the potential to cause both DNA and chromosomal damage.

An Intelligence Bulletin published by the US Center for Disease Control and Prevention's National Institute for Occupational Safety and Health (NIOSH, 1992) concluded that malondialdehyde (MDA) and other aldehydes arising from lipid peroxidation (especially acrolein) present a serious carcinogenic hazard. Indeed, adenomas and carcinomas of the thyroid gland, together with adenomas of the pancreatic islet cells, were induced in rats by MDA in a prolonged gavage study; nasal and laryngeal cancers arose in rats and hamsters, respectively, during long-term acetaldehyde inhalation experiments. Hence, both these aldehydes satisfied the

NIOSH criteria for classification as carcinogens, and therefore it has set exacting limits for their occupational exposure.

We have previously shown that MDA is also generated by thermally stressing culinary oils, although at concentrations much lower than those of the more reactive  $\alpha,\beta$ -unsaturated aldehydes. Paradoxically, in view of its somewhat exaggerated importance as an aldehydic LOP, much of the occupational exposure to MDA appears to arise from its use in research laboratories investigating lipid peroxidation processes.

Volatile emissions from heated culinary oils used in Chinese-style cooking are indeed mutagenic; exposure to such indoor air pollution may render humans more susceptible to contracting lung or further cancers, together with rhinitis and diminished lung function (8–10). The high temperatures used in standard (especially Chinese) frying result in fumes that are rich in volatile LOPs, including acrolein.

An entire book (Chadwick, D. J., J. Goode (Novartis Foundation), *Acetaldehyde-related pathology: Bridging the transdisciplinary divide*, ISBN 9780470057667) together with numerous governmental health and safety documents, has been dedicated to the pathology (most especially carcinogenicity) of the *n*-alkanal acetaldehyde (with a corresponding OSHA PEL of 200 ppm), which is much less hazardous than equivalent levels of  $\alpha,\beta$ -unsaturated aldehydes such as acrolein (OSHA PEL 0.1 ppm).

Further investigations have focused on the possible association of prostate cancer with the consumption of fried foods (11) and the clastogenic potential of thermally stressed culinary oils (12). It is also conceivable that congenital malformations can arise from the reactions of DNA base adducts with aldehydes. Alternatively, DNA-polymerase and -repair enzymes can be covalently modified by these reactive aldehydes, processes giving rise to modifications in the rate and extent of DNA replication and repair, and hence chromosomal damage.

**Teratogenic actions.** In principle, if aldehydic LOPs induce DNA and chromosomal damage during embryo development, fetal malformations may arise. In 1996, Viana *et al.* (13) investigated the ability of the chain-breaking antioxidant  $\alpha$ -tocopherol ( $\alpha$ -TOH, vitamin E) to prevent the teratogenic effects of uncontrolled diabetes mellitus in rats (a study based on the hypothesis that diabetic animals have an elevated level of oxidative stress and therefore *in vivo* lipid peroxidation when expressed relative to that of healthy controls). They found that a PUFA-rich culinary oil (which served as a vehicle for oral administration of  $\alpha$ -TOH) increased the rate of malformations and reabsorptions in both normal and diabetic pregnancies. Further investigations (14) revealed that subjection of this safflower oil vehicle to thermal stressing episodes (according to standard frying practices for a period of 20 minutes) markedly enhanced its teratogenic effects. That is, the evidence indicates that the LOPs therein are primarily responsible for these actions.

**Further adverse health effects of dietary LOPs.** Further documented health effects of LOPs include their pro-inflam-



matory and gastropathic properties (for the latter, oral administration of HNE to rats at a dose level of only  $0.26 \mu\text{mol}\cdot\text{dm}^{-3}$ , a level similar to that of healthy human blood plasma, induced peptic ulcers), and also a significant elevation in systolic blood pressure and an impaired vasorelaxation observed in rats fed pre-heated soy oil (15).

## POTENTIAL SOLUTIONS AND FUTURE PERSPECTIVES

The most obvious solution to the generation of LOPs in culinary oils during frying is to avoid consuming foods fried in PUFA-rich oils as much as possible. Indeed, consumers, together with those involved in the fast-food sector, could employ culinary oils of only a low PUFA content, or MUFA- (or even SFA)-rich alternatives such as olive, selected canola, palm, or coconut oils for frying (MUFAs such as oleoylglycerol adducts are much more resistant to peroxidative degradation than are PUFAs (1), and hence markedly lower levels of only selected classes of aldehydes are generated during frying).

One further potential solution to this problem is supplementation or further supplementation of culinary oils with antioxidants such as  $\alpha$ -TOH. Note that concentrations of this antioxidant naturally present in cooking oils (for example, approximately  $2 \text{ mmol}\cdot\text{kg}^{-1}$  in corn oil, but only *ca.*  $0.3$ - $0.4$  and  $0.2 \text{ mmol}\cdot\text{kg}^{-1}$  in palm and soybean oils respectively) appear to be ineffective in preventing the thermally induced generation of high levels of CHPDs and aldehydes in these products (1), and our results have shown that additional supplementation with such agents also fails to offer protection.

Previous studies that investigated the prospective health effects or benefits of dietary PUFAs (i.e., those involving feeding trials with humans or animals or, alternatively, related epidemiological ones) should be scrutinized. With hindsight, it seems to us that many of these experimental investigations were flawed since, in addition to some major design faults, they failed to take into account or even consider the nature and concentrations of any cytotoxic LOPs present in the oils or diets involved. Similarly, corresponding epidemiological (or meta-analysis-based) investigations incorporated only the (estimated) total dietary intake of selected PUFAs and further fatty acids, and ignored any LOPs derived or derivable from frying/cooking. Even if PUFA containing culinary oils are unheated, it is virtually impossible to rule out the presence of traces of LOPs within them (analysis of apparently pure PUFAs or their corresponding TAGs obtained from reputable commercial sources has revealed that these materi-

CONTINUED ON NEXT PAGE



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als contain traces of CHPDs and/or aldehydes that are readily detectable by NMR).

Our original research investigations performed in the early 1990s in this key food toxicology/health research area have been repeated, replicated, and exemplified in many research group laboratories worldwide (e.g., 16) and have been available to the scientific community since then (1,2). Until just recently, this major problem has received scant or limited attention from the food industry and health researchers. Future clinical trial or epidemiological investigations aimed at determining relationships between the incidence of selected human diseases and dietary LOP consumption may serve to clarify the nature of such associations. We agree that completely pure, authentic, and essential PUFAs offer no threats to human health, but we point out that LOPs arising from the frequent and common use of PUFA containing frying/cooking media (or those produced during prolonged storage episodes) certainly do so.

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*Victor Ruiz Rodado is a postgraduate doctoral training student also at Leicester School of Pharmacy, and Christopher J. L. Silwood is a scientific consultant with major research interests in the bioanalytical chemistry, and lipid analysis/oxidation areas.*

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# Developing a high-performance, low-streak degreaser

“High-streak formulation”



## Julia Wates

A high-level review of technologies used in commercial aqueous household cleaners identified a gap in the market when products are classified according to their effectiveness at removing greasy soils and their streaking behavior.

All-purpose cleaners (APCs) contain relatively high amounts of surfactants—particularly nonionic surfactants. These products are excellent at removing greasy soils but can leave visible residues behind after drying. This results in an

- Many commercially available surfactant-based cleaners exhibit excellent degreasing performance but cause streaking. Glass cleaners cause less streaking but cannot remove tough grease.
- Numerous so-called multi-surface cleaners have been introduced in an attempt to fill this gap, but current commercial products do not deliver a strong combination of heavy duty grease removal and low streaking.
- This article describes the development of a surfactant-based cleaner that does.

unattractive streaky appearance and limits the type of surface on which these cleaners can be used. In addition, some APCs contain high levels of volatile organic compound (VOC) solvents, such as glycol ethers and alcohols, which are becoming increasingly less desirable as regulations get tighter.

At the other extreme are glass cleaners in which the main active ingredients are solvents and ammonia, with only trace amounts of surfactants. Such products cause significantly less streaking but cannot remove tough grease, so they are only effective on lightly soiled surfaces.

Multi-surface cleaners, which were introduced to fill the gap in performance between APCs and glass cleaners, tend to be similar to glass cleaners but with higher levels of so-called “low residue” surfactants like alkyl glucosides, amine oxides, and anionics. Most products in this class represent a compromise between cleaning and streaking and are not very effective at removing heavy duty grease.

Consequently, the challenge was to develop a surfactant-based cleaner that combines excellent degreasing with very low-streaking, preferably without high levels of VOC solvents. However, it quickly became apparent that the desired cleaning performance on heavy duty soils could not be achieved with classic low residue surfactants. So, the problem was approached from the opposite direction: by investigating whether the high-streaking of a good degreaser could be reduced by adding something else to the formulation. An earlier study (“New Technologies in Surface Care,” Julia Wates, 101st AOCs Annual Meeting, Phoenix, May 2010) had shown that adding a dispersion of colloidal silica to a cleaner leaves behind a hydrophilic layer on the surface after drying. This made the surface easier to clean the next time, especially in the case of greasy soil.

The breakthrough in the current project came when it was demonstrated that hydrophilic surface modification by colloidal silica can also reduce the appearance of streaking without



## “Low-streak formulation”



◀ **FIG. 1.** Appearance of dilute cleaning formulations spread on glass and photographed in a light box.

adversely affecting cleaning performance. This is important because cleaning and streaking typically oppose each other, so a change in composition that improves one of these parameters is usually detrimental to the other. Experiments in which cleaning, streaking, and formulation stability were simultaneously optimized demonstrated that a successful formulation needed at least two carefully selected surfactants plus the correct type of colloidal silica combined in the right ratios. A prototype low-streak blend was developed that met those requirements.

The specific requirements for a low-streak degreaser exemplified by the prototype (WO 2012/080197: “Low-streak degreaser composition,” J. Wates, M. Dery, A. Slikta and O B. Ho) are that it must contain at least one nonionic surfactant having a critical packing parameter of  $>0.95$ , inorganic nanoparticles such as colloidal silica, and a second surfactant having a critical packing parameter of  $<0.85$ .

The critical packing parameter or CPP of a surfactant molecule is a number that indicates the relative sizes of the hydrophilic head group and the hydrophobic tail. The larger the head group is relative to the tail, the lower the CPP. CPP is important because it is an objective measure that can be used to distinguish

between the primary nonionic surfactant (usually an alcohol ethoxylate), which is largely responsible for the degreasing performance of a cleaner, and secondary or co-surfactants (such as alkyl glucosides, amine oxides, or amphoterics) that are not very effective degreasers on their own but provide other functions in a formulation. There is a relationship between CPP and the shape of a surfactant molecule which, in turn, determines how a surfactant self-assembles or forms aggregates in aqueous solutions and on surfaces. Table 1 summarizes the connection between CPP and the shapes of surfactant molecules and their aggregates. Secondary or co-surfactant molecules that have low CPP values are shaped like cones or truncated cones, and they tend to form spherical or cylindrical micelles with the large head groups on the outside and the smaller tails on the inside. On the other hand, when the CPP is close to 1, as is the case for many alcohol ethoxylates, the cross sections of the head and tail are similar in size. This results in the individual molecules being cylindrical, and their aggregates are lamellar phases or planar bilayers.

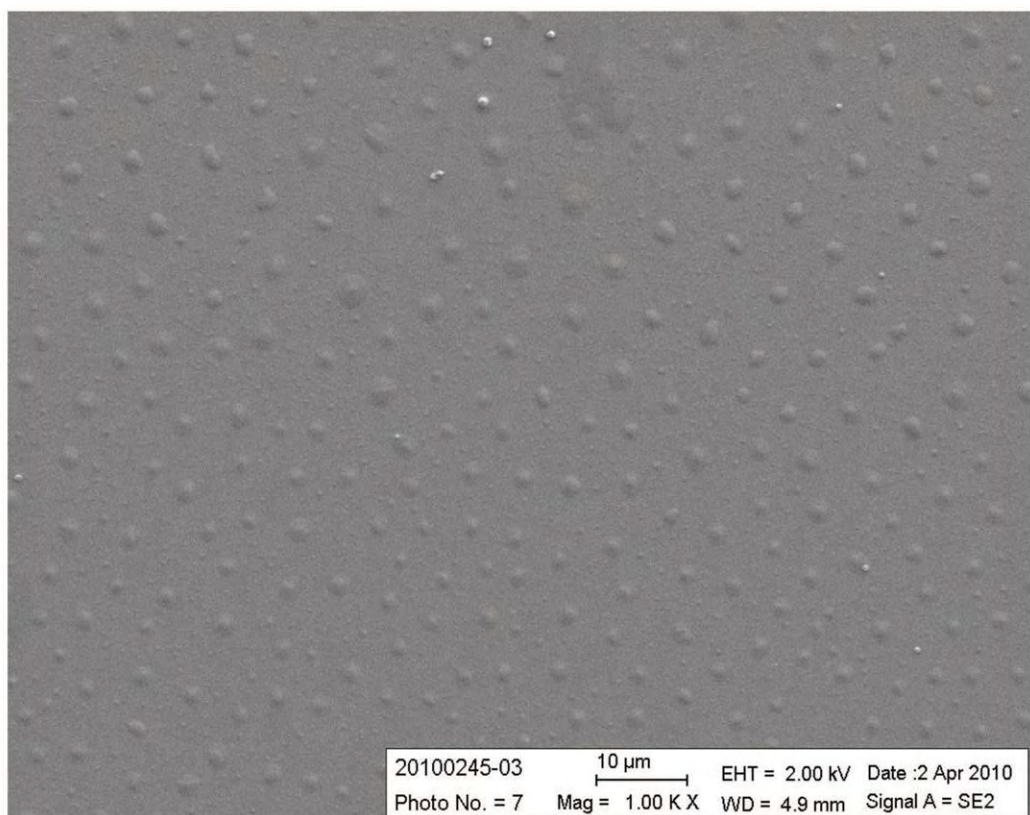
Streaking is a dynamic process that occurs as water and other solvents in a cleaner evaporate from a treated surface and the surfactant concentration in the formulation gets higher and higher. Streaks are simply visible residues of surfactants and other ingredients that are left behind on a surface after the application and drying of a cleaning product. Nonionic surfactants with high CPP values that form lamellar phases or other large aggregates at high concentrations are most likely to leave visible residues or streaks. In contrast, secondary surfactants with low CPP values that tend to form smaller aggregates are lower streaking, suggesting that streaking can be reduced by controlling surfactant self-assembly on the surface. The aggregation behavior of a single surfactant can be predicted by its CPP, but self-assembly of mixed surfactants will depend on the ratios and CPP values of all the components and may be quite different from that of any individual surfactant. By combining surfactants with different

CONTINUED ON NEXT PAGE

**TABLE 1.** Relationship between critical packing parameter and shape of surfactant molecules and self-assemblies

CPP	Shape of molecule	Shape of surfactant aggregates
$< 0.33$	Cone	Spherical micelles
$0.33 - 0.5$	Truncated cone	Cylindrical micelles
$0.5 - 1$	Truncated cone	Lamellar (vesicles)
$\sim 1$	Cylindrical	Lamellar (planar bilayers)
$> 1$	Inverted truncated cone	Reverse micelles

**FIG. 2.** SEM image of high-streak formulation on glass at 1000 x magnification



CPP values in the right way, it should be possible to manipulate the phase behavior of the mixed systems and minimize formation of lamellar regions at high surfactant concentration without sacrificing good degreasing performance in the dilute cleaner.

To determine whether this theory is valid in practice, an investigation was conducted into the mechanism of streaking for two dilute cleaning formulations. The high-streak formulation was a classic aqueous degreaser based on a primary non-ionic surfactant with CPP  $\sim 1$ , while the second formulation was the low-streak prototype containing an optimized blend of nonionic and secondary surfactants with colloidal silica. Both formulations were applied to various surfaces and allowed to dry. Their appearances were then compared with different techniques. Figure 1 (page 627) shows the two cleaners spread on glass mirror tiles and photographed in a light box against a black background.

When the high-streak formulation was viewed under a light microscope at low magnification, the streak appeared as a lane with spots in it. A scanning electron microscope (SEM) image of the streak at 1000 x magnification (Figure 2) revealed that these spots are evenly spaced “blisters” having an average diameter of 2–5  $\mu\text{m}$ . The structure of the blisters was studied in more detail with atomic force microscopy (AFM), a technique used to investigate the shape and features of surfaces at very small scales.

Information is gathered by “feeling” the surface with a fine-tipped cantilever that is deflected by surface forces in a manner that is analogous to a stylus moving over the grooves of a vinyl record. In this way, a picture is built up of what the surface looks like. Figure 3 shows an AFM image looking down at a single

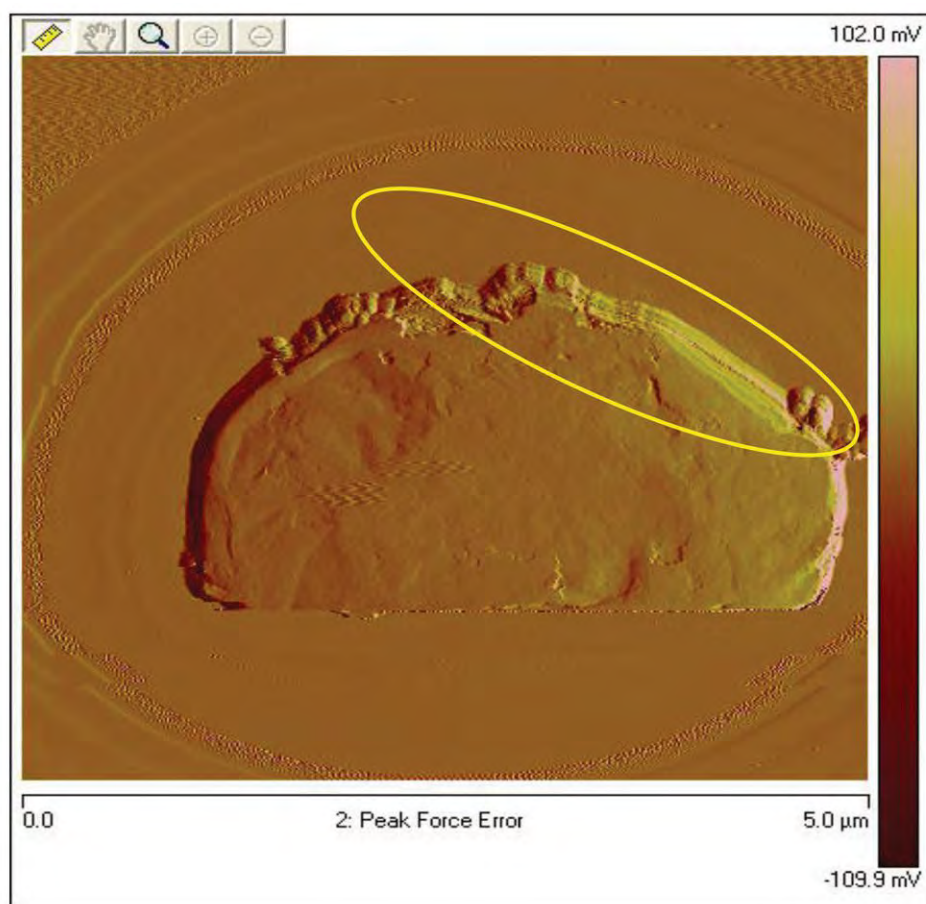
blister on an oxidized silicon wafer spin-coated with the high-streak formulation. The scale of the image is 5  $\mu\text{m}$  across, and the blister appears as a flat spot surrounded by empty space. Closer inspection shows that the blister is built up from layers of lamellar structures perpendicular to the treated surface. These are assumed to be surfactant, and this observation is consistent with the proposed mechanism for streaking.

A simple drying experiment was run using a rotary evaporator to simulate what happens as the cleaner loses water and dries on a treated surface. Phase separation was observed at  $\sim 50\%$  solids content and although the mixture remained fluid and pourable, the separation was irreversible and the mixture could not be diluted back with water to a single phase. This suggests a mechanism for streaking in which phase separation upon drying results in the formation of multiple nucleation centers on the surface leading to the 2–5  $\mu\text{m}$  blisters with almost no material in between. The blisters are large enough to scatter light, making them visible to the naked eye as streaks.

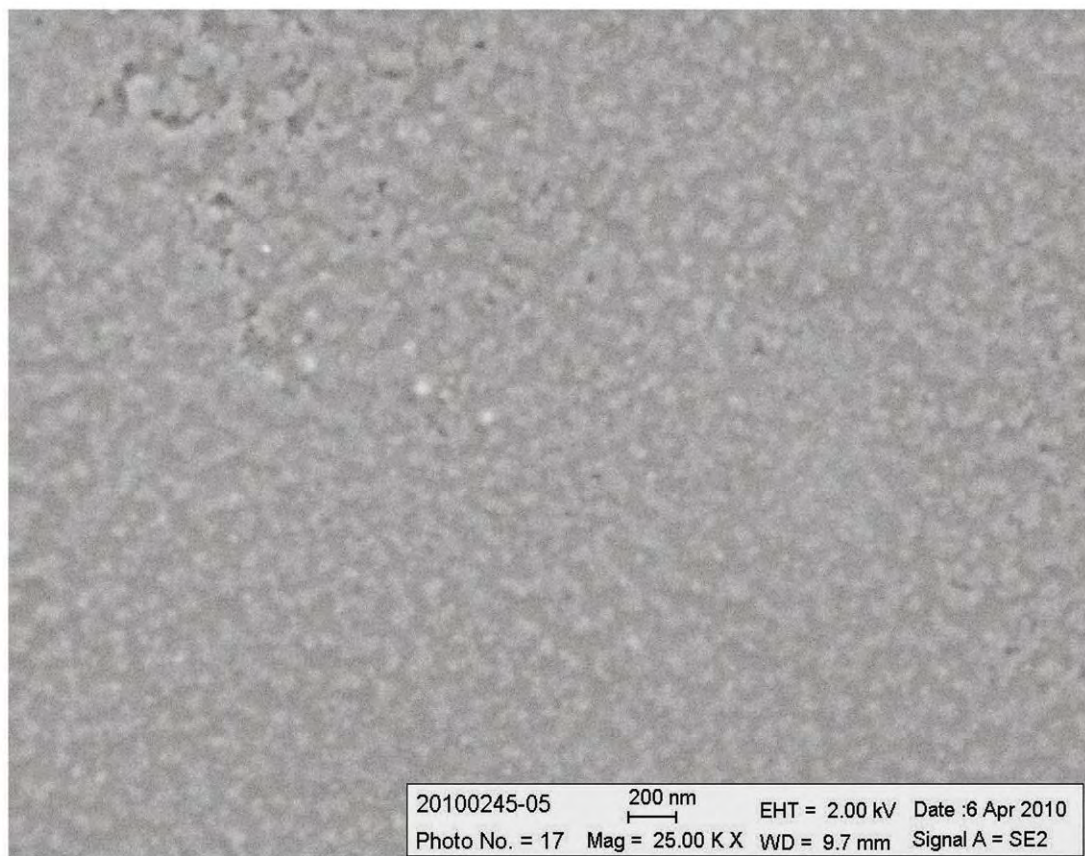
Moving next to the low-streak formulation, nothing was observed under the light microscope at low magnification where the cleaner was applied. Even with the SEM at much higher magnification, the material appeared to be evenly distributed on the surface and there were no remarkable structures visible. At the highest SEM magnification of 25K (Figure 4), a fine pattern emerged with darker regions that looked like holes where there was no material present. The AFM images for the low-streak formulation are consistent with this interpretation. Figure 5(a), page 630, has a scale of 5  $\mu\text{m}$  across and confirms

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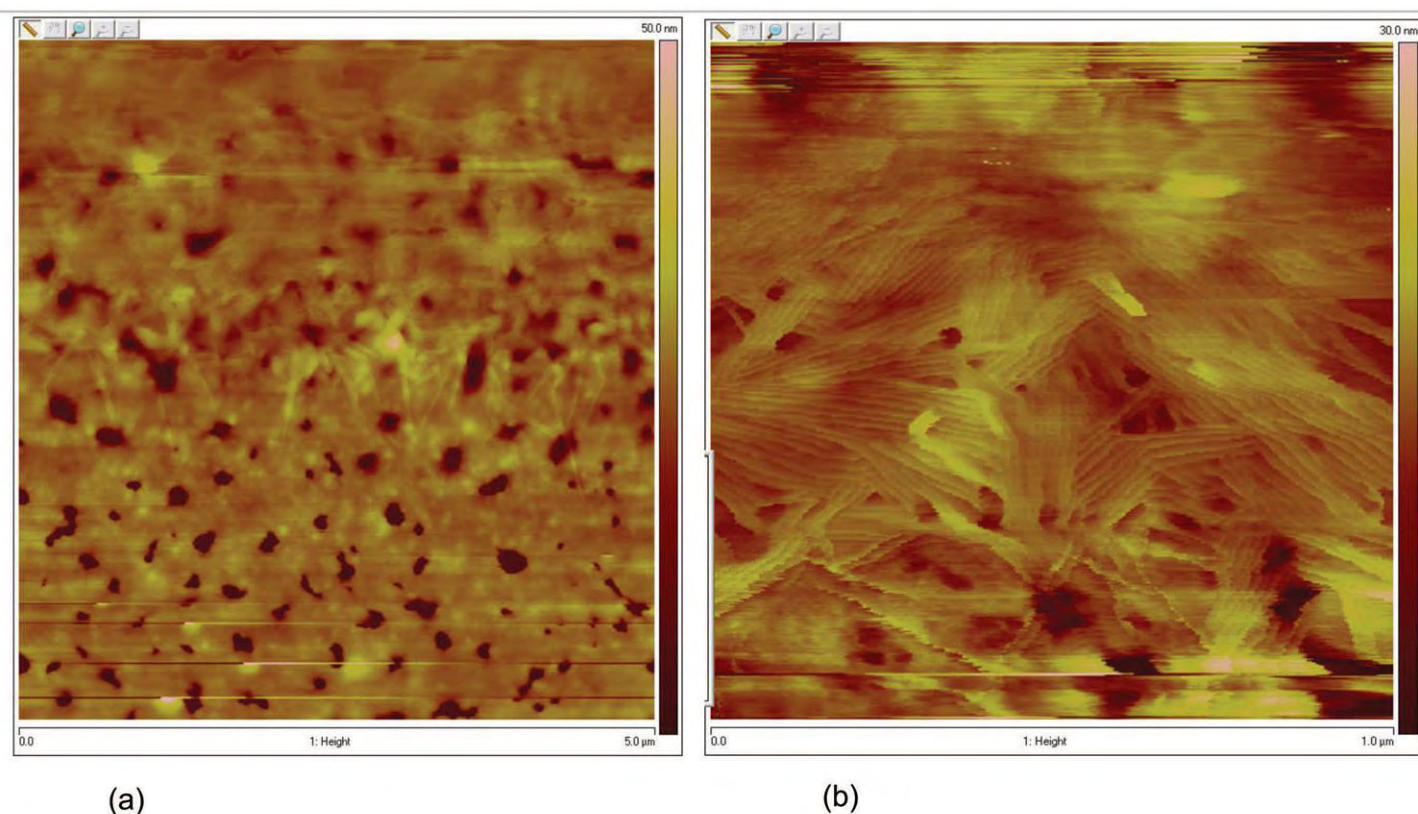


◀ **FIG. 3.** AFM image of high-streak formulation highlighting layers of surfactant lamellar structures. Scale is 5 μm across



◀ **FIG. 4.** SEM image of low-streak formulation on glass at 25K x magnification





**FIG. 5.** AFM images of low-streak formulation. (a) scale is 5  $\mu\text{m}$  across (b) scale is 1  $\mu\text{m}$  across

that the surface is covered by sub-micron holes. Figure 5(b) has a smaller scale of 1  $\mu\text{m}$  across and it shows that the holes are separated by material made up of rod-like structures with a repeat dimension of 10-20nm. The most likely explanation for the generation of these structures is that the colloidal silica particles in the formulation interact with any remaining water in the surface film on drying, and are attracted by capillary forces into the gutters between the wormlike micelles that form at high surfactant concentration. Consequently, the micelles are coated with silica and become more rigid and rod-like. In contrast with the behavior of the high-streak formulation, there are no “large” (micron-scale) structures present on the surface that can scatter light.

The study was completed by running the drying experiment for the low-streak formulation. In this case, the evaporated material was highly viscous and not pourable which is consistent with the presence of wormlike micelles. However, the mixture remained transparent and was easily diluted back to the original water level, indicating that there was no phase separation. What this means is that the material forms an evenly distributed film on the surface as it dries; there are no structures large enough to scatter light and therefore no streaks. Interestingly, when the silica is removed from the formulation, the behavior moves toward that of the high-streak formulation and there is some phase separation at very high solids content. So, streaking can be reduced by balancing the critical packing parameters of the surfactants, but to obtain zero streaking the silica is needed to stabilize the wormlike mixed micelles formed during

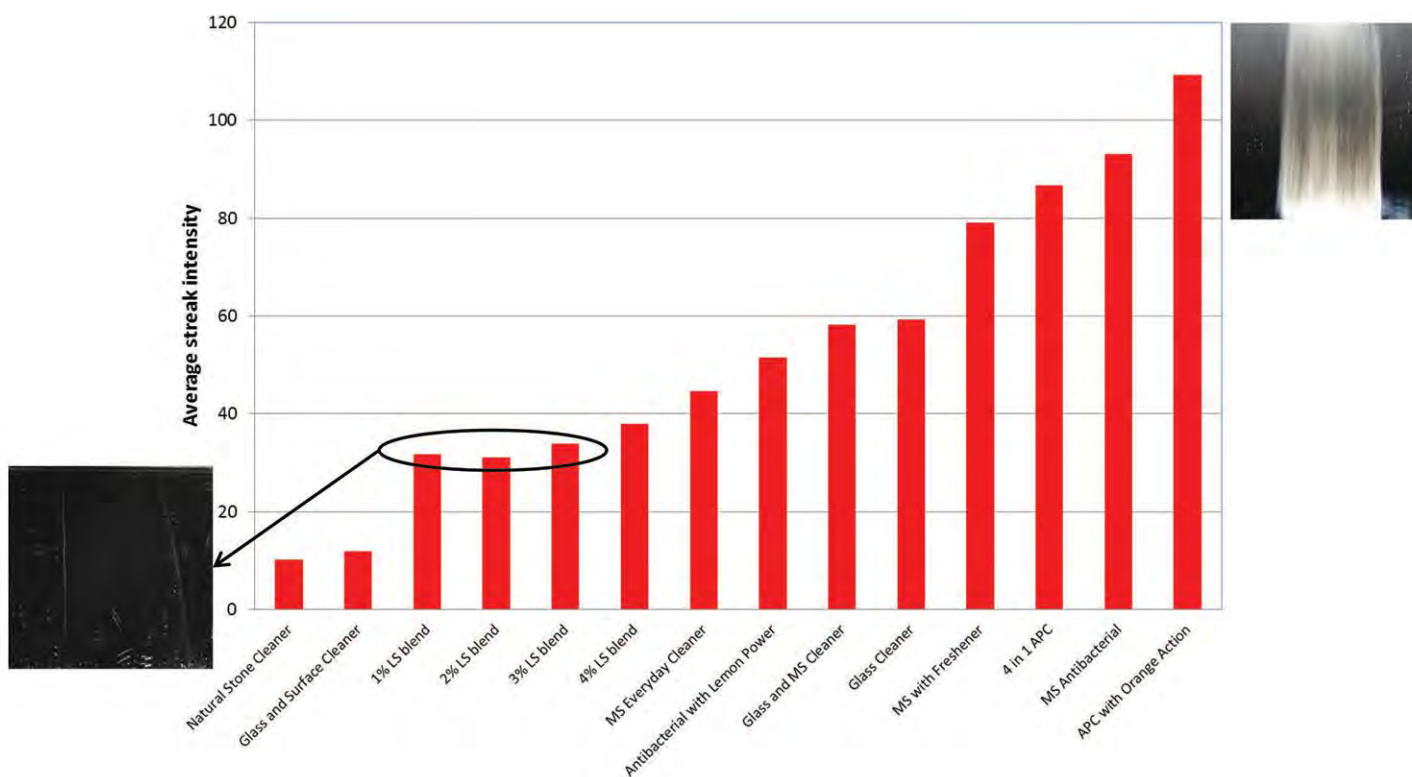
drying and prevent the system from eventually transitioning to a lamellar phase.

The graph in Figure 6 compares streaking data for the prototype low-streak blend formulated at different concentrations with commercial ready-to-use household cleaners applied to glass mirror tiles and photographed in a light box. Average streak intensities were generated by image analysis. A low number indicates low-streaking (anything below around 30 is invisible to the naked eye). Only two commercial cleaners demonstrated less streaking than the low-streak prototype formulations. At the other extreme, an “all-purpose cleaner with orange action” was very streaky.

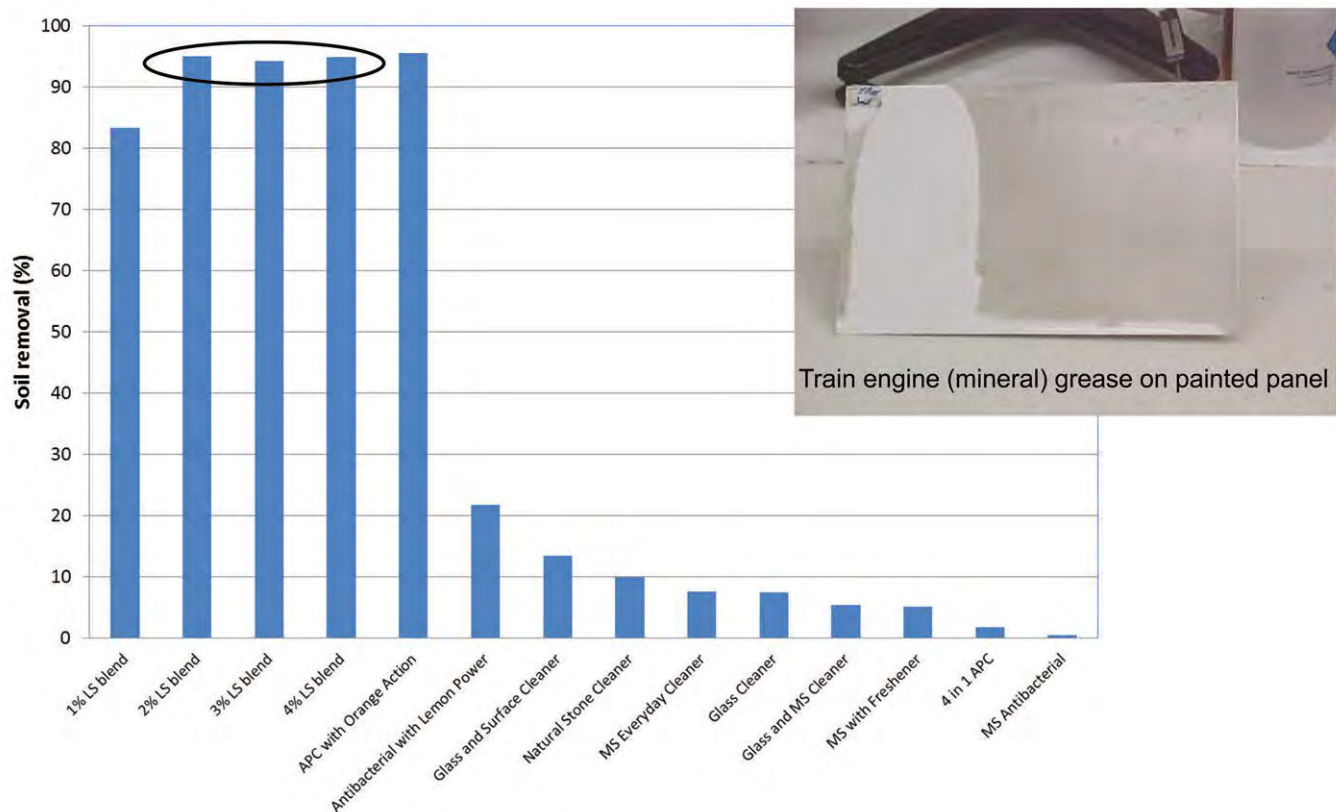
Although the main focus of this article has been on streaking, the low-streak prototypes also had to meet stringent targets for degreasing performance. Figure 7 shows the results of a non-mechanical test in which the cleaners are poured onto a painted metal panel coated with mineral grease and rinsed with water.

The prototype low-streak formulations were able to remove more than 90% of this very tenacious soil. The only commercial cleaner that gave comparable degreasing performance was the “all-purpose cleaner with orange action.” But, as previously demonstrated, this cleaner was also the highest streaking product. Figure 8 (page 632) shows the performance of four formulations in mechanical cleaning tests in which a greasy kitchen soil was baked onto stainless steel panels and removed with a four-lane scrub tester. The commercial all-purpose and multi-surface

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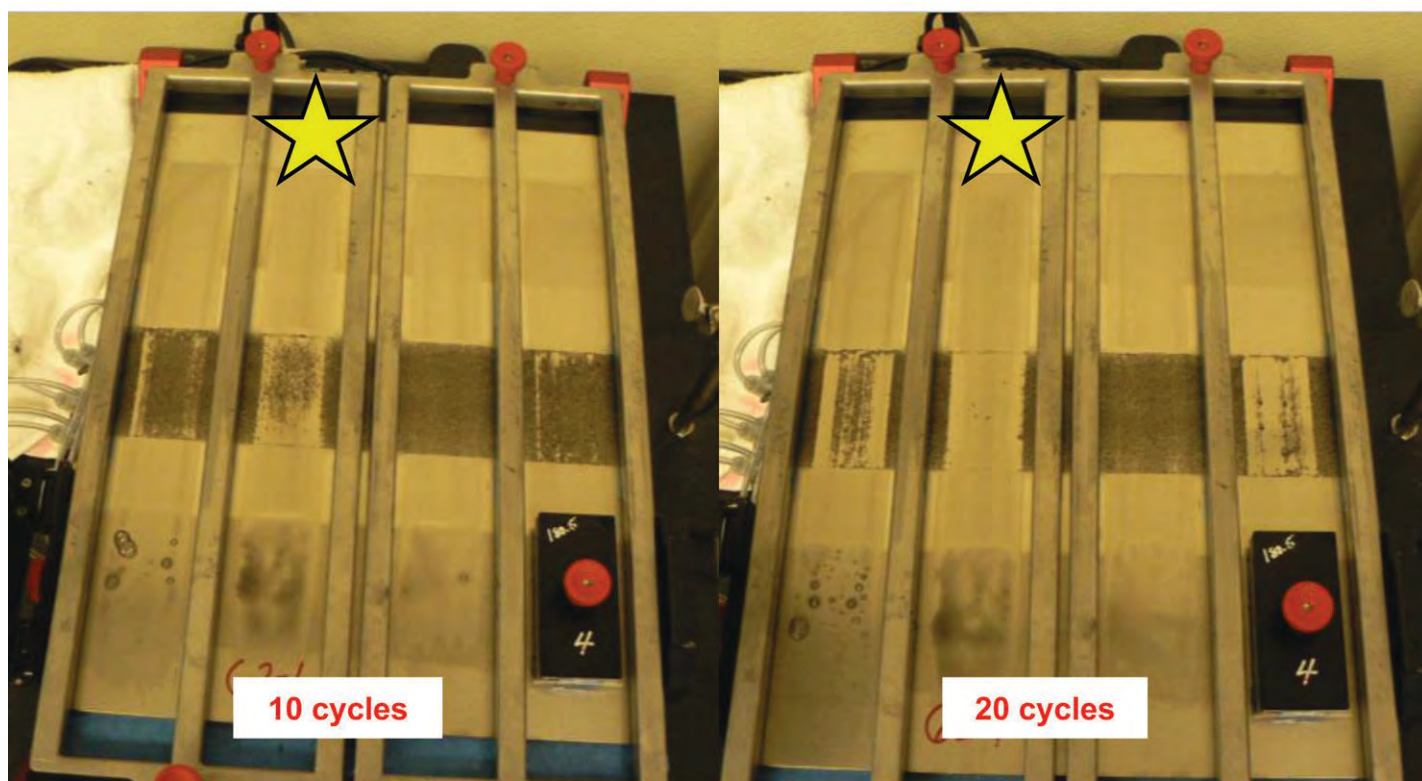


**FIG. 6.** Streaking data on glass for prototype low-streak formulations and commercial household cleaners. Low-streak (LS) blend formulated with 0.4% Na<sub>4</sub> EDTA, balance water



**FIG. 7.** Non-mechanical degreasing for prototype low-streak formulations and commercial household cleaners. Low-streak (LS) blend formulated with 0.4% Na<sub>4</sub> EDTA, balance water





**FIG. 8.** Mechanical cleaning performance on greasy kitchen soil (left to right): all-purpose cleaner, low-streak formulation, glass cleaner, multi-surface cleaner. 2% low-streak blend, 0.4% Na<sub>4</sub> EDTA, 0.25% ethanolamine (optional), balance water

cleaners performed quite well after 20 scrub cycles, while a glass cleaner barely removed any soil. The prototype low-streak formulation gave excellent soil removal under these test conditions.

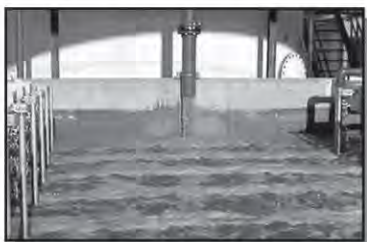
Finally, it should be mentioned that although the reference products in this study were household cleaners, the same technology has been successfully used in industrial and institutional applications where a combination of excellent grease removal

and low-streaking is required, such as in heavy-duty window cleaners for external use on buildings in high-traffic areas.

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# Welcome New Members

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

\*New and reinstated members joined from January 1, 2014 through September 24, 2014.

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# What's the automotive aftermarket industry hiring surfactants to do?



**Liliana Minevski**

It's not easy to find an industry that does not use surfactant-based chemistry. Table 1 (page 640) depicts the wide range of uses for surfactants within ITW Global Brands (a diversified company with seven segments that supplies products to a variety of industries). The automotive aftermarket (AAM) is a heavy user of surfactants within the polymer and fluids category. AAM applications in this category include interior and

exterior detailing and vehicle maintenance. Typical products may include glass treatments, wash and wax, tire and wheel care, and under-the-hood additives for engine, oil, fuel systems and fuel tanks; and users include car enthusiasts, motorheads, and "bare necessity"-type consumers.

Application of AAM products containing surfactants provides consumers with nearly instantaneous gratification as they enjoy the almost immediate result of a clean vehicle having shiny, protected, lubricated, and disinfected plastic, metal, rubber, and upholstery surfaces—all of which can be achieved by spraying, wiping, or rubbing as the wetting and emulsifying properties of the surfactants lift off and disperse soil.

## SOIL AND SURFACES

Soils encountered in AAM applications are hydrophobic—for example, oils and greases—and hydrophilic—such as interior stains from fruits and vegetables or exterior stains from sap dripping on glass and paint surfaces. Such soils are usually complex systems made of multiple components that are inorganic and organic in nature and that are affected by aging, heat, and environmental conditions. Similarly, the surfaces that are treated (plastic, rubber, glass, and metal) are also complex systems described as hydrophobic and hydrophilic. Such surfaces can be porous and nonporous, vary in shape and size, and are sometimes difficult to approach—particularly wheel bolts, various engine parts, and the crevices between the dashboard and windshield.

*Soil removal and surface modification.* In the complex world of soil and surfaces, the question is how to accomplish soil removal. The answer depends on the energy of soil and surfaces and the forces of interaction between two systems in which the interfacial surface energy is either lowered or increased to enhance wetting or emulsification. Thus, to remove soil one needs to understand the interactions between several paired systems: soil–surface, soil–product, and surface–product. For example, if interaction between soil and surface is high, the soil is not removed and remains on

- **The automotive aftermarket (AAM) involves the sale, repair, and installation of vehicle parts, chemicals, equipment, and accessories for automobiles after they've been sold by the original equipment manufacturer.**

- **A variety of AAM products, such as glass treatments, wash and wax, tire and wheel care, and under-the-hood additives for engines, oil, fuel systems and fuel tanks make use of surfactants.**

- **This article reviews the use of surfactants in AAM applications and how surfactant use benefits consumers, impacts the environment, and is currently regulated.**





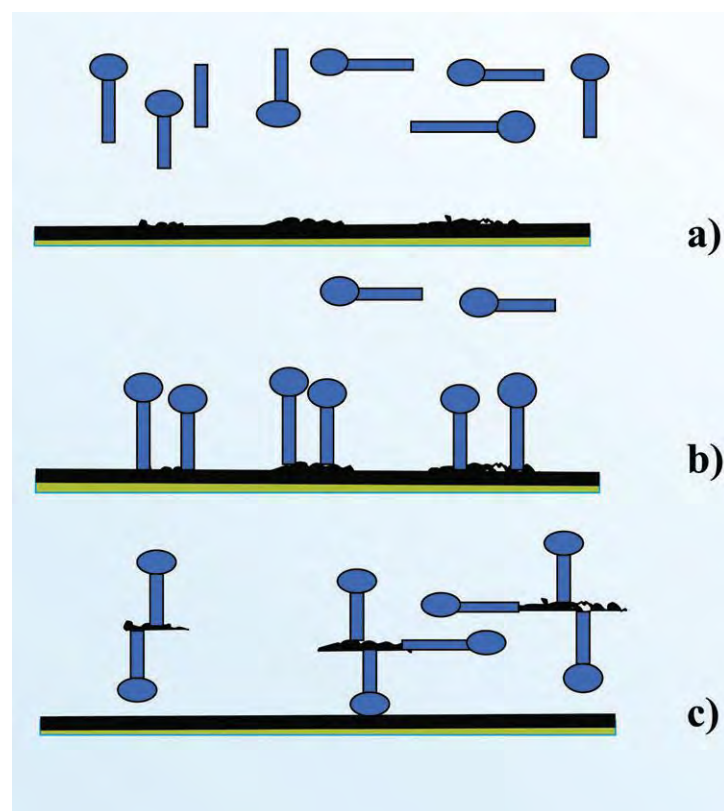
the surface. On the other hand, if interaction between soil and product or surface and product is high, soil is released or dissolved (see Fig. 1).

In some other applications, soil removal is secondary, and modifying the surface to either wet or bead water by increasing or decreasing surface energy becomes essential. Two examples of surface modification are shown in Figure 2 (page 640). A liquid material with lower surface energy will tend to wet a solid material with a higher surface energy. In contrast, if a liquid placed on a substrate has a higher surface energy than the substrate, it will tend to bead up. For example, a car wash detergent will reduce the surface tension of water while a car wax will deposit a low-energy surface on the paint.

**Cleaning tires and wheels.** Improving the appearance of tires and wheels is accomplished by wetting the surface, then lifting and suspending contaminants. Usually, tire dressing completes the process of detailing a car with an “as new” or “wet” shiny black finish (see Fig. 3a,b, page 641).

Dirt on wheels and tires is organic and inorganic in origin. Mineral oil from vehicle exhaust and road surfaces, fine particles of carbon black, and graphite and resins from brake pads are considered organic. Dust, traffic grime, metal particulates, and inorganic fibers from brake dust are considered inorganic. The material to be cleaned from each vehicle wheel or tire varies with different vehicles, driving locations, and environmental conditions—though the damage to coatings and wheels is always more pronounced in the presence of moisture as water droplets react with soil and brake dust.

Wheel cleaners have traditionally been designed to be aggressive so as to clean tough brake dust and road film. These products



**FIG. 1.** Surfactant molecules in action: (a) evenly distributed in a cleaning solution above a soiled surface; (b) attaching to soil and/or surface; (c) lifting soil from the surface

CONTINUED ON NEXT PAGE

**TABLE 1.** Various industries and possible uses of surfactants <sup>a</sup>

Industry type	Application
Automotive OEM	Modifying metal and plastic surfaces
Test, measurement, and electronics	Lowering surface tension or improving wetting of variety of fluids or surfaces
Food equipment	Water and oil repellency of metal surfaces
Polymers and fluids	Modifying properties of adhesives and sealants, lubrication, cleaning, sanitizing, AAM, solar, and wind
Welding	Improving properties and application of consumables
Construction products	Lubrication and corrosion protection
Specialty products	Improving performance of jet inks, plastic consumables, foils

<sup>a</sup>Abbreviations: OEM, original equipment manufacturer; AAM, automotive aftermarket.

are either harsh on aluminum wheels or too weak to clean brake dust. This is where good wetting surfactants are put to use, those that are strong enough to clean brake dust and safe for any metal surface.

A synergistic combination of nonionic (e.g., alkanolamide, alcohol ethoxylate) and anionic (e.g., di-octyl-Na-sulfosuccinate) surfactants with excellent wetting, emulsifying, and cleaning characteristics is safe for all metallurgies and pH balanced for consumer friendly application (Fig. 3a).

In addition to cleaning tires and wheels, surfactants are used as leveling wetting agents to spread tire dressing (Fig. 3b) and to reduce the surface tension of tires. This aids the application of silicone-based products for better shine and durability.

**Fuel system clean-up.** The performance of a vehicle can be improved by applying surfactants onto particulate soil, where they suspend the soil and help prevent redeposition of particulate soil via charge repulsion.

With time, a vehicle's fuel system, including its combustion chamber, intake valves, and fuel injectors, become covered with carbon deposit, varnish, and gum. If a car is driven roughly, such deposits can build up on the port and intake valves (Fig. 3c) very quickly. Each time the engine stops, small droplets of fuel left at the tip of the fuel injector dry, harden, and carbonize over

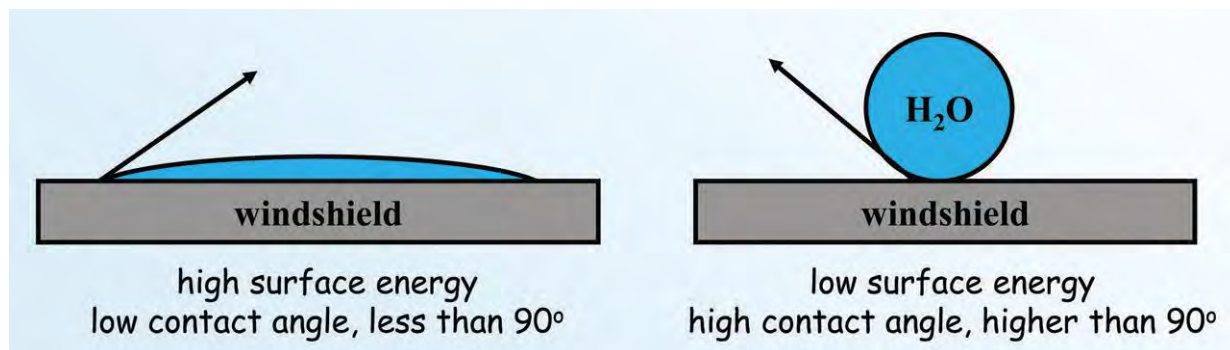
time (Fig. 3d). The resulting obstruction blocks the fuel flow and changes the spray pattern from a fine mist to larger droplets and streams, which prevent the fuel from vaporizing and burning quickly and efficiently. The outcome is a hindered cold start, power loss, increased rough idle, and hesitation.

Removal of oxidized fuel is accomplished by using a fuel additive system with the right type and amount of chemistry (e.g., polyisobutylamine or polyetheramine). A complete fuel system cleaner cleans fuel injectors, intake valves, and combustion chamber deposits by preventing further fuel absorption. Clean vehicle parts allow fuel to enter the combustion chamber for proper atomization and good engine performance.

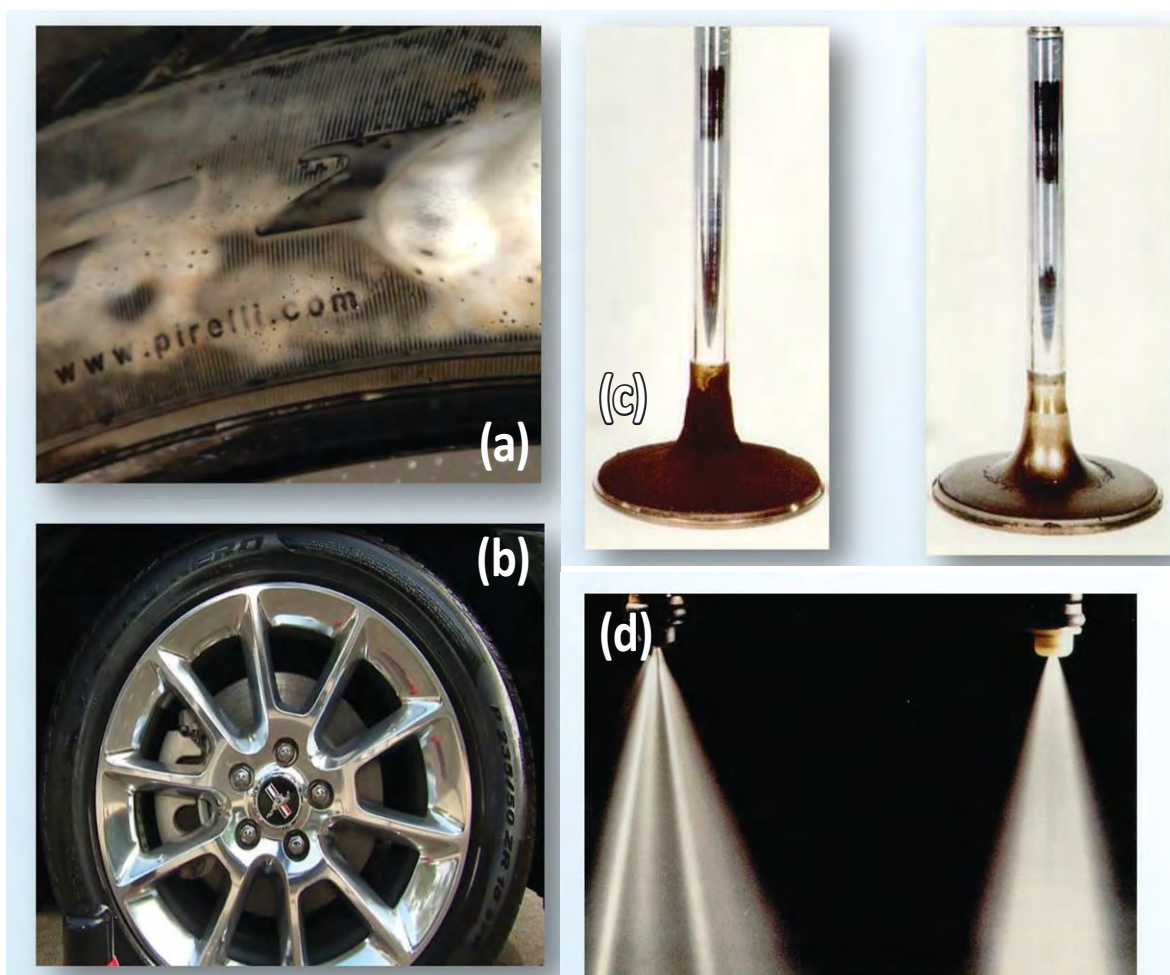
**Fog and water removal.** To remove fog or water, glass surfaces must be modified to exhibit either hydrophilic or hydrophobic properties. The nature and degree of glass modification (Fig. 4.) is characterized by the contact angle at the air/glass interface, where better wetting (hydrophilic) and better beading (hydrophobic) occur at lower and higher contact angles, respectively.

When tiny droplets of water are formed on glass, due to temperature differences between the two sides of the glass, the light that normally passes through the glass refracts and reflects within

CONTINUED ON PAGE 642

**FIG. 2.** Water wetting vs. beading





**FIG.3.** Surfactants in action. (a) Taking off accumulated dirt; (b) a clean and dressed tire; (c) left: intake valve with deposit and right: cleaned valve; (d) fuel injectors 25% and 0% plugged, left and right, respectively



**FIG. 4.** Stages of glass surface modification



FIG. 5. “Real time” windshield properties: treated and untreated surface during (a) fog and (b) rainy conditions

the water droplets and does not transmit (see right-hand side of the windshield in Fig. 5a). For the glass to exhibit non-fogging conditions and for light not to be attenuated, the windshield surface must be modified with a thin layer of wetting agent such as silicone polyether copolymer or sodium polyacrylate. Such surface-active agents increase the surface energy and lower the water contact angle to about zero degrees. The presence of superabsorbent or water absorbent on the glass under fogging conditions is shown on the left-hand side of the windshield in Figure 5a.

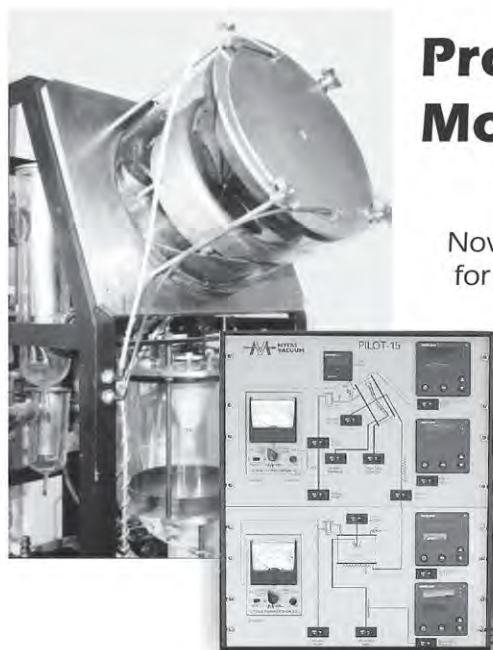
While wetting of glass is beneficial inside the vehicle, water beading is a desired glass property on the outside of the vehicle, where the desired outcome is to remove water droplets or large amounts of muddy water splashed on the windshield while driving (Fig. 5b).

Surface modification occurs at the treated air/glass interface, where in the presence of moisture silanol groups ( $\text{Si-OH}$ ) are negatively charged ( $\text{Si-O}^-$ ) and undergo a reactive condensation reaction with polydimethyl siloxane. The result is a low-surface energy glass covered with a lasting water-repelling monolayer

and improved safety and reaction time under stormy weather elements.

## ENVIRONMENTAL IMPACT AND COMPLIANCE

In the United States, federal and state government influence development of AAM products. Such initiatives protect not only the environment but also the consumer. For this reason, scientists are formulating products to comply with regulations set by the US Environmental Protection Agency (EPA) and two California environmental offices: (i) the California Air Resources Board (CARB), which oversees the content of volatile organic compounds in products sold in the state, and (ii) the Office of Environmental Health Hazard Assessment (OEHHHA), which oversees exposure to toxic chemicals and ingredients known to cause cancer or birth defects, that is, California's Proposition 65 list. The list contains more than 800 chemicals, including diethanolamine (DEA), cocamide DEA, and methanol. An initiative to list ethylene glycol,



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found in surfactants, emulsifiers, car antifreeze, and brake fluid, is currently in the public comment stage. So is the VOC-compliant solvent acetone. Since regular grades of this solvent contain some benzene impurities, manufacturers will have to replace those with more expensive grades of “clean” acetone to avoid the “contains a chemical known to cause cancer” label.

The Consumer Specialty Products Association (CSPA), a trade association representing the interests of companies engaged in the manufacture, formulation, distribution and sale of consumer products, is another organization that oversees AAM products. CSPA gives product guidelines and has published a consumer products ingredient dictionary, which includes ingredients accepted by the US EPA’s Design for the Environment.

Many industrial and scientific organizations have joined forces with the US EPA (e.g., with the EPA-sponsored Green Chemistry Challenge) to promote greener, better products and to help researchers design products that reduce or eliminate the use of hazardous substances. Though many consumer products display self-made or industry-recognized seals identifying environmental compliance, AAM products to date have tended not to identify such certification—even when they comply with all the above mentioned regulations and standards. This may change, since merchandisers and auto retailers are recognizing CSPA’s efforts for ingredient disclosure and chemical reduction in automotive chemicals and appearance products.

## ARE THERE ANY GAPS, AND WHAT DOES INDUSTRY NEED?

All industries, including AAM, are constantly looking for new product portfolio from scientific and performance standpoints. Continuous improvement and superior formulae and benchmarking products are a must in highly competitive and fast-moving consumer goods businesses. Innovative solutions and a deep knowledge of end-user needs are moving forward the appearance and performance of AAM categories.

Product differentiation is achieved through cost effective ingredients and test standards in combination with blind, field, and focus group evaluations. Statistical design is used to substantiate product label claims since industry needs powerful products that are designed by evaluating different surfactants, utilizing their physicochemical properties, and implementing specific test procedures that simulate desired applications.

Conceptually, the question “Are there any gaps in the grand surfactant space?” is answered with another question: “What are the main features and benefits of a surfactant-based product?”

The answer that immediately comes to mind is that not only the ingredient (surfactant) but the fully formulated product needs to deliver high performance, stability, cost effectiveness, regulatory compliance, and an aesthetic appearance. With attractive packaging, such characteristics ultimately build strong reputable brands, consumer loyalty and sustainable differentiation in the marketplace.

*Liliana Minevski is Director of R&D and Technology, ITW Global Brands in Houston, Texas, USA. She has a Ph.D. in physical chemistry from Texas A&M University and can be contacted at Liliana.Minevski@itwgb.com.*



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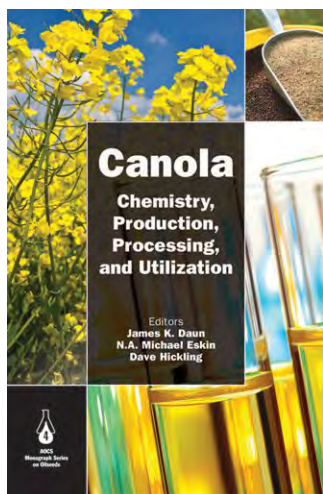
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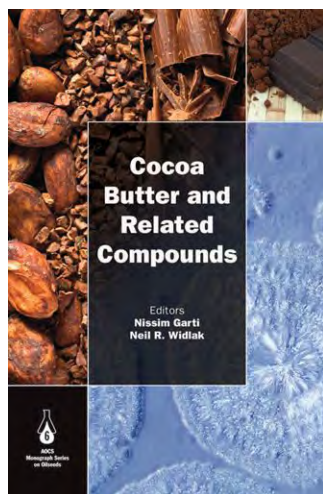


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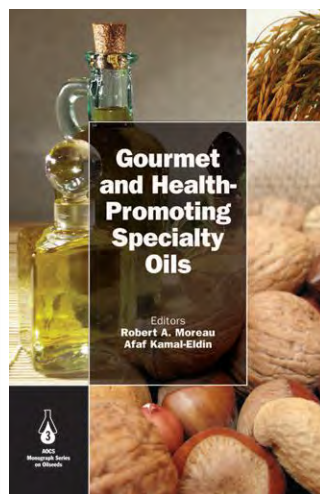


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This book covers the progress of the last 10 years of studies on cocoa butter. Descriptions of aspects including physical characteristics such as rheology, hardness, melt profiles, and more, studied by new and advanced techniques are included. Similarly, the polymorphism of cocoa butter is reconsidered in light of studies done by synchrotron DSC, FTIR, and SAXS techniques. These data are complemented by new understandings on the cause of the crystallization and transitions of the polymorphs. Other aspects, such as the effect of minor components, emulsifiers, and other fats, are discussed in great detail.

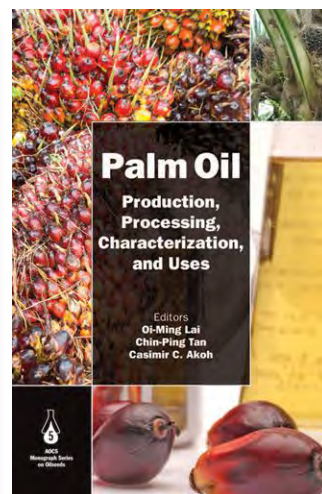


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This book serves as a rich source of information on the production, processing, characterization, and utilization of palm oil and its components. It also includes several topics related to oil palm genomics, tissue culture, and genetic engineering of palm oil. Physical, chemical, and polymorphic properties of palm oil and its components as well as the measurement and maintenance of palm oil quality are included and may be of interest to researchers and food manufacturers. General uses of palm oil/kernel oil and their fractions in food, nutritional and oleochemical products, and the potential use of palm oil as an alternative to trans fats are discussed. Some attention is also given to palm biomass, bioenergy, biofuels, waste management, and sustainability.

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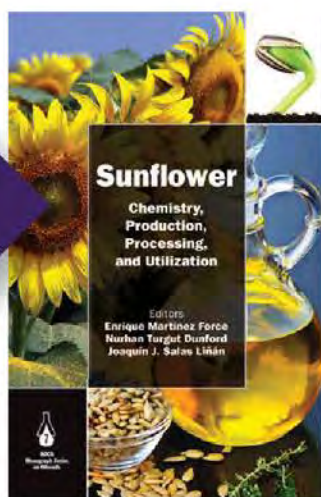
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# Lowering the boiling curve of **biodiesel** by metathesis

**Jürgen Krah, Christoph Pabst, Kevin Schaper, Olaf Schröder, Michael Meier, Peter Eilts, Jürgen Bünger, Axel Munack**

The tightening of emissions standards places increasingly high demands on vehicle manufacturers and fuel quality. Currently, biodiesel is the biogenic component predominantly used as blend component in diesel fuel. Unfortunately, the high boiling curve of fatty acid methyl esters (FAME) is not very well suited for modern passenger car engines with diesel particle filters (DPF). During the DPF regeneration phase, diesel fuel and biodiesel can dilute the engine oil [1]. While the diesel fuel can be evaporated from the oil pan, the biodiesel remains in the oil and leads to oil dilution and subsequently to the formation of oligomers and polymers that result in oil sludge. Therefore, we decided to

use metathesis to change the boiling behavior of biodiesel and enable its evaporation from the engine oil [2].

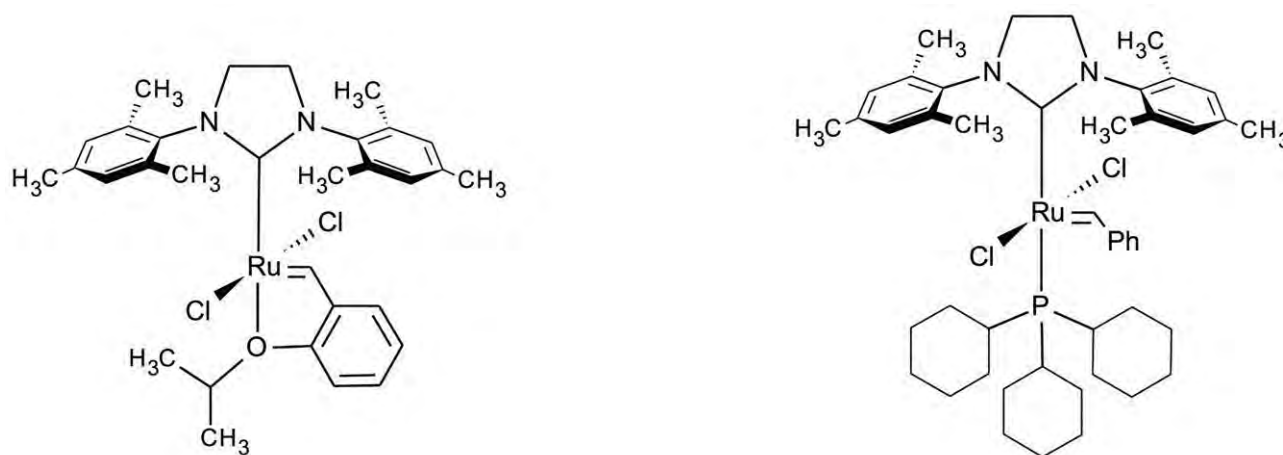
## METATHESIS REACTIONS TO MODIFY BIODIESEL

We conducted self-metathesis and cross-metathesis reactions to adapt the boiling curve of biodiesel to the boiling curve of fossil diesel fuel.

The mechanism of metathesis reactions was first described by Yves Chauvin in 1971. In 1990, Richard R. Schrock developed a more effective catalyst and two years later Robert H. Grubbs described ruthenium-based catalysts, which are also effective and more stable towards water and oxygen. All three scientists obtained the Nobel Prize in Chemistry in 2005 [3]. Two of the catalysts developed by Grubbs are shown in Fig. 1.

During the metathesis reaction, two alkenes interchange their alkylidene rests. In case of only one alkene, the reaction is called self-metathesis. Alternatively, if two different starting alkenes are used, the reaction is called cross-metathesis (Fig. 2). Depending on the percentage of the educts, an equilibrium of products is obtained including self-metathesis products.

Self-metathesis changes the alkylidene rests of unsaturated methyl esters in biodiesel. Using self-metathesis, the products are both lower and higher molecular. The higher molecular products have higher boiling points than biodiesel and are therefore tentatively undesired.



**FIG. 1.** Structure of the Hoveyda–Grubbs second generation (left) and Grubbs second generation catalysts



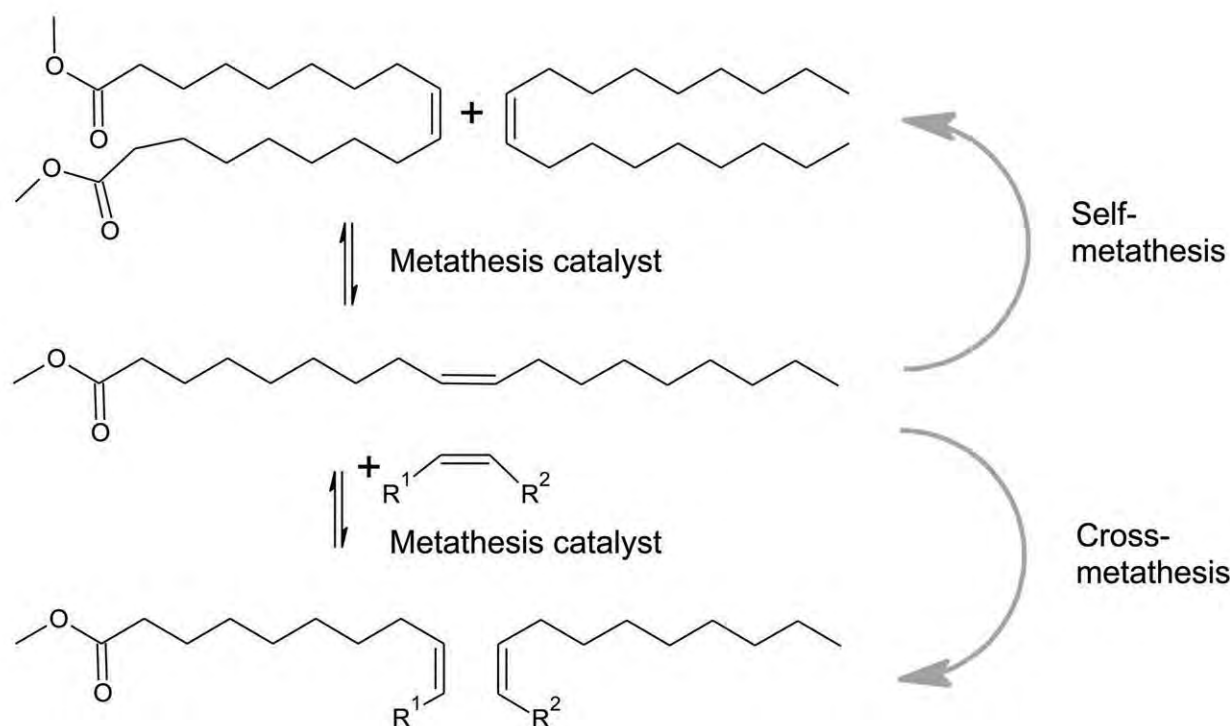


FIG. 2. Self- and cross-metathesis reactions of oleic acid methyl ester

Therefore, short alkenes are useful reaction partners for cross-metathesis reactions.  $\alpha$ -Olefins are easily obtained petrochemically and, in principle, the variation of  $\alpha$ -olefins can modify the boiling line significantly.

Thus, 1-hexene was used for first metathesis reactions with biodiesel. The product mixture showed a boiling range of about 230 °C. Table 1 (page 648) shows the main components of the metathesis reaction from rape seed oil methyl ester (RME) and 1-hexene, measured by gas chromatography-mass spectrometry (GC-MS). Next to the listed molecules, also substances with a molar mass higher than that of biodiesel were formed in this reaction. These products could not be measured by GC-MS, but were detected by size exclusion chromatography (SEC).

This metathesis reaction was carried out with different catalysts, different catalyst loadings, different equivalents of hexene, and under different reaction conditions. Finally, more than ten different metathesis fuels were generated. The boiling curves of these fuels were more or less comparable to the boiling curve of fossil diesel fuel. In Fig. 3 (page 648), the comparison of the boiling curves of biodiesel, fossil diesel fuel, and a metathesis fuel is shown.

Since metathesis fuels will probably not be available as neat fuels in the near future, M20 blends in diesel fuel were produced and tested (Fig. 4, page 649). From 10 different M20 metathesis blends, one was chosen for extended emission tests in a heavy-duty truck engine (Mercedes OM 904 LA, Euro IV) by a selection process that took into account: the boiling behavior, the biogenic content and the regulated emissions from a single-cylinder engine (Farymann 18W).

In addition to the regulated emissions (NO<sub>x</sub>, CO, HC and PM), the non-regulated exhaust gas components ammonia,

polycyclic aromatic hydrocarbons, aldehydes, as well as mutagenicity of the exhaust and the particle size distribution were tested as well. Fossil diesel fuel (DF), biodiesel from rape seed oil (RME) and a B20 blend of RME in DF were used in comparison with a metathesis blend (M20).

In operation with metathesis fuel blends, the emissions of the OM 904 LA only showed very slight deviations from B20. The nitrogen oxide emissions for RME were much higher than for DF, and also the B20 or metathesis blends showed a slight increase. The opposite effect was observed with particle mass, where the use of RME led to a reduction of 25%. However, this trend was not observed for the blends. Their particle masses lay

CONTINUED ON NEXT PAGE

- Biodiesel is presently the blend component predominantly used in diesel fuel.
- Unfortunately, the high boiling curve of fatty acid methyl esters (FAME) is not very well-suited for modern passenger car engines with diesel particle filters.
- Scientists in Germany have used metathesis to lower the boiling curve and conducted emissions tests of the resulting fuels in three different diesel engines.

**TABLE 1.** Reaction products of the cross-metathesis reaction of RME with 1-hexene

Product	Boiling temperature [°C]	Content [%]
5-Decene + 1-Decene	172	13.3
5-Undecene	192	2.7
5-Dodecene		0.9
5-Tetradecene	251	15.2
4-Decenoic acid methyl ester	215	18.1
9-Octadecene		2.5
Methyl myristoleate	306	31.8
Hexadecanoic acid methyl ester (C16:0)	332	6.3
9-Octadecenoic acid methyl ester (C18:1)	351	9.2

within the order of magnitude of DF at 0.01 g/kWh. A significant reduction in hydrocarbon and carbon monoxide emissions was also observed for RME. For the mixtures, a reduction was only found for HC emissions. One of the metathesis components exhibited slight advantages in terms of carbon monoxide emissions, since no increase in CO emissions, compared to DF, was determined here, as with the other blends. However, almost all regulated emissions lay within the Euro IV specification that applies to the used engine. All specifications were attained for three of the four fuels used. Only the nitrogen oxide emissions of RME slightly exceeded the threshold value of 3.5 g/kWh.

A small difference in particle size distribution was observed. Here, the metathesis fuels, if compared to the B20 blend, displayed a slight increase in particle count in the size range from 28 nm to 1000 nm. In the larger range from 1  $\mu\text{m}$  to 10  $\mu\text{m}$ , the values for B20 were substantially higher (Fig. 5). In case of the other tested non-regulated exhaust gas components, only small differences between the metathesis fuel blends and B20 were

observed. The carbonyls were also in the same order of magnitude and no significant differences could be discerned between the fuels used. With regard to mutagenicity, the use of a vanadium oxide SCR catalytic converter led to such low emissions of mutagenic substances that slight mutagenic tendencies in the emissions could only be measured with DF.

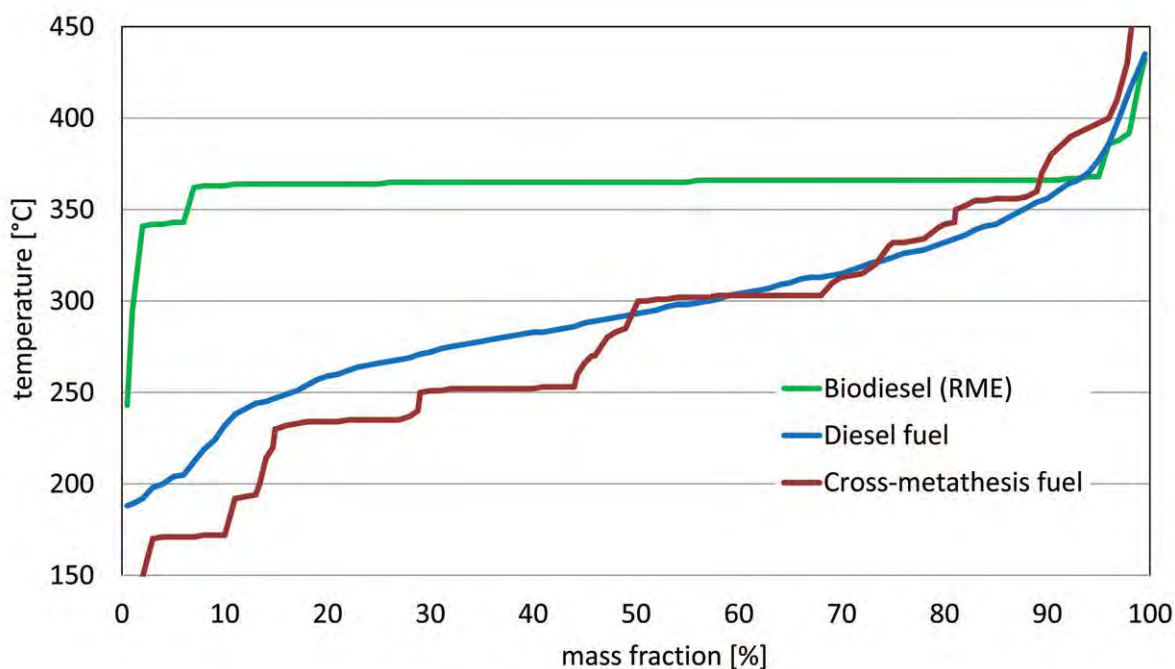
Further tests regarding the combustion behavior of the metathesis fuels in a special equipped AVL single-cylinder research engine based on the MAN D28 engine did not reveal any important differences with respect to the comparative fuels DF and RME, within the obtainable precision of the available measuring technology.

## CONCLUSION

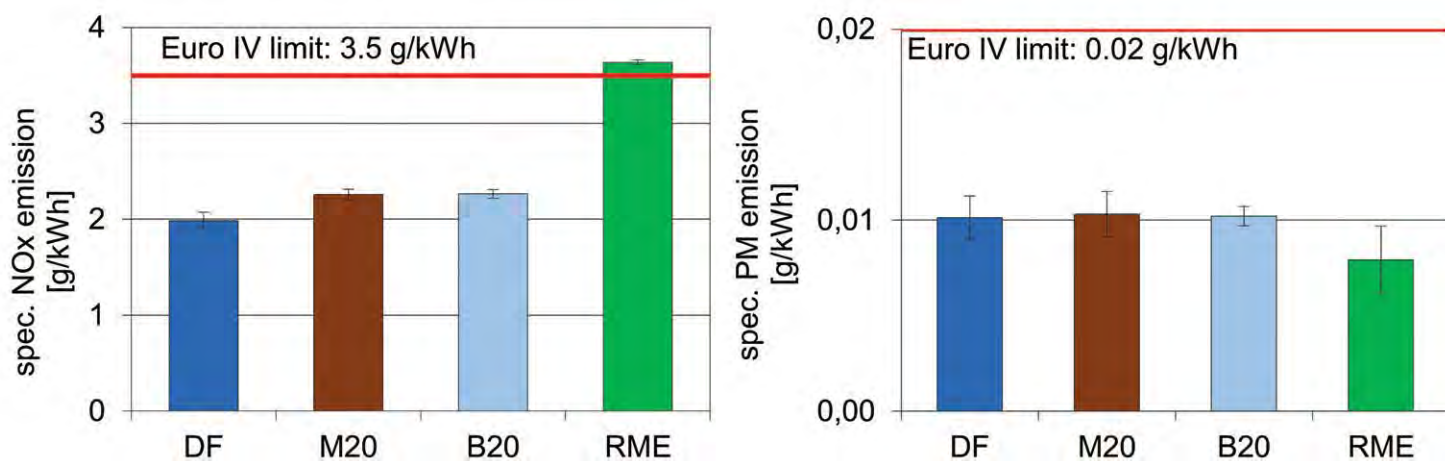
The boiling curve of biodiesel can be lowered using olefin metathesis. The resulting fuels were investigated intensively

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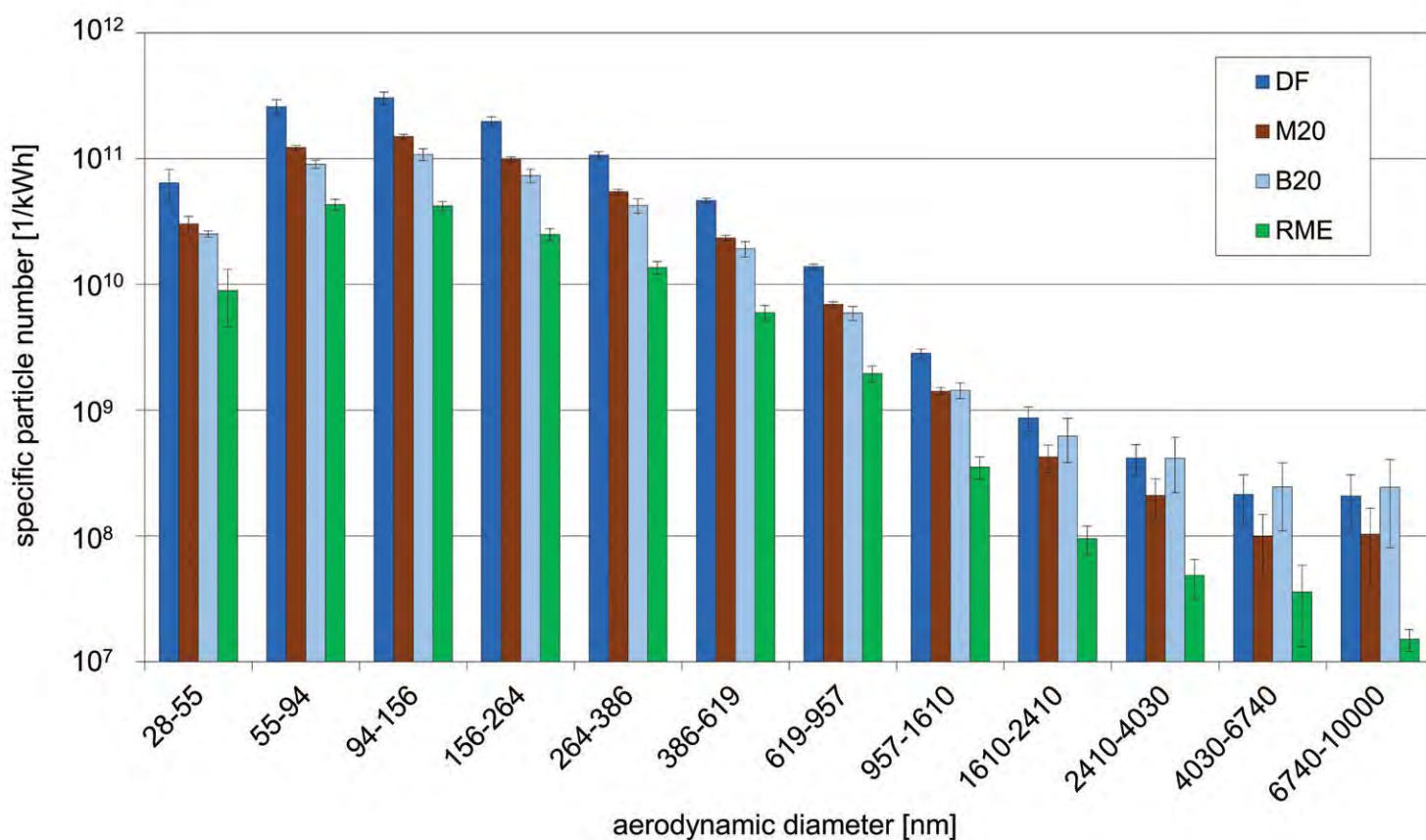
**FIG. 3.** Boiling curves of biodiesel, fossil diesel fuel and a cross-metathesis fuel  
Emission tests







**FIG. 4.** Emission of fossil diesel fuel, 20 % blends of both metathesis fuel and biodiesel in DF, and neat biodiesel, tested in an OM 904 engine with SCR catalyst (ETC test)



**FIG. 5.** Particle size distribution of fossil diesel fuel, 20 % blends of both metathesis fuel and biodiesel in DF, and neat biodiesel, tested in an OM 904 engine with SCR catalyst (ETC test)

# INFORMATION

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and tested in three different diesel engines with respect to their emissions.

Within the framework of the implemented tests, no evidence contradicting the suitability of the metathesis fuels for engine combustion was found.

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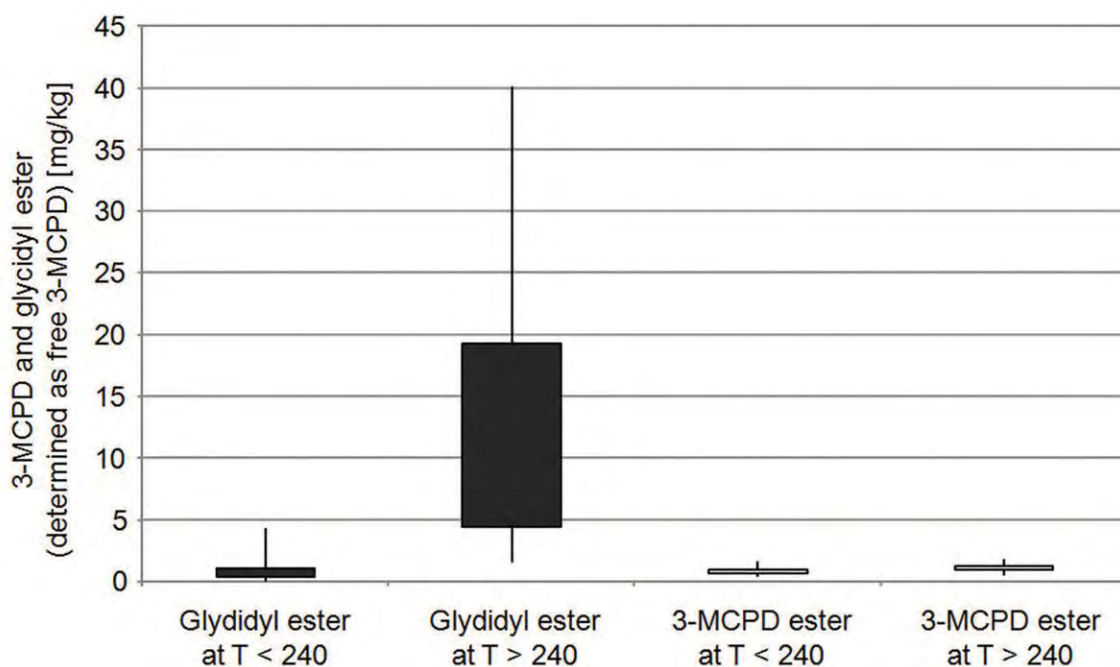
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**Fig. 1.** Influence of temperature on the formation of 3-MCPD-E and GE during deodorization of palm oil



# Approaches to mitigating esters of both 3-MCPD and glycidol

**Bertrand Matthäus and Frank Pudel**

- Esters of 2- and 3-monochloro-1,2-propanediol and glycidol in edible oils are a concern, because these compounds in their free state are classified as possibly and probably carcinogenic to humans.

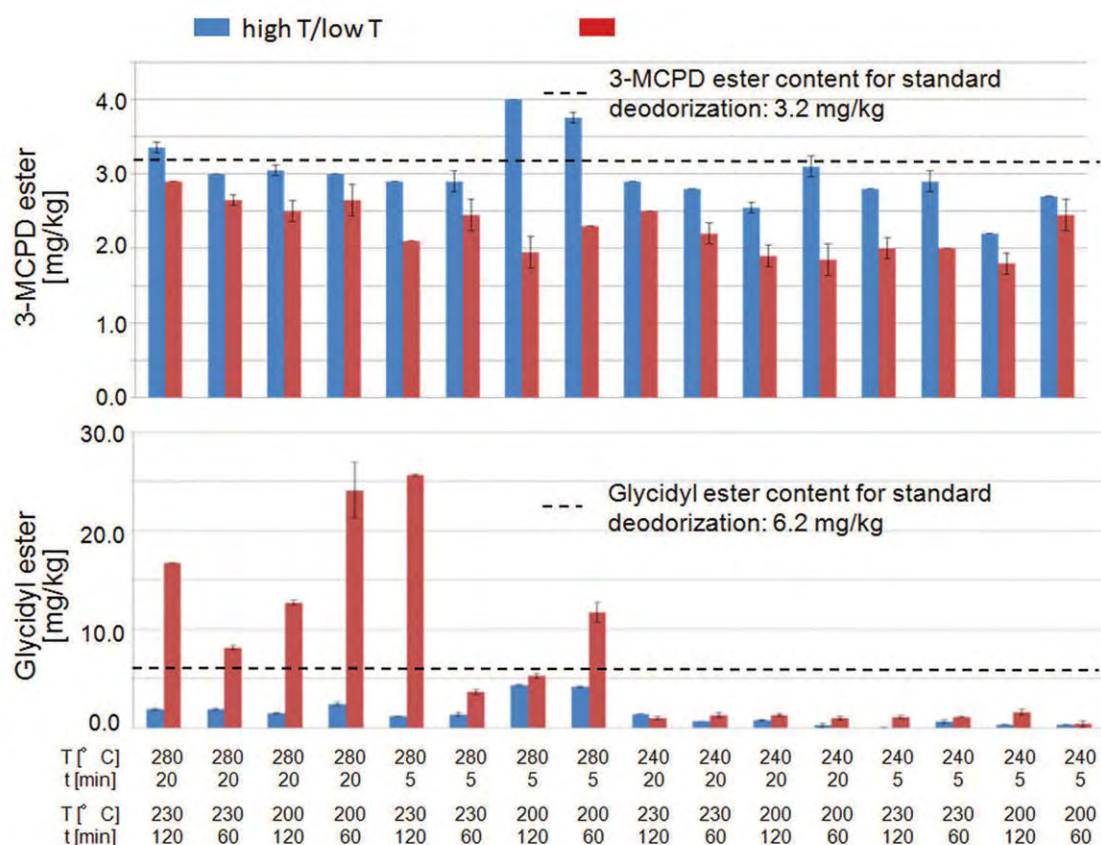
- Fortunately, there are many opportunities throughout the production chain to reduce the content of these esters.

- This article describes several strategies for mitigation—in raw materials, during refining, and in fully refined oils.

In 2006, fatty acid esters of 3-monochloro-1,2-propanediol (3-MCPD esters; 3-MCPD-E) were found in refined edible oils (1), and later high amounts of fatty acid esters of glycidol (glycidyl esters; GE) were described (2). Additionally, Seefelder *et al.* (3) and Kuhlmann (2) described the finding of 2-MCPD esters in refined oils.

The compounds are of great concern, since free 3-MCPD and glycidol are classified as “possibly” (group 2B) and “probably” (group 2A) carcinogenic, respectively, to humans by the International Agency for Research on Cancer (IARC) (4). For free 3-MCPD, a tolerable daily intake (TDI) of 2 µg/kg body weight per day was defined. Today, it is generally accepted that 80% to 100% of the esters are degraded to free compounds by enzymes in the human body, making the application of the IARC statement reasonable (5–7). Consequently, there is a need to develop





**Fig. 2.** Effect of different time/temperature conditions on the formation of 3-MCPD and GE during two step deodorization

alternative techniques for the processing of refined fats and oils to reduce the content of the esters (8).

Mitigating the formation of MCPD-E and GE is possible at several steps in the processing chain as edible oils make their way from raw materials to ready-to-eat products. Mitigations at various stages include reducing or preventing precursors in raw materials before processing and selecting suitable raw materials; changing the conditions of oil extraction—particularly the refining process—and introducing new steps to the process; and using suitable absorbent materials or enzymatic treatment to reduce esters in oils after they have been refined.

Most mitigation efforts have involved palm oil due to its strong ability to form esters and important role in food processing. However, many of the approaches outlined in this article also apply to the production of other edible oils.

## PRECURSORS

Ester formation requires the presence of certain precursors that are genetically defined in the plant, taken up by the plant, or formed during growing or processing. Processing conditions (temperature, time, and pH value) also have a strong influence on ester formation. The literature describes how triacylglycerols (TAGs), monoacylglycerols (MAGs) and diacylglycerols (DAGs), phospholipids, or glycerol can serve as precursors to the formation of free 3-MCPD (9–12) in the presence of chlorine-donating ions. Later, research showed that these precursors are also responsible for the formation of esters. Regarding the formation pathway, Rahn and Yaylayan (13) and Destailats *et al.* (14) explained that

**A full reference list is available in this issue's digital edition.**

3-MCPD-E are formed as a result of a nucleophilic attack of chloride ions on lipids via acyloxonium ion formation.

In practice, a poor correlation between the amount of DAGs in edible oils and the ability to form 3-MCPD-E and related compounds was found for different types of crude oils (15). On the other hand, different investigations showed that the capacity for ester formation increases significantly starting with a content of DAG > 4% in the oil (15, 16). This is a strong indication that a careful selection and handling of the raw materials, especially palm fruits, is necessary to avoid the formation of DAGs post-harvest. Higher amounts of 3-MCPD-E and related compounds have also been observed to result from higher amounts of free fatty acids and DAGs in other oils such as corn oil, coconut oil, and even low-quality olive oil.

It is logical to assume that differences in climate, soil, growth conditions, genotype, harvest technique, and processing will have a similarly strong influence on the ability of the crude oil to form 3-MCPD-E and related compounds, as these factors influence the amount of chlorine-containing compounds (such as inorganic or organic chlorine-containing compounds arising from salts in the soil or pesticides) and partial acylglycerols in the oil. In fact, remarkable differences in the ability of palm oils from different

CONTINUED ON NEXT PAGE

locations to form 3-MCPD-E and related compounds under standardized heating conditions were found (15).

A key factor in the development of mitigation strategies is knowing the source of the chlorine donor. Interestingly, the chloride content of the water used to generate strip steam during deodorization has no influence on ester formation (17), but it has become clear that the chlorine donor must be available in an oil-soluble form to enable the reaction with other precursors (15). Craft *et al.* (18) described a case in which lipophilic organochlorides were formed while fresh fruit bunches were being sterilized inside the oil mill. Hydrophilic compounds taken up from the environment had accumulated in the oil palm plant, resulting in a chlorine cascade. Sources of inorganic chloride are widespread. Examples include chloride-containing saline soils, ferric chloride used as coagulant in water treatment in Malaysia, potassium chloride as a cheap and widely used fertilizer, and ammonium chloride as a nitrogen source for oil palms. Additionally, within a sustainable plantation management system, empty fruit bunches and palm oil mill effluent are used as fertilizer, which can result in an enrichment of chloride ions in the plantation. Herbicides and insecticides are another possible source of organochlorides (19, 20).

- Some general recommendations for mitigation before refining include:
- harvesting immediately after detecting loose fruits;
- harvesting and loading carefully, and transporting fruits to the mill quickly;
- collecting loose and/or damaged fruits and processing them separately;

- inactivating triacylglycerol splitting enzymes quickly after harvest; and
- avoiding the use of potential chloride donors

## REFINING

Although refining contributes significantly to the development of esters, producing crude oil without refining is not a realistic mitigation strategy, as refining is essential to improve and optimize oil quality and stability after extraction. Consequently, it is necessary to optimize the refining process.

In the first step of the refining process, degumming, it seems advisable to avoid acid pH-values in the crude oil. Schurz (21) and Ramli *et al.* (22) showed that the use of acid during degumming could activate the formation of esters upon further processing.

Chemical neutralization has two positive effects on the mitigation of the esters: 1) the acidity of the oil before deodorization is reduced and 2) potential precursors can be removed from the oil when the neutralized oil is washed with water to eliminate the soapstock. Thus, a neutralization step is recommended before deodorization to avoid the lower pH values that result from higher contents of free fatty acids. From this point of view, chemical refining seems to be a better option for lower contents of 3-MCPD-E and related compounds than physical refining.

Bleaching before deodorization can strongly influence the pH value when neutral or acidic bleaching earths are used. Ramli *et al.* (22) recommended the use of natural or activated bleaching

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# Automatic laundry washing machine changes and impact on detergent formulation chemistry

Jim Pell and Phil Sliva

High efficiency (HE) appliances are gaining market share. While the newer washing machines have obvious differences in water volume, agitator style, and cycle time, our performance evaluations indicate that the new machines in North America dramatically improve soil and stain removal. On the other hand, we saw some compromise with respect to soil suspension and re-deposition. This new challenge opens up opportunities

for novel chemistry as well as other technologies that may have been discounted previously due to their low concentration in the wash water.

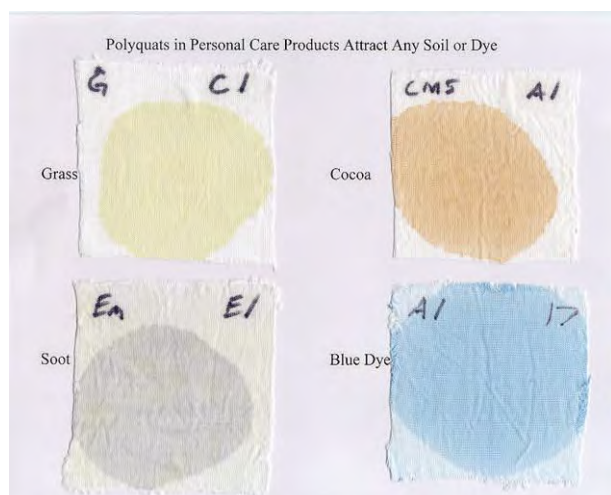
We were particularly interested in the local re-deposition caused by reduced water volume machines. We have seen this in European laundry, where low water volume washing machines have been the norm for decades. The cause of the phenomenon was accidental application of certain personal care products onto fabrics. These hair or skin care products were not particularly visible to the consumer after being applied to the fabric. However, if a personal care product contained a polyquaternary compound ("polyquat"), any part of the fabric where it was applied would develop a very visible stain during the wash process. An example of this phenomenon is presented in Fig. 1.

The polyquats attract soil and dyes from the wash water, forming a nearly permanent stain on the fabric. The lower water volumes of front-loading machines in Europe exacerbate the re-deposition potential of these products because the relative soil concentration is so high. Our testing of the newer HE machines shows that this localized stain phenomenon is likely to be a concern for many consumers in North America as well.

## HOW MACHINES WERE EVALUATED

We evaluated several types of machine for this comparison. For the North America region we used top-loading standard efficiency Kenmore 80 Series machines with an agitator stem as our control. The reduced water use washers were represented by LG TROMM WM2688HWM front-loading washers, an older generation Whirlpool Cabrio WTW6600SW, and a current generation Whirlpool Cabrio WTW8800YW1. Both of the Cabrio washers were HE top-loading machines with no agitator. Please see Fig. 2 for a comparison of the two types of top-loading washers.

Comparisons were also made between two types of European washers. The standard control for the European region was an older model Miele Novotronic W 1918, while the reduced



**FIG. 1.** These test swatches show how polyquats in personal care products will attract virtually any soil or dye from the wash water.





FIG. 2. A top-loading machine with (left) and without (right) an agitator

resource machine for the region was represented by a current generation Bosch WAE244S1GB.

A mix of cotton, cotton/polyester blend, and terry cloth fabric was used to make up standard 2.71 kg bundles. The bundles and washers were stripped using 25 g of a linear alkyl-sulfonate based stripping detergent and 580 g sodium hexametaphosphate in softened water for two cycles. To ensure as little carry over as possible, a third cycle was run using only softened water. These cycles were all run using the hottest wash water and the largest load settings on each machine. The bundles were then dried in an electric tumble dryer.

A set of soil swatches designed to evaluate both the various detergency performance attributes of a laundry detergent and the ability of a detergent to prevent soil re-deposition were used as part of the test. Included was a set of cotton swatches with hair mousse applied to evaluate polyquaternium aided re-deposition. One gram of a commercially available hair style product that contained Polyquaternium 11 and Polyquaternium 4 (Amway Satinique® brand) was applied to a 100% cotton cloth (style 493) and allowed to dry overnight. All of the cloth swatches were individually measured for reflectance before washing using a Hunter 45°/0° spectrophotometer with a D65 illuminate and 10° observer. These swatches were then attached to the terry cloth towels present in the bundles and washed in the machines being evaluated using a standard setting with Amway's bleach-free powder detergent. An ingredient listing in order of descending concentration can be reviewed at [www.amway.com](http://www.amway.com) on the Legacy of Clean® SA8® Laundry Detergent product page. In the case of the North American washers, the settings were warm water wash, medium load size with standard agitation, and a cool water rinse.

- High efficiency (HE) appliances are increasingly gaining market share.
- Recent evaluations show that the newer washing machines improve soil and stain removal but have more issues when it comes to soil suspension and re-deposition.
- Reduced water volumes open opportunities for new chemistry and technologies.

The European washers were set to a normal agitation cycle with a 40°C wash setting. Water was captured and weighed at the end of each wash and rinse cycle. Municipal water from Grand Rapids, Michigan, USA (approximately 120ppm as  $\text{CaCO}_3$ ) was used during all cycles of this evaluation.

When the wash and rinse cycles were complete, the laundered swatches were removed from the terry cloth hand towels, then dried and pressed flat using a photographic print dryer. Final measurements of reflectance were then made with the spectrophotometer. Detergency performance results were tabulated

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**TABLE 1.** Water used in each machine

	Wash Water Volume (L)	Rinse Water Volume (L)	Overall Water Volume (L)
Kenmore 80 series	64	64	128
Whirlpool Cabrio WTW8800YW1	38	18	56
LG TROMM WM2688HWM	13	38	51
Miele Novotronic W 1918	20	30	50
Bosch WAE244S1GB	9	25	34

**TABLE 2.** Cycle time for each machine

	Wash Cycle Time (min)	Rinse Cycle Time (min)	Overall Time (min)
Kenmore 80 series	14	20	34
Whirlpool Cabrio WTW8800YW1	29	23	52
LG TROMM WM2688HWM	29	23	52
Miele Novotronic W 1918	45	45	90
Bosch WAE244S1GB	33	31	64

and presented as the delta  $R_d$  portion of the  $R_d a_{Rd} b_{Rd}$  color scale. We find delta  $R_d$  to be an excellent indicator of soil removal and soil re-deposition by measuring how much “lighter” a soil swatch becomes and how much darker a clean cloth gets after washing.

## NORTH AMERICAN WASHERS

### WATER REDUCTION

When compared to the Kenmore 80 series, the HE machines used much less water. The LG TROMM WM2688HWM washers consumed 80% less water while washing and 60% less throughout an entire cycle. The Cabrio WTW8800YW1 showed a reduction in water use of 41% in the wash and 56% over an entire cycle. Unfortunately, water volume information was not available for the Cabrio WTW6600SW. Water used in each machine is shown in Table 1.

### CYCLE TIME INCREASES

All of the North American HE machines had an increase in cycle time when compared to the Kenmore 80 series washer. Both the LG TROMM WM2688HWM and the Whirlpool Cabrio WTW8800YW1 wash cycle times were 107% longer with a total cycle time 53% longer than the Kenmore 80 series washer. Unfortunately, cycle times were not available for the Cabrio WTW6600SW. Cycle time for each machine is shown in Table 2.

## DETERGENCY AND RE-DEPOSITION COMPARISON

There are large soil removal differences between these washers. All of the HE machines removed more soil than the Kenmore 80

series washer, with the LG TROMM WM2688HWM washer showing the largest increase at 72%. The performance of the Cabrio WTW6600SW washer showed a 7% increase, and the Whirlpool Cabrio WTW8800YW1 washer showed a 65% increase compared to the Kenmore 80 series. The Whirlpool Cabrio WTW8800YW1 also showed a 35% increase in soil removal when compared to the Cabrio WTW6600SW machines. Reflectance differences indicating overall detergency and soil removal are shown in Figure 3.

The newer HE machines show slightly more re-deposition values for the unsoiled re-deposition swatches. Swatches treated with the hair styling product containing polyquats show a large decrease in reflectance. In the North American washers tested, this culminates with a 217% lower delta  $R_d$  in the LG TROMM WM2688HWM indicating that these swatches are visibly darker/dingier in a single wash. The decrease in reflectance due to re-deposition of soil is shown in Figure 4.

## EUROPEAN WASHERS

### WATER REDUCTION

The Bosch WAE244S1GB washers tested also showed a reduction of water use when compared to Miele Novotronic W 1918 machines. The Bosch WAE244S1GB showed a 55% reduction in wash water and a 17% reduction in rinse water use. That equates to a total reduction of 32% across all cycles (Table 1).

### CYCLE TIME REDUCTION

Unlike the North American washers which increased in cycle time, the European washers actually showed a decrease in cycle

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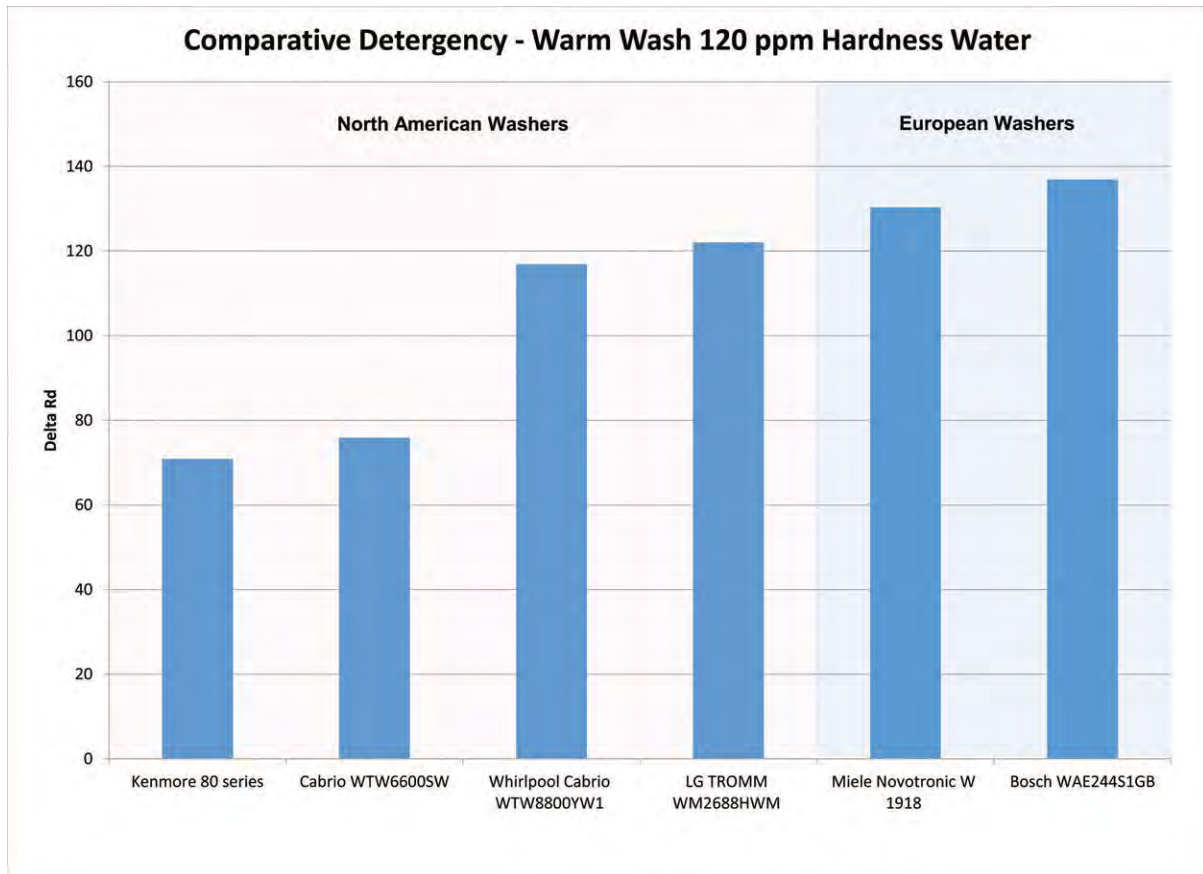


FIG. 3. Comparisons of detergency performance by North American and European washing machines

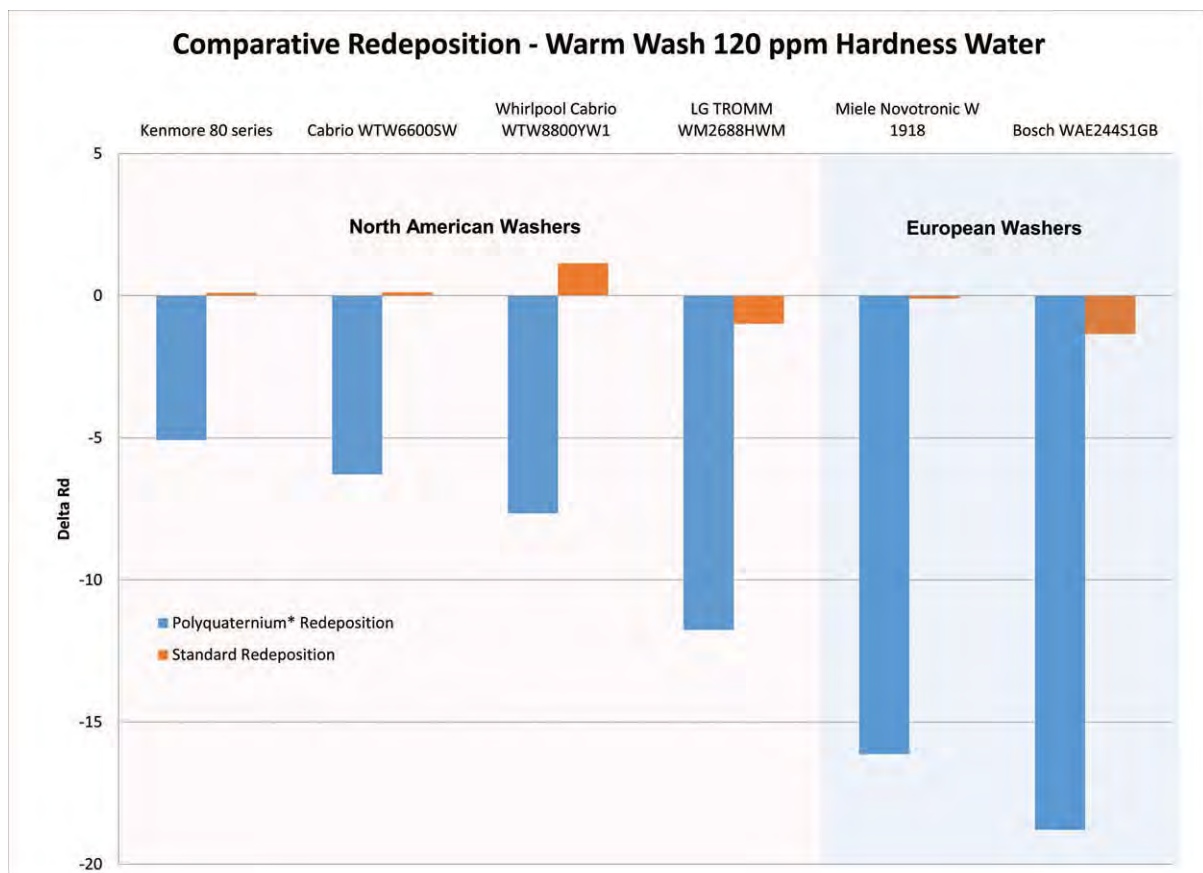


FIG. 4. Comparisons of soil re-deposition during washing in North American and European machines

time. When compared to the Miele Novotronic W 1918, the Bosch WAE244S1GB showed a reduction of 26% in wash time and 31% in rinse time for a total cycle time reduction of 29% (Table 2, page 658).

## DETERGENCY AND RE-DEPOSITION COMPARISON

The Bosch WAE244S1GB showed a minimal performance increase as measured by reflectance of 5% over the Miele Novotronic W 1918. However, a large difference can be seen in the increased localized re-deposition caused by polyquats as the reflectance of these swatches decreased by 17%. The untreated swatches also experienced a slight increase in re-deposition. Reflectance differences indicating overall detergency and soil removal are shown in Figure 3 (page 659). The decrease in reflectance due to re-deposition of soil is shown in Figure 4 (page 659).

## DISCUSSION AND OPPORTUNITIES

The continuing trend toward washing machines that use less water than their predecessors has and will bring about more challenges and opportunities in laundry detergent formulation.

First, with the reduction of water in the wash cycle and an increase in wash cycle time (North America only) the newer HE machines remove more soil from clothing, but they must also suspend it in less water. This is especially important if a fabric has been in contact with personal care products that contain polyquats. While this type of localized staining requires circumstances that are somewhat infrequent, the result is very visible to consumers. The personal care product must be applied directly to fabric and washed in a high soil wash load. Amway has had customers who experienced this phenomenon in Europe which prompted research and development of a patented solution to address the concern (Sliva, P. US Patent #631,310,031 B1, 2001).

Second, with the increase of detergent concentration in the wash liquor, certain opportunities and challenges arise. With wash water volumes of 9 liters in European washers and 13 liters in the North American front load washers, chelants and some polymers that were once considered cost prohibitive may now become

economically available for use. These materials may enhance water hardness control, bleachable soil removal, and the soil re-deposition properties of a given formula.

Increased concentration can also impact fabric and color safety. While the increased detergent concentration will make activated bleach systems more effective, it may also create fabric care concerns. In addition, dye transfer from colored clothing may also increase the importance of including color transfer inhibition agents to detergent formulations.

Finally, the newer HE machines in North America extract water much more effectively as they have greater spin rates than previous machines. The extraction cycle therefore creates a greater potential for pressing wrinkles into clothing.

Obviously, these significant changes in machine design will require more detailed evaluations. Meanwhile, laundry formulation chemists and their suppliers will be busy taking advantage of the new opportunities and challenges.

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*Jim Pell is a Business Intelligence Analyst at Amway where he has supported formulation research and implementation for over 17 years. His current focus is maintaining and gaining recognition for Amway products in the US EPA's Design for the Environment program. As a key member of the Amway Sustainability Expert Team, Jim monitors and advises formulators on competitive landscape and technology changes. Jim received his Bachelor of Science from Grand Valley State University. He can be contacted at [Jim\\_Pell@Amway.com](mailto:Jim_Pell@Amway.com).*

*Phil Sliva is a Principal Research Scientist and has developed home care consumer products for Amway for over 20 years. Phil leads Amway's eco-validation program for the Home Care line and is Co-Chair of the ACS Green Chemistry Institute Formulators' Roundtable. He is also a member of the American Cleaning Institute Sustainability Committee. Prior to joining Amway, Phil developed laundry and dish care products at the Procter & Gamble Company. He has 5 US patents and received his Bachelor of Science from the University of Detroit. He can be contacted at [Phil\\_Sliva@Amway.com](mailto:Phil_Sliva@Amway.com).*

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# How optimizing your tank and tote cleaning process can improve your bottom line

Andrew Delaney

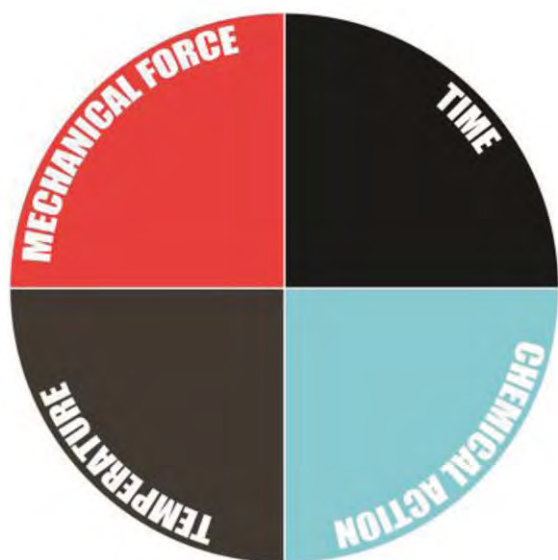


FIG. 1. Sinner's Circle: Four factors of cleaning

## FOUR FACTORS OF CLEANING

To understand how to optimize a cleaning process, one must first understand the basics of cleaning. Herbert Sinner (1900–1988), a chemical engineer for Henkel, first summarized the basic principles of cleaning in 1959. His summary, now referred to as The Sinner's Circle, describes the four factors that can be manipulated in any cleaning scenario: temperature, chemical action, time, and mechanical force.

When the effectiveness of any factor is increased, it will result in a decrease of one or multiple other factors. Washing dishes is an effective example of how the four factors interact. Hot water (temperature) is going to remove stuck-on food better than cold. Adding soap (chemical action) makes the process even easier, and you can either soak a dish overnight (time) or scrub the dish clean (mechanical force).

When cleaning tanks and other containers, it is imperative to examine the effectiveness and efficiency of your procedure. The Sinner's Circle (Fig. 1) can be easily applied to cleaning-in-place (CIP) as a way to compare processes. The most common cleaning practices are: wetting (static spray balls), rotary wetting (rotary spray heads), boiling out or fill and drain, manual cleaning, and rotary impingement cleaning (Fig. 2).

Boiling Out or Fill and Drain are the processes of soaking tanks with hot water and caustic for a period of time and then draining them. For one major chemical manufacturer, cleaning different chemicals tanks between batches was crucial to the quality of their product. Each clean required filling 6,000 (24,000 liters) to 21,000 gallon (80,000 liters) tanks with water and cleaning agents then bringing the water to a boil. Each wash required 4,000 gallons of water which added up to 1.5 million gallons (5.7 million liters) per year. This is an extremely excessive amount considering other cleaning methods are able to achieve the same results in just minutes by using a tenth or less of the water.

Manual cleaning is a very common method of tank cleaning; however, the method is beginning to lose credibility. Although

- Every processing company uses tanks or totes that must be cleaned between batches to ensure the reliable production of an uncontaminated, quality batch.
- Inefficient, ineffective, and/or outdated cleaning processes can result in unnecessary expenditures that can have a negative effect on the bottom line.
- This article explains how optimizing your cleaning-in-place (CIP) process can result in substantial cost reductions and revenue recovery.



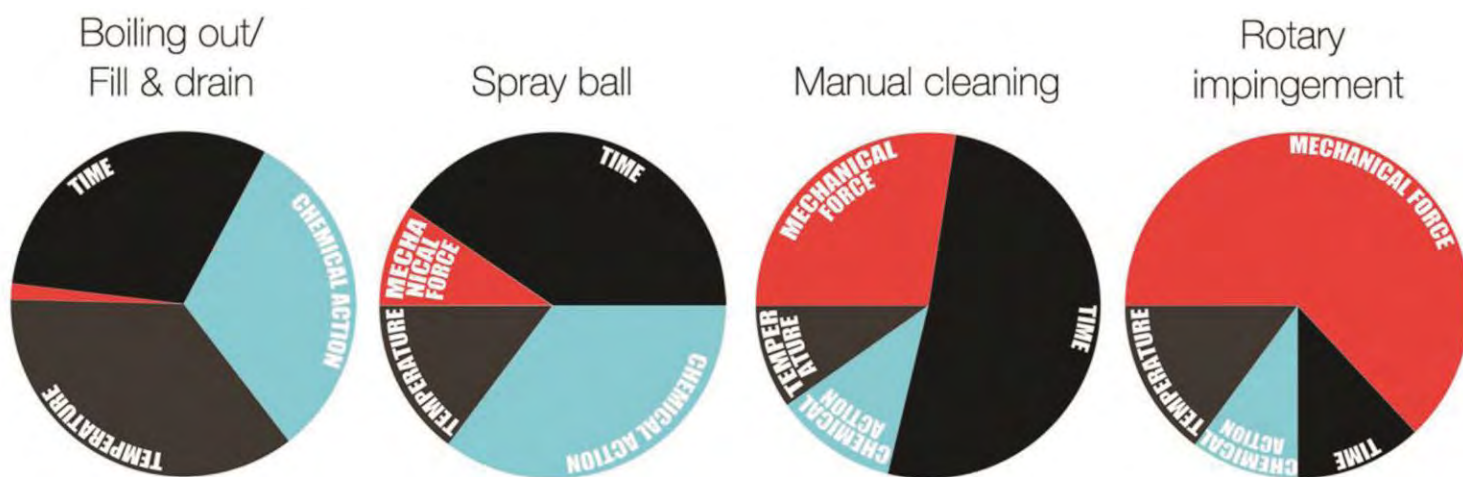


FIG. 2. Sinner's Circle for four selected cleaning processes

nearly every other process is automated, many companies still rely on manual cleaning as an effective way not only to clean but also to validate the cleaning process as well. Human error aside, no manual clean can ever be absolutely replicated. In addition, entry into confined spaces is risky and the potential damage to the tank is high.

Static spray balls and rotary spray heads are designed with nozzles or small orifices. The water and chemicals are typically split four or more ways depending on the manufacturer. These devices can be positioned inside a tank at any angle and can wet a tank quickly. For easy-to-clean residues such as powders or dry materials, spray balls and spray heads can be a suitable option. Opt for a rotary spray head if the cascading cleaning effect provides a sufficient clean. If quick dispersion is needed to wet or rinse a tank quickly or if water usage is not an issue, a spray ball may be a good option however, it is best to conduct an ROI before purchasing any tank cleaning device.

## OPTIMIZING YOUR CIP WITH ROTARY IMPINGEMENT

The goal is to use the least amount of resources while achieving the most effective tank clean. Delivering greater impact (mechanical force) to dirty tank walls will reduce the amount of resources (time, temperature, water, and chemicals) required to clean. Rotary impingement machines are designed to do just that. They combine pressure and flow to create high-impact cleaning jets. Cleaning occurs at the point where the concentrated stream impacts the surface. This impact and the resulting tangential force radiating from that point blast contaminants from the surface, scouring 100% of the tank interior. Distributing the impingement force in a precise, repeatable, and reliable 360° pattern—a full-coverage, indexing pattern—ensures the entire tank or vessel interior is cleaned, every time. This combination of impact and a controlled indexing manner results in an economic ideal, because impact is a one-time investment; water, chemicals, temperature, and time are continuous expenditures.

There are many real-life examples of savings through rotary impingement tank cleaning. One such example is a flavoring manufacturer who was using the manual cleaning method to clean hundreds of reservoirs ranging from 125–550 gallons (470–2100

liters). One of those tanks was an orange oil storage tank with one 2 inch (5 centimeter) opening and no usable drain-line. This particular tank had not been cleaned in over three years and the oil residue was hardened, making it an extremely difficult cleaning situation. The hard sediment was clogging the lines carrying the product to the production line. Typically, cleaning this tank by hand would take one or two days and involve scooping and shoveling the product out and using 750 gallons (280 liters) of water.

By utilizing the force of a rotary impingement cleaning device, the hardened oil was emulsified and easily sucked out with a customized vacuum extractor connected to the tank-cleaning device. With this method, cleaning times were reduced from 2 days to 4 minutes and water usage was reduced to 16 gallons (60 liters) from 750.

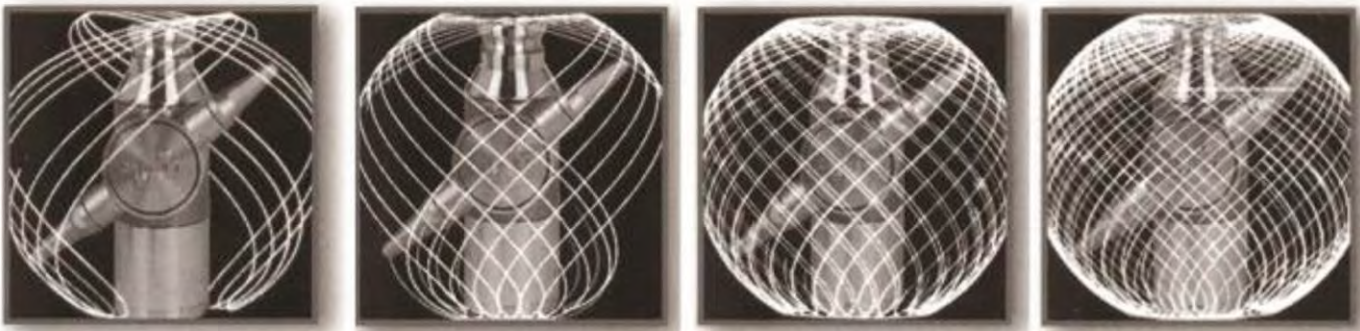
Rotary impingement machines are becoming recognizable across all processing industries. One such example is a Pennsylvania (USA) milk company using vertical spray dryers to produce large quantities of milk powder. With sizes of up to 22 feet (6.7 meters) in diameter and up to 60 feet (18 meters) high, the facility's spray dryers produce a total upward of 9,000 pounds (4,000 kilograms) of milk powder per hour.

Until recently, the company used traditional static spray balls and high-pressure water injection to clean the dryers. The routine cleaning cycle usually consisted of two 45-minute wash cycles utilizing a 3% cleaning agent followed by a 20–30 minute rinse. However, a thorough cleaning of the entire unit often required personnel to insert the spray ball up to 16 separate times into various openings throughout the dryer. This was a very time-consuming and labor-intensive operation to ensure that all the areas were cleaned. Water usage throughout the process was approximately 250 gallons (950 liters) per minute.

With a standard cleaning process already in hand to measure against, the company's manager of engineering and maintenance had the opportunity to install and use two rotary impingement cleaning machines.

A rotary impingement tank cleaner was inserted inside the large upper chamber and dome of the spray dryer, while a more compact machine was positioned in the small cone (also referred to as a "bustle") located at the bottom of the dryer. During the new

CONTINUED ON NEXT PAGE



Stages of a full rotary impingement machine wash cycle

## Rotary impingement tank-cleaning machines

Rotary impingement tank-cleaning machines maximize force while reducing water and chemical use and tank downtime. Two to four spray nozzles, rotating on a vertical and horizontal plane, deliver forceful water jets to tank interiors. Increasing the amount of force sent through the nozzles automatically decreases the amount of water, time, and chemicals needed to get an efficient and effective clean. The high-impact water jets blast away difficult-to-clean residues easily in just minutes. Other common methods may require manual cleaning and tank entry, excessive water usage, and multiple wash cycles leading to tank downtime and stalls in production.

Automated and fluid-driven, rotary impingement tank-cleaning machines are guaranteed to rotate in a complete 360° pattern, every time. Other benefits include their low-maintenance schedule and resistance to clogging or plugging.

These devices are suitable solutions for optimizing tank CIP when companies need to reach sustainability goals, boost productivity, avoid cross-contamination of products, achieve a higher level of clean, and meet US Occupational Safety and Health Administration (OSHA) standards for worker safety.

cleaning cycle, each rotary impingement machine was inserted only once, resulting in immediate savings in time and manpower.

With the new units, wash times were reduced to one 15–30 minute cycle. Additionally, water usage was estimated to be about 100 gallons (380 liters) per minute. “We estimate that by using the two [rotary impingement] units, we save about two and a half hours in time and around 7,500 gallons of rinse water per cleanup,” the manager said. “And, we also save on the amount of chemicals we use.”

Another example is a food company that needed to regularly clean 12 process vessels with burnt-on sauce residue. It was using static spray balls to clean all four production lines with three tanks each. Cleaning time was 2.5 hours per tank. The company estimated its tank downtime to be 3,650 hours per year. Its water usage was more than 3.5 million gallons (13 million liters) per year. It was producing 1,460 batches per year.

The company decided to purchase a rotary impingement device, operating at 15 gallons (57 liters) per minute. The cleaning process with the new device includes a 2 minute pre-rinse, 5 minute recirculated wash, and a final 2 minute rinse, for a total of 9 minutes. The tank downtime is now 362 hours per year, down from over 3,650. The company is now able to create more than 2,400 batches per year, an increase of more than 950 batches.

The company reports it is using 85% less water with the rotary impingement machine and has saved 90% in time spent cleaning. The reduction in tank downtime allows for 65% more batches to be produced.

### TOTE CLEANING WITH ROTARY IMPINGEMENT

As the most widely used container for storing and transporting materials, totes that are improperly and inefficiently cleaned can

have direct repercussions to the overall productivity of a facility. Rotary impingement tote cleaners contribute to substantial improvements in productivity and reductions in cost. Companies no longer have to waste time and money outsourcing cleaning or be charged for dirty returns. Cleaning times for 1,000 liter totes are 2–7 minutes with a rotary impingement machine. Cleaning can be done in-house, effectively and efficiently using the least amount of resources including time, energy, manual cleaning, chemicals, and water as illustrated by a chemical company who uses totes for shipping its products.

After the shipments have been delivered, the totes are recycled for re-use. Many of the totes for receiving raw materials from suppliers are also recycled for re-use at the company. The director of operations was frustrated by the time, inconsistency, and costs of sending dirty totes to the company’s tote supplier for refurbishing/cleaning. He also had to ensure that re-used totes were 100% clean to avoid the dangers of cross-contamination between product shipments. Oftentimes, if the manual cleaning was done in-house, the results were less than perfect.

He decided to purchase a rotary impingement machine to see if it could reclaim the costs of outsourcing. In order to accomplish this, the device had to reduce the time spent cleaning totes in-house and increase the level of in-house cleaning effectiveness. The rotary impingement machine accomplished these goals and paid for itself in one week. As a result, the company spent 50% less time cleaning while eliminating outsourcing and achieving a consistent clean.

### MAKING INFORMED DECISIONS

Rotary impingement machines are not the answer for all tank-cleaning applications, though. For example, when residues are relatively easy to clean, such as powders, rotary impingement



machines can be overkill to the budget, and the water jet impact exceeds the mechanical force need to achieve effective cleaning. Whereas a spray ball or rotary spray head typically costs under \$1,000, rotary impingement machines are generally \$3,000–\$5,000. Just as with any purchase, an ROI (return on investment) calculation should be conducted to determine the payback of a rotary impingement machine vs. a rotary spray head and a static spray ball. If the payback on a rotary impingement is more than five years after installation, then opt for a rotary spray head or spray ball. An easier test is to ask yourself a simple question: Is this spray ball getting my tanks clean quickly and effectively? If the answer is yes, then stick with the spray ball. If not, then it may be time to upgrade to the higher-impact water jet offered by rotary impingement cleaning.

If quick dispersion is needed to coat a tank quickly with a chemical or disinfectant, rotary impingement machines are not the answer. At this time they do not have the capability for quick dispersion.

Some of the largest savings companies have experienced through rotary impingement cleaning are in the reduction of water usage. In some geographic areas, such as on the coast or in offshore locations, water is in abundance and therefore limiting its use is not considered a top priority. In that case, a rotary impingement machine may not offer the substantial savings that other companies have experienced.

It's also important to note that some rotary impingement machines can be damaged if operated at a pressure higher than what is recommended by the manufacturer.

## A UNIQUE SOLUTION FOR EVERY APPLICATION

Keep in mind that not all rotary cleaners are created equal. First, each device is built for a company's specific goals and applications. Tank size, internal obstructions, and residue are considered when selecting and sizing the right machine for the application. Second, in order to experience a quick ROI, the tank-cleaning machine must be built to last. There are many "off-the-shelf" rotary cleaning devices on the market today that easily clog, break down, and have high repair costs. The results are inefficient cleaning and massive tank downtime. To make the most out of your CIP, your tank-cleaning machine should be durable. Furthermore, maintenance and necessary repairs should be quick, easy, and affordable.

Rotary impingement tank-cleaning machines are designed with cutting-edge technology by experienced engineers whose goal is to save companies' resources and time. When tank and tote cleaning is done properly, it can result in the recovery of millions of hours in time and billions of gallons of water. Audit your CIP and see what you can do to make your sustainability goals a reality. The right rotary impingement machine can enable significant results, directly impacting the bottom line.

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*Andrew Delaney is president of Alfa Laval Tank Equipment Inc. He can be contacted at [andrew.delaney@alfalaval.com](mailto:andrew.delaney@alfalaval.com).*

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# PATENTS

## Charged lipoprotein complexes and their uses

Dasseux, J.-L., US8617615, December 31, 2013

The present disclosure provides charged lipoprotein complexes that include as one component a negatively charged phospholipid that is expected to impart the complexes with improved therapeutic properties.

## Catalyst for transesterification process

Banavali, R., and J. Trejo, Rohm and Haas Co., US8685881, April 1, 2014

The present invention relates to a method for producing esters from triglycerides by using solid heterogeneous catalysts comprised of calcined calcium carbonate, particularly for obtaining biodiesel.

## Polyurethanes made from hydroxy-methyl containing fatty acids or alkyl esters of such fatty acids

Van der Wal, H.R., and C.F. Camiel, Dow Global Technologies LLC, US8686057, April 1, 2014

Polyurethane polymers are made from a reaction mixture that contains a polyisocyanate, a hydroxymethyl-containing fatty acid or ester, and another polyol, polyamine, or aminoalcohol. The carboxylic acid or ester group on the hydroxymethyl-containing fatty acid or ester is capable of engaging in a variety of reactions with the polyisocyanate and/or amine or hydroxyl groups present in the reaction mixture. This allows for good-quality, high molecular weight polymers to be produced even though the hydroxymethyl-containing fatty acid or ester tends to be a low functionality material.

## Process for preparing diesel fuels using vegetable oils or fatty acid derivatives

Hanks, P.L., *et al.*, ExxonMobil Research and Engineering Co., US8686203, April 1, 2014

A process for preparing fuels, such as diesel fuels or jet fuels, by hydrotreating vegetable oils or fatty acid derivatives that may be applied to existing equipment for treating fossil fuels.

The process comprises feeding hydrotreating a combined oxygenate feed stream, such as fatty acid methyl esters, and a hydrocarbon feed stream until not more than 86 wt% of the esters in the oxygenate feed stream are converted to hydrocarbons, and optionally further hydrotreating the product stream within at least a second hydrotreatment reaction zone until at least 90 wt% of the esters in the oxygenate feed stream are converted to hydrocarbons, before removing and separating a hydrocarbon stream suitable for use as fuel.

## Detergent for metal

Maeyama, S., *et al.*, Lion Corp., US8685912, April 1, 2014

A detergent composition for metal according to the present invention contains a specific nonionic surfactant, a specific nitrogen-containing organic compound, a specific carboxylic acid or salt thereof, and a specific alkanol amine, and has a pH of at least 9 that is measured at 25°C when the detergent composition is diluted to 1% by mass of an aqueous solution. According to the present invention, the detergent composition for metal having excellent corrosion inhibition properties, excellent effluent treatment properties, excellent foaming suppressing properties, and excellent liquid stability in addition to high detergent properties for metal can be provided.

## In-shower lotion compositions comprising up to 10% free fatty acids wherein ratio of unsaturated to saturated fatty acids is at least 1:1

Patel, R., and R.M. Paredes, Conopco, Inc., US8691197, April 8, 2014

This invention relates to rinse-off skin-conditioning compositions comprising relatively low amounts of oil/emollient and relatively low amounts of aqueous phase stabilizer/structurant. Use of unsaturated fatty acid (or at least minimum amount of unsaturated fatty acid as percentage of total fatty acid) has been found to result in unexpectedly high hydration. In a second embodiment, use of branched fatty acid (at minimum amount branched as percentage of total) also results in superior hydration. Also mixtures of unsaturated and branched fatty acids can be used. In a second embodiment, the invention relates to method of enhancing hydration using compositions as noted.

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





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# EXTRACTS & DISTILLATES

## Characterization of the liposoluble fraction of common wheat (*Triticum aestivum*) and spelt (*T. aestivum* ssp. *spelta*) flours using multivariate analysis

Bodroža-Solarov, M., et al., *J. Sci. Food Agric.* 94: 2613–2617, 2014, <http://dx.doi.org/10.1002/jsfa.6655>.

In the present paper, a special method for derivatization of liposoluble extract of common wheat and spelt flours was employed which enables simultaneous detection of fatty acid and non-saponifiable lipid fractions. Gas chromatographic–mass spectrometric analytical data for both fractions were separately analyzed by multivariate statistical techniques to model classes of different common wheat and spelt cultivars. Cluster analysis was used, and the results obtained revealed that better discrimination of samples was achieved by analyzing the peak area after 16 min retention time (non-saponifiable lipids), rather than commonly used peak area between 12 and 16 min (fatty acid fraction), due to more distinctive positions of points in factor space, even though the distances between points for fatty acid fraction (12–16 min) were greater. Similar results were obtained by principal components analysis, where all wheat points almost coincided whereas

spelt showed good discrimination. Comparison of chromatogram areas for non-saponifiable lipid fraction between common and spelt wheat showed a statistically high difference and hence has a potential for use in authenticity control.

## Matrix liposomes: A solid liposomal formulation for oral administration

Pantze, S.F., et al., *Eur. J. Lipid Sci. Technol.* 116: 1145–1154, 2014, <http://dx.doi.org/10.1002/ejlt.201300409>.

Liposomes can be used as oral dosage form to improve the bioavailability of both hydrophilic drugs, such as peptides and proteins and lipophilic drugs. But liposome dispersions are not very stable under the harsh conditions of the GI-tract. Also a controlled release of embedded compounds is not possible. Therefore we developed a lipid based liposomal carrier system for peroral delivery of peptides. The lipid bilayer of standard egg-PC/cholesterol liposomes were stabilized by gelatine. The gelatine also works as a thickening agent by forming a matrix in which liposomes are embedded. Liposomes were prepared by dual asymmetric centrifugation with the addition of gelatine. Size and size distribution of prepared liposomes with up to 20% gelatine achieved the same quality compared to conventional liposomes. Size and size distribution, encapsulation efficiency of different compounds and the dissolution behaviour of the solid dosage form in simulated intestinal fluid were examined. Matrix liposomes containing 20% gelatine showed an approximately linear release of liposomes. Moreover, the dissolution rate could be adjusted by modification of the gelatine concentration and the absolute amount of liposomes released at a certain time was found to be dose dependent.

*More Extracts & Distillates can be found in this issue's supplement (digital and mobile editions only).*

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JOURNAL OF THE AMERICAN OIL CHEMISTS' SOCIETY

## Journal of the American Oil Chemists' Society (October)

- Evaluation of the performance of a portable mid-infrared analyzer for the rapid determination of total trans fat in fast food, Mossoba, M.M., *et al.*
- Chemistry of color fixation in crude, physically refined and chemically refined rice bran oils upon heating, Rekha, B., B.R. Lokesh, and A.G. Gopala Krishna
- Rapid determination of olive oil chlorophylls and carotenoids by using visible spectroscopy, Cayuela, J.A., K. Yousfi, M. Carmen Martínez, and J.M. García
- Multi-length-scale elucidation of kinetic and symmetry effects on the behavior of stearic and oleic TAG. II: OSO and SOO, Baker, M.R., L. Bouzidi, N. Garti, and S.S. Narine
- Identification of aromatic fatty acids in butter fat, Schröder, M., H. Abdurahman, T. Ruoff, K. Lehnert, and W. Vetter
- Enzymatically modified fats based on mutton tallow and rapeseed oil suitable for fatty emulsions, Kowalska, M., A. Żbikowska, and B. Kowalski
- A comprehensive evaluation of the density of neat fatty acids and esters, Knothe, G. and K.R. Steidley
- Comparison of seed oil composition of *Nigella sativa* L. and *N. damascena* L. during seed maturation stages, Telci, I., *et al.*
- Selection for some olive oil quality components through the analysis of fruit flesh, Velasco, L., Á. Fernández-Cuesta, R. De la Rosa, M. Victoria Ruiz-Méndez, and L. León
- Oil content and composition of sesame (*Sesamum indicum* L.) genotypes as affected by irrigation regimes, Kadkhodaie, A., J. Razmjoo, M. Zahedi, and M. Pessarakli
- Food additives reducing volatility of antioxidants at frying temperature, Hwang, H.-S. and J.K. Winkler-Moser
- Effect of acid value on TBHQ and BHT losses in heating oils: identification of the esterification products of TBHQ and free fatty acids, Li, J., *et al.*
- Evaluation of the phenolic content and antioxidant activity of different seed and nut cakes from the edible oil industry, Sarkis, J.R. A.P.F. Côrrea, I. Michel, A. Brandeli, I.C. Tessaro, and L.D.F. Marczak
- Structure and physical properties of organogels developed by sitosterol and lecithin with sunflower oil, Han, L., *et al.*
- Thermal stability of rapeseed oil fortified with unsaturated fatty acid sterol esters, *et al.*
- Experimental design applied for cost and efficiency of antioxidants in biodiesel, Orives, J.R., D. Galvan, J.L. Pereira, R.L. Coppo, D. Borsato
- Plasma Polymerization Modified polyvinylidene fluoride (PVDF) membrane development and characterization for degumming of soybean oil, Onal-Ulusoy, B., E. Tur, E. Akdoğan, and M. Mutlu
- Quantitation of metals during the extraction of virgin olive oil from olives using ICP-MS after microwave-assisted acid digestion, Llorent-Martínez, E.J., M.L. Fernández-de Córdova, P. Ortega-Barrales, and A. Ruiz-Medina



## Lipids (October)

- Dietary hydoxycholeic acid exerts hypolipidemic effects by reducing farnesoid X receptor antagonist bile acids in mouse enterohepatic tissues, Watanabe, S. and K. Fujita
- High dietary cholesterol masks Type 2 diabetes-induced osteopenia and changes in bone microstructure in rats. Lapmanee, S., *et al.*
- Inhibition of endothelial lipase activity by sphingomyelin in the lipoproteins, Yang P., *et al.*
- Longitudinal evolution of the concentration of gangliosides GM3 and GD3 in human milk, Giuffrida, F., I.M. Elmelegy, S.K. Thakkar, C. Marmet, and F. Destailhats
- Fatty acid profiles of commercially available finfish filets in the United States, Cladis, D.P., A.C. Kleiner, H.H. Freiser, and C.R. Santerre
- Expression of genes controlling unsaturated fatty acids biosynthesis and oil deposition in developing seeds of Sacha Inchi (*Plukenetia volubilis* L.), Wang, X. and A. Liu
- Fatty acid-specific alterations in leptin, PPARα, and CPT-1 gene expression in the Rainbow trout, Coccia E., *et al.*
- Single nucleotide polymorphism scanning and expression of the pig PPARGC1A gene in different breeds, Li, Q., *et al.*
- EPA and DHA status of South Asian and white Canadians living in the National Capital Region of Canada, Nagasaka, R., *et al.*
- Analysis of fluorescent ceramide and sphingomyelin analogs: a novel approach for *in vivo* monitoring of sphingomyelin synthase activity, Huang T., *et al.*

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# Professional Pathways



## Why did you join AOCS?

During my first few months at Solex, I became aware of AOCS as the primary platform for the oilseeds industry. I found the organization to be a great source of the technical- and market- related information I was seeking at the time, and I continue to use its resources to stay current and to catch up on latest news and information.

I have since found AOCS to be a global meeting ground for professionals in the vegetable oil industry. For any person or company stepping into this industry, AOCS provides the perfect mix of knowledge and networking. I joined AOCS in 2008, and since then have attended regular meetings and presented at many conferences. The articles and papers I have written or presented have reached the target audience and led to many fruitful contacts and developments.

## Describe your career path.

After graduating as a chemical engineer in 2014, I joined DCM Shriram Consolidated Ltd., a chemical and fertilizer company, in Kota, Rajasthan, India. I worked as an assistant engineer in the polyvinyl chloride (PVC) plant, where I supervised the daily operations of various plant sections and worked on special environmental projects related to effluent treatment and process improvements. After two years of plant experience I moved to the head office in Delhi to join the newly formed group for business development and strategic planning. I spent a year working on technical and commercial feasibility studies on expansion and diversification projects. Since then, business development has become my vocation. I arrived in Canada in 1998, and joined Alfa Laval in Toronto. I worked as an application specialist in the thermal division, where I introduced new products to the oil and gas industry in West Canada. I changed positions and managed Alfa Laval's environment division in Canada, where I focused on water and waste water thickening and dewatering processes. My next stop with Alfa Laval was the oil and gas market for drilling solids control and oil sands applications for centrifugal separation. In 2008, I joined Solex as vice president of product development. Solex is a leader in the thermal processing of bulk solids in a wide range of industries. With new applications and challenges to be solved every day, I stepped into the company to provide focus and growth in new markets and applications. For the past six years now, I have been immersed in the oilseeds industry worldwide, working to introduce plate technology for indirect heat transfer.

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

*Farah Skold is a chemical engineer specializing in business development. She has worked in technical sales for various industries, including those related to oil and gas, water and wastewater treatment, fertilizers and chemicals, and most recently oilseeds and grains. Skold specializes in industrial applications for centrifugal separation and indirect heat transfer with plate heat exchangers—particularly with respect to the development and market launch of new products and products and applications that help processors achieve higher energy efficiency.*



## What do you love about your job?

What I find most fulfilling is the opportunity I get to meet and work with so many interesting people. The vast diversity of backgrounds, nationalities, experiences, and interests of these professionals is a constant source of stimulation for me.

There is a world of knowledge and experience that people carry within them, and I have the chance to explore it. My job provides me with an opportunity to collaborate with many exciting people, and the quest to reach common goals for process improvement or energy efficiency is always exciting.

## How do you see the industry changing in the next five years?

The trend toward finding energy efficient process improvements is going to continue. I expect and hope that we will see more collaborative work and a sharing of ideas and technical knowhow that will allow the industry as a whole to continually improve and bridge the gap between the levels of efficiency and productivity in different regions. Ever-increasing energy costs will drive all industries to find process and equipment improvements that will help reduce energy consumption.



## Describe memorable job experiences.

There are so many memorable experiences that it is hard to pick! Over the course of my career, I have had the opportunity to visit plants in a wide range of industries. Every time I attend the commissioning of a new piece of equipment, the experience is memorable and exciting, as it usually marks the culmination of many months of discussions, meetings, and design reviews involving a team of people spanning over many functions and sometimes even continents. I like to savor these rewarding moments and recognize them as proof of the strength of good team work. However, I will mention one particular experience that I had as a newly graduated engineer in the PVC plant. There, I witnessed my first major plant shutdown during the month of June in the sweltering heat of an Indian summer. Despite long hours and the heat, I recall feeling like a kid in a candy shop. Every piece of equipment—from distillation columns, to reactors to re-boilers and dryers—was stripped down and opened for cleaning and service. It was my first opportunity to see and study the equipment which until then I had only seen as diagrams in text books, in a few lab scale units, or from the outside while they were in operation. It was truly an exciting transition from books to real life.

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

There have been many people who have helped shape my career at every stage. I was influenced by many senior managers at DCM Shriram in India, whose good sense for business and capability to adapt to ever-changing market scenarios made a deep impression on me. Upon my arrival in Canada, I was influenced by my manager, Lars Henrikson, and his approach to running a Sales Organization with a balance of efficiency and innovation; Lars taught me not to accept mediocrity in my own work.

In the oilseeds industry, I have been fortunate to have worked with the likes of Tim Kemper and Marc Kellens at Desmet Ball-estra, and to have experienced firsthand the passion and sheer pleasure and pride they take in their work.

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

In this day and age of social networks and online interactions, it is easy to forget the importance of good old-fashioned human contact. All businesses and careers are based on personal interactions. Attending industry events, volunteer work, submitting papers and posters, and so on, are excellent opportunities to expand your network. A curious mind and a sense of humor are a great combination for fostering long-lasting connections.

## If you were starting your career again, what would you do differently?

I have no regrets when I look back at the career path I have taken. I can hardly claim that any of it was pre-planned. The important thing is to recognize an opportunity and grab it with courage and conviction.

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

The opportunities for advancement are always there, because the industry has an ever increasing demand for technical expertise and experience. I believe that hands-on experience at the plant level is critical for any technology or equipment solution provider. Consequently, a good understanding of process and challenges at the production level is essential for any professional who is considering a career that involves providing technical solutions to the industry.

## How would you describe the culture in your field, and how has it developed?

The plate heat exchanger industry has evolved rapidly in the past decade. The culture is fast-paced with new product launches and innovative technologies appearing regularly. Industries or applications with more mature markets, such as centrifugal separation, have changed at a much slower pace. Although innovations in such industries are few and far between, they still place a heavy emphasis on technical acumen and experience and are very welcoming to younger talent.

I have found that there is a constant demand and need for innovation. The attitude is cautious, but there is always a few daring pioneers who look beyond the existing standard and others are usually quick to follow.

## In your area/field and considering today's market, is it more important to be well rounded or a specialist?

I would consider myself a strong advocate for being a well-rounded professional. Being well-rounded in a wide range of fields is a foundation to becoming an expert in a narrower field of expertise. So, I would definitely start by getting a broad range of experience and then find an area of expertise that compliments that breadth of knowledge.

## What is your opinion toward the value of obtaining or possessing a graduate degree during a challenging economy?

Obtaining a graduate degree is an exercise in learning scientific thought and good problem-solving techniques. Being able to organize one's thoughts in a logical pattern and follow an idea through to a meaningful conclusion—and being able to communicate this to others—are just a few of the skills one can develop during one's graduate studies. Besides, the social and practical experience is rewarding in itself.

During a challenging economy, it may be disheartening in the beginning to face a lack of opportunities and the inability to distinguish oneself from the crowd. However, in my opinion, a degree is an essential tool to opening new doors. ■

## 3-MCPD (cont. from page 654)

earths with a more neutral pH value, as he found that the formation of 3-MCPD-E after deodorization increased with the acidity of the bleaching earth.

Since the formation of 3-MCPD-E and GE is the result of a heat-induced reaction, deodorization time and temperature have the strongest effect on ester formation (Fig. 1, page 652). GE show a continuous increase with increasing temperature. However, once a critical temperature is reached, 3-MCPD-E formation appears to follow a reaction that is completed in the early stage of the heating period and requires only minimal heating ( $>180^{\circ}\text{C}$ ) (23). Destailats *et al.* (24) showed that the critical temperature for the formation of GE from DAG is approximately  $200^{\circ}\text{C}$ ; above this temperature, the formation accelerates rapidly with increasing temperature. These different temperatures for the formation of the esters indicate on the one hand that the particularly high temperatures of physical refining force the formation of GE, but on the other that it might be possible to reduce their formation by lowering the temperature during deodorization while the amount of 3-MCPD-E is relatively unaffected.

### OTHER REFINING TECHNIQUES

Dual deodorization can be used to reduce the formation of esters by lowering the total temperature load of the oil during deodorization. A comparison of various combinations of high temperature/short-term and low temperature/long-term deodorization to conventional (one-step) deodorization shows a significant reduction of GE when a short-term deodorization at a higher temperature was followed by a long-term deodorization at a lower temperature (Fig. 2, page 653). However, reduction of 3-MCPD-E starting two-step deodorization at a higher temperature results in an opposite trend, with slightly higher amounts produced. In general, the potential to minimize 3-MCPD esters by two-step deodorization is lower than for GE.

Another approach is short-path distillation, which allows the esters to be reduced completely. The resulting palm oil is red, since the heat bleaching of carotenes does not take place at such a low temperature. Depending on the quality of the raw oil, a steam washing at  $160^{\circ}\text{C}$  or  $180^{\circ}\text{C}$  may be necessary to improve the oil's sensory quality. The steam washing results in a slight increase of the content of 3-MCPD-E to 1 and 1.9 mg/kg, respectively.

Laboratory-scale tests have shown that additives can be used to prevent the formation of the esters, to destroy formed esters, or to compete with precursors. Examples include:

- the addition of alcohol to refined bleached palm oil, which results in the formation of volatile chlorinated adducts that are stripped off during deodorization (18, 25);
- the addition of bicarbonates or carbonates during deodorization to neutralize free hydrogen chloride (26, 27); and
- the addition of 2% citric acid before deodorization, which results in a remarkable reduction of GE and no effect on the 3-MCPD-E (28).

### REMOVAL FROM FULLY REFINED OILS

The use of adsorption materials such as calcinated zeolite or synthetic magnesium silicate after the deodorization step can reduce the content of GE by 40% but has no influence on the 3-MCPD-E (29). Zeolite enables a spontaneous reduction of the esters at  $60^{\circ}\text{C}$ , while synthetic magnesium silicate needs at least 60 min at  $80^{\circ}\text{C}$  to reach a similar result.

Shimizu *et al.* (30) described the removal of GE from both TAG and DAG oils by the use of activated bleaching earth, which transforms GE to glycerol monopalmitate, glycerol palmitate oleate, and glycerol dipalmitate.

The literature also describes the degradation of 3-MCPD-E in a biphasic system. The esters are degraded into free 3-MCPD by *Candida antarctica* lipase A and afterwards the free 3-MCPD was transferred into glycerol via treatment with halohydrin dehalogenase from *Arthrobacter* sp. AD2 and an epoxide hydrolase from *Agrobacterium radiobacter* AD1 (31).

### CONCLUSION

With so many different approaches to reducing the formation of 3-MCPD and GE available today, it should be possible to produce edible oils with remarkably lower amounts of 3-MCPD-E and GE. However, producing oils with lower amounts of the esters will only be feasible if customers are willing to pay for the additional efforts. As a general rule, mitigation is easier and more successful the sooner it starts within the processing chain. Therefore, it is highly recommended that mitigation strategies be introduced on the plantation. If this is not possible, adapting the refining process or the removal of the esters from the refined oil is practicable. Also, the combination of different approaches is conceivable and meaningful.

*Bertrand Matthäus is a food chemist and scientist at the Max Rubner-Institut in Detmold, Germany. He can be contacted at [bertrand.matthaus@mri.bund.de](mailto:bertrand.matthaus@mri.bund.de).*

*Frank Pudel is with Pilot Pflanzenöltechnologie Magdeburg, a private, nonprofit research and development institute dealing with oil production and processing.*

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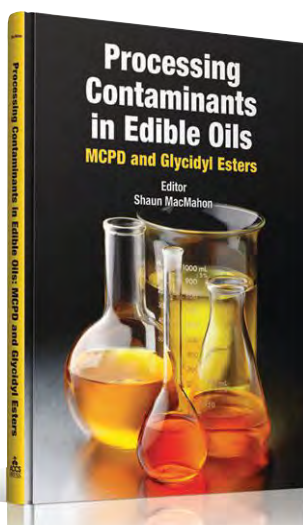
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# Leading innovation with customer, project, and innovation management

Linsen Liu

Major technologies commercialized to replace replacing partially hydrogenated oils (PHO) include blending, chemical and enzymatic interesterification, low-trans hydrogenation, and low-linolenic or high-oleic oils developed through biotechnology. Currently, no simple solution can match the broad application range of PHOs in food applications. No-trans oils generally have narrower functionality or less flexibility in applications, and many trans solutions have to be tailored to meet perfor-

- Several major technologies have been commercialized to replace partially hydrogenated oil (PHO), but currently no simple solution can match the broad application range of partially hydrogenated oil (PHO) in food applications.
- Many food manufactures have adopted short-term solutions based on palm while developing long-term solutions based on specialty oils.
- This article provides a management framework for scientists and managers to improve their effectiveness in developing no-trans and low-saturated oil formulas.

mance requirements in target applications. Although implementing multiple tailored-solutions in a plant may reduce operational efficiency significantly, strategically, food manufacturers want to eliminate trans fat without increasing the level of saturated fat.

Consequently, many food manufactures have adopted short-term and long-term solutions. Most short-term solutions are based on palm oils, which are affordable and have an established technology and supply chain. Long-term solutions are typically based on specialty oils which achieve no-trans and low-saturated fat objectives simultaneously. Most food manufactures have accomplished short-term solutions and are currently developing or implementing long-term ones. This article provides a management framework for scientists and managers to improve their effectiveness in developing no-trans and low-saturated oil formulas.

## CUSTOMER MANAGEMENT

Because oils are generally supplied as ingredients to customers in the food industry, customers are the major source of product development and innovation projects. To meet customer needs at the lowest possible cost, a technical or project leader must be effective in working with customers to identify their needs, transfer those needs into a product specification, and develop solutions effectively. Customers who lack oil expertise often ask suppliers to match an existing specification. However, because most no-trans oils are based on non-hydrogenated oils and typically have narrower functional ranges than the trans fats they are replacing, matching the specification of a trans fat may be impossible, too costly, or unnecessary.

Consequently, a technical or project leader must be trained and skilled in working with customers to determine their needs through the process of customer management. The customer management process is used to identify customer goals and to quantify business opportunities. It is a screening process and requires training to master, but with-





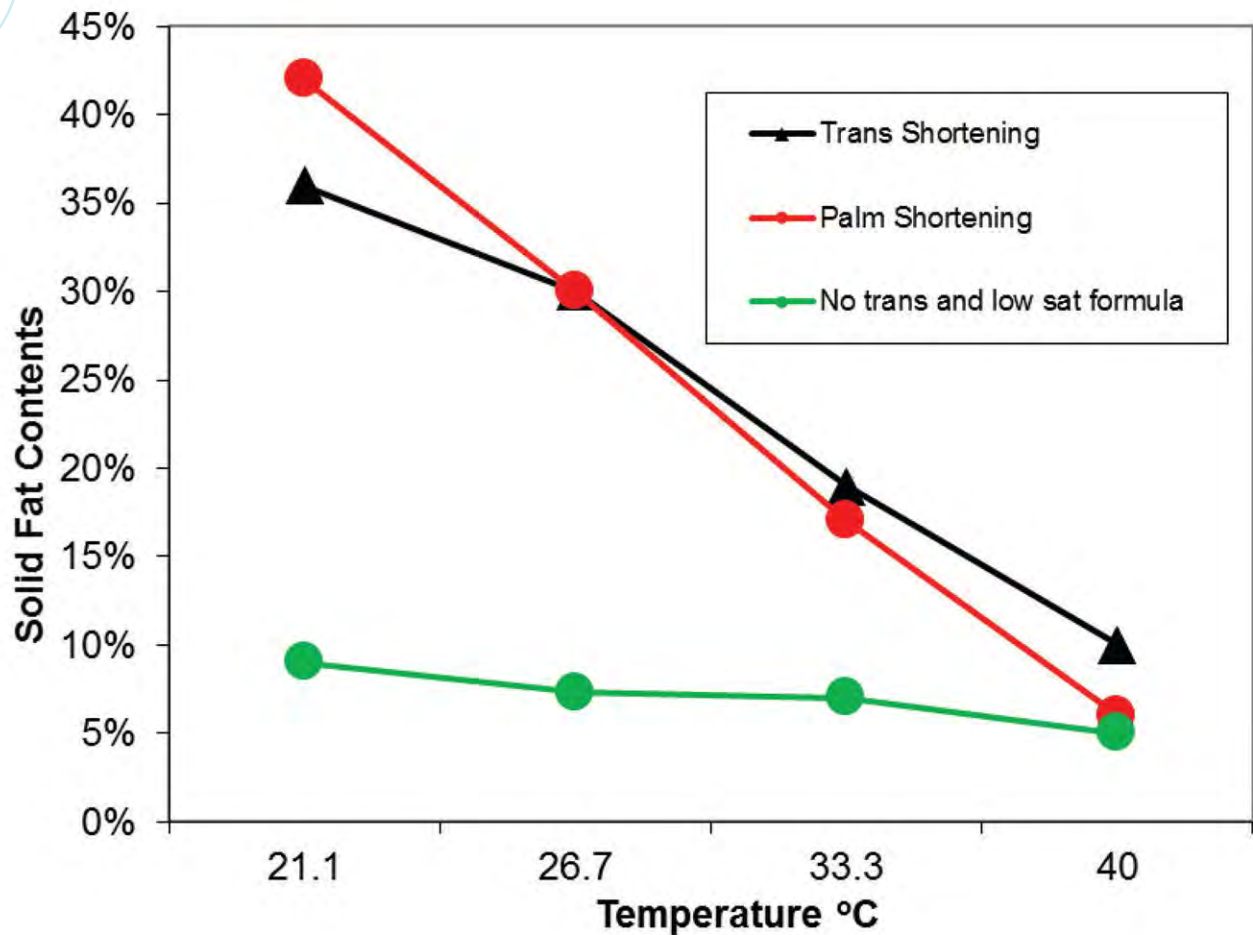
To improve the success rate of projects, food manufacturers and oil suppliers often sponsor joint projects in the hope these will improve information sharing and result in a better understanding of customer needs. However, many joint projects conducted by suppliers and customers end in dissolution after both parties have invested several years and millions of dollars—primarily because the project leaders lack skills in customer management. Often, such project managers follow customers' leads indiscriminately under the principle that "the customer is always right." This attitude and incompetence in customer management can waste millions of dollars on both sides. That many American food manufacturers have begun hiring in-house lipid scientists may be evidence that joint projects cannot satisfy customer needs to find long-term solutions for trans fat. Therefore, to be competitive in innovation, an oil supplier must have a technical team that is competent in customer management.



## REJECT

Once customer needs have been clearly defined through the customer management process, these needs are translated into a product specification that can be used to find a solution within the context of a project.

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**Fig. 1.** Comparison of solid fat contents of a commercial trans fat, palm shortening, and an innovative new low-saturated shortening with no trans

A project is defined as a temporary endeavor to create a unique product or service. The purpose of project management is to insure projects are properly authorized and implemented according to project objectives. To accomplish that, a project manager or technical leader needs basic project management skills.

A project has four general phases: feasibility, design, development, and deployment. In the first phase, a feasibility study is conducted to determine if a product can be achieved at the target profitability within a given timeline. In the second phase, product concepts and key specifications are evaluated against customer requirements, and they are also reviewed by a cross-functional team that considers such factors as performance, cost, sourcing, supply chain, manufacturing, country of origin, and risk management. This cross-functional review helps insure that products will be designed for easy production at minimal risk and cost.

Development is the most costly phase for research and development, as this is when product concepts are converted into product prototypes—and that takes time and money. Mismanagement during this stage could waste precious resources, disappoint customers, and damage a supplier's reputation. During the final phase, deployment, the project is transferred to manufacturing. At this point in the

process, the focus is to deliver consistent quality and timely technical services to the customer.

The proper execution of each phase depends on five cohesive processes: initiating, planning, executing, controlling, and closing. Initiating involves authorizing each phase of the project and defining its scope and objective. Planning is a process in which all stakeholder requirements are identified and integrated into a work plan that includes milestones and tasks. Each task should be definitive and measurable so it can be assigned to an individual who will serve as that task's "owner." Project planning insures that a project is properly scoped out and that all stakeholders are consulted, while execution involves the accomplishment of individual tasks and milestones by the individuals to whom they were assigned. Controlling is a process used to identify and correct deviations or gaps in meeting the project objective. The most important function of controlling is to prevent the team from deviating from the project scope. Without a defined work plan, projects tend to creep from their scope and become activity-driven; this can cause a project to drag on for years. Closing consists of gaining customer approval on deliverables or prototypes so a project can progress to the next phase. A project charter is often used to capture a project's objective, scope, and economics. After defining the charter and using it to gain the



buy-in of senior managers or project sponsors, the project manager leads the cross functional team to plan, execute, and close the project. Unfortunately, project management is not always vigorously applied in the oil and food industries, allowing many projects to become open-ended endeavors that go on for years and waste millions of dollars.

## INNOVATION MANAGEMENT

An enterprise should have a broad range of projects covering its short- and long-term business needs. In the food ingredient industry, short-term development projects are usually customer-based while long-term projects anticipate customers' future needs. Similar projects are often bundled together into a program. A portfolio refers to a series of projects and programs undertaken by an enterprise to meet its mission. Companies use program and portfolio management to manage the internally driven and market-forward innovation that builds their product pipelines. Many companies in North America have program and portfolio management offices designated to manage long-term innovation.

Innovation can be continuous or disruptive. Continuous innovation improves existing technology or products by making them more efficient or easier to use. Line extensions, such as new flavors or new features, are typical examples.

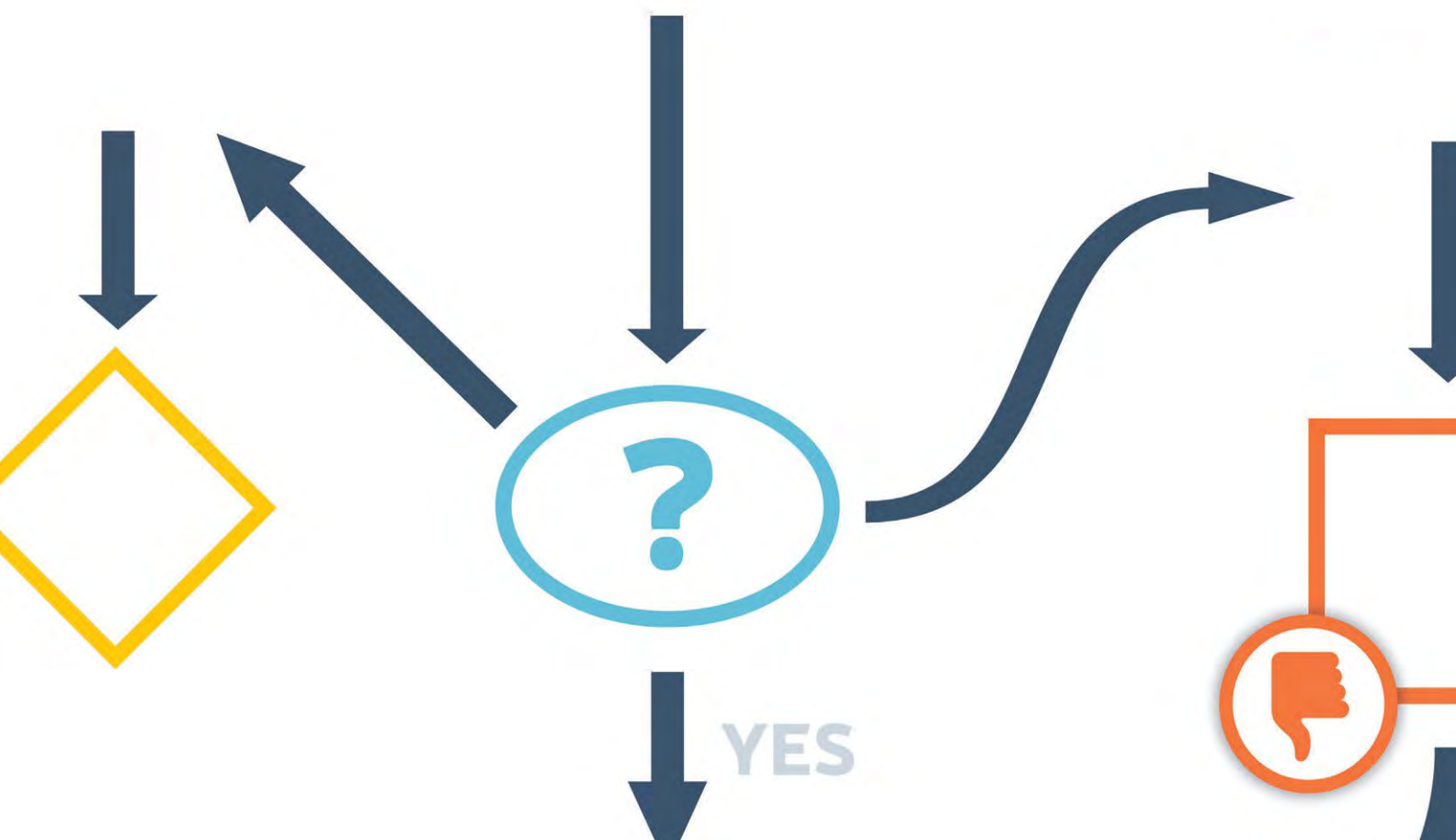
Disruptive innovation, on the other hand, delivers a game-changing solution. For instance, the superior storage, speed, price, and availability of flash drives quickly made floppy disks obsolete. The market value of continuous innovation can be quantified through marketing research, but disruptive innovation often has no historic data to support marketing research. Often, disruptive technologies are seemingly inefficient and too costly in the beginning. However, history has

proven that such innovations can ultimately wipe out existing technologies. In the mid-1990s, many executives at large oil-supplying companies focused on improving hydrogenation instead of developing trans replacement, because at that time they could sell every drop of hydrogenated oil they made. Today, hydrogenation is down to less than one third of its peak capacity and on its way to being eliminated. In contrast, the high-oleic and low-linolenic oils developed before the 1990s were initially considered unrealistic and too expensive, but today have reached billions of pounds in commercial production and have become one of the basic ingredients for replacement of trans fat.

Algal oils, omega-3 vegetable oils, and other specialty low-saturated vegetable oils, are still emerging, and their impacts will be tested in the coming decades. Balancing R&D investments in continuous and disruptive innovation to meet short-term and long-term needs can be challenging. In the oil industry, disruptive technology may take decades to mature and, therefore, needs both visionary leadership and venture capitalism to nurture.

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Yuliya Nam-Wright

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Prices for most vegetable oils have been trending downwards since April, having been driven down by exceptionally good supplies of soybean and palm oil.

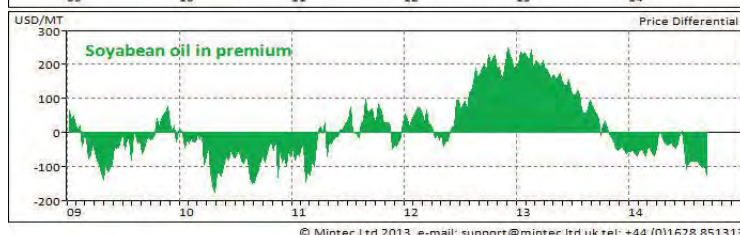
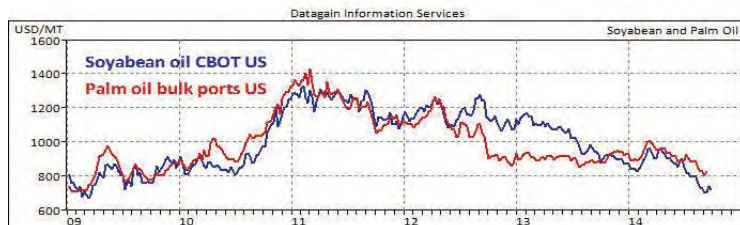
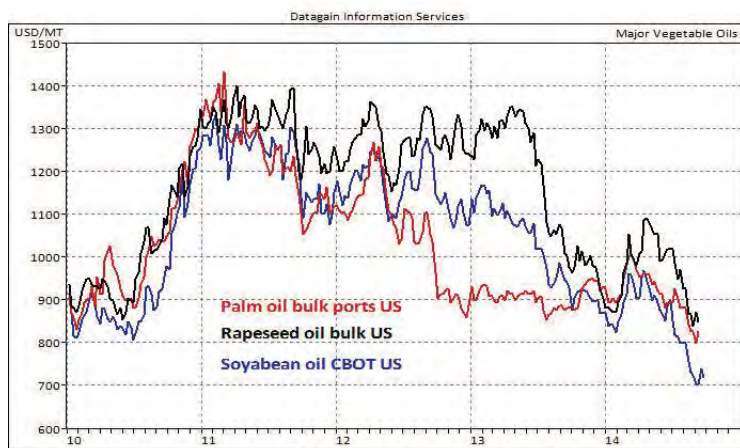
Prices for soybean oil have declined the most, with futures on Chicago Board of Trade falling to a five-year-low in September. World production of soybeans is forecast at 311.1 million metric tons (MMT) in 2014/15, up 10% year-on-year, driven by good supply from all major producing countries. Production of soybean oil is forecast to rise 5% to 47.0 MMT. Ending stocks of soybeans are expected to increase sharply by 35% year-on-year to 90.2 MMT as a result of consecutive seasons of production increases.

Palm oil prices also fell, driven by good supply, although less significantly than soybean oil prices. Soybean oil has been trading at a discount to palm oil since the end of 2013, and the discount has widened in recent months. This was partly due to growing consumption in major producing countries, supported by governmental initiatives to promote biodiesel production.

The Malaysian government is also planning to introduce further policies to raise domestic consumption of biodiesel towards the end of this year. As a result, monthly usage of Malaysian palm oil for biodiesel is expected to rise from 20,833 metric tons (MT) to 41,667 MT by the end of 2014, and will increase further to 58,333 MT in the first quarter of next year. World production of palm oil is forecast to reach a record high of 63.3 MMT in 2014/15, up 9% year-on-year. Consumption is set to rise by 6% to 60.7 MMT, and ending stocks will rise 11% to 8.8 MMT.

Global production of sunflower seed is forecast to reach 40.5 MMT, down 6% from the last year's record-high crop. Crush is expected to fall 1% to 37.4 MMT and production of sunflower oil is forecast to fall 1% to 15.5 MMT.

World production of rapeseed is forecast to fall 1% year-on-year to 70.7 MMT, also down from the last year's exceptionally good crop. Production of rapeseed oil is set to rise slightly by 1% to 26.5 MMT, while ending stocks of rapeseed will reach 6.8 MMT, down 4% year-on-year.



# EXTRACTS & DISTILLATES

## Quantification of $\gamma$ - and $\alpha$ -tocopherol isomers in combination with pattern recognition model as a tool for differentiating dry-cured shoulders of Iberian pigs raised on different feeding systems

Rey, A.I., et al., *J. Sci. Food Agric.* 94: 2649–2654, 2014, <http://dx.doi: 10.1002/jsfa.6603>.

Quantification of  $\gamma$ - and  $\alpha$ -tocopherol in dry-cured shoulders of Iberian pigs was evaluated as a tool for differentiating feeding backgrounds or regimens. Samples ( $n = 115$ ) were obtained over two different seasons from the four categories of pigs described in the Industry Quality Policy, i.e. pigs fed in free-range conditions (free-range), pigs fed in free-range conditions and provided feed supplements (Free-feed), pigs fed outdoors with feed and with access to grass (feed-out) and pigs fed in intensive conditions with feed (feed). Linear discriminant functions were calculated and validated. The validation results showed that 20% of the muscle samples were not correctly classified into the four feeding categories, giving an 80% success rate. The Feed group had the lowest proportion of errors, with 100% of samples correctly classified. For the free-range group, 87% of samples were assigned to the correct feeding system by cross-validation; however, 13% were considered as free-feed. A higher rate of correct classification can be obtained when using three categories or by calculating the weight gain in free-range conditions using regression equations. Taking into account the high variability of the samples and the high success in classification, these results are of interest and may be applied in practical situations.

## Implication of modified molecular structure of lipid through heat-related process to fatty acids supply in *Brassica carinata* seed

Xin, H., et al., *Ind. Crops Prod.* 62: 204–211, 2014, <http://dx.doi: doi.org/10.1016/j.indcrop.2014.08.025>

This study was conducted to explore the effect of different autoclave heating times (30, 60 and 90 min) on fatty acids supply and molecular stability in *Brassica carinata* seed. Multivariate spectral analyses and correlation analyses were also carried out in our study. The results showed that autoclaving treatments significantly decreased the total fatty acids content in a linear fashion in *B. carinata* seed as heating time increased. Reduced concentrations were also observed in C18:3n3, C20:1, C22:1n9, mono-unsaturated fatty acids (MUFA), polyunsaturated fatty acids (PUFA), omega 3 ( $\omega$ -3) and 9 ( $\omega$ -9) fatty acids. Correspondingly, the heated seeds showed dramatic reductions in all the peak intensities within lipid-related spectral regions. Results from agglomerative hierarchical cluster analysis (AHCA) and principal component analysis (PCA) indicated that the raw oilseed had completely different structural make-up from the autoclaved seeds in both  $\text{CH}_3$  and  $\text{CH}_2$  asymmetric and symmetric stretching region. However, the oilseeds heated for 30, 60 and 90 min were not grouped into separate classes or ellipses in all the lipid-related regions, indicating that there still exhibited similarities in lipid biopolymer conformations among autoclaved *B. carinata* seeds. Moreover, strong correlations between spectral information and fatty acid compositions observed in our study could imply that lipid-related spectral parameters might have a potential to predict some fatty acids content in oilseed samples, i.e. *B. carinata*. However, more data from large sample size and diverse range would be necessary and helpful to draw up a final conclusion.

## Preliminary formulation development for aqueous surfactant-based soybean oil extraction

Do, L.D., et al., *Ind. Crops Prod.* 62: 140–146, 2014, <http://dx.doi: 10.1016/j.indcrop.2014.08.026>.

Soybean oils are increasingly being used for a range of non-food applications, including production of biofuels and oleochemicals. While most soybean oil is produced by hexane-based extraction methods, concern about environmental and health effects from hexane extraction has led to increased interest in development of aqueous extraction methods. Among aqueous methods, surfactant-based aqueous extraction of vegetable oils has shown particular promise as an alternative to hexane-based extraction methods. The objectives of this work were to explore the use of surfactant-based methods for the extraction of soybean oils, and to test whether the use of mixed anionic–cationic and anionic–cationic–nonionic surfactant mixtures could successfully be used to reduce the salinity requirements for surfactant-based extraction. All three formulations tested were capable of producing ultra-low ( $<0.01$  mN/m) interfacial tensions with soybean oil. One of the formulations, a four-component (three surfactant, one hydrotrope) mixture, was able to reduce the salinity requirement from 5% down to 0.75%. A range of experiments was conducted to better understand the factors influencing extraction yield for surfactant-based extraction of soybean oil. Extraction experiments were conducted with a single extended surfactant system

which has been used previously for extraction of other oilseeds. Extraction yields as high as 88.6% were observed for the conditions tested. Extraction yield was strongly dependent on salinity, and was found to increase with increasing shaker agitation rate, decreasing solid to liquid ratio, and decreasing particle size.

## The use of natural and synthetic phospholipids as pharmaceutical excipients

Van Hoogevest, P. and A. Wendel, *Eur. J. Lipid Sci. Technol.* 116: 1088–1107, 2014, <http://dx.doi.org/10.1002/ejlt.201400219>.

In pharmaceutical formulations, phospholipids obtained from plant or animal sources and synthetic phospholipids are used. Natural phospholipids are purified from, e.g., soybeans or egg yolk using non-toxic solvent extraction and chromatographic procedures with low consumption of energy and minimum possible waste. Because of the use of validated purification procedures and sourcing of raw materials with consistent quality, the resulting products differing in phosphatidylcholine content possess an excellent batch to batch reproducibility with respect to phospholipid and fatty acid composition. The natural phospholipids are described in pharmacopeias and relevant regulatory guidance documentation of the Food and Drug Administration (FDA) and European Medicines Agency (EMA). Synthetic phospholipids with specific polar head group, fatty acid composition can be manufactured using various synthesis routes. Synthetic phospholipids with the natural stereochemical configuration are preferably synthesized from glycerophosphocholine (GPC), which is obtained from natural phospholipids, using acylation and enzyme catalyzed reactions. Synthetic phospholipids play compared to natural phospholipid (including hydrogenated phospholipids), as derived from the number of drug products containing synthetic phospholipids, a minor role. Only in a few pharmaceutical products synthetic phospholipids are used. Natural phospholipids are used in oral, dermal, and parenteral products including liposomes. Natural phospholipids instead of synthetic phospholipids should be selected as phospholipid excipients for formulation development, whenever possible, because natural phospholipids are derived from renewable sources and produced with more ecologically friendly processes and are available in larger scale at relatively low costs compared to synthetic phospholipids.

## Lipophilization of ascorbic acid: A monolayer study and biological and antileishmanial activities

Kharrat, N., *et al.*, *J. Agric. Food Chem.* 62: 9118–9127, 2014, <http://dx.doi.org/10.1021/jf5029398>.

Ascorbyl lipophilic derivatives (Asc-C<sub>2</sub> to Asc-C<sub>18:1</sub>) were synthesized in a good yield using lipase from *Staphylococcus xylosus* produced in our laboratory and immobilized onto silica aerogel. Results showed that esterification had little effect on radical-scavenging capacity of purified ascorbyl esters using DPPH

assay in ethanol. However, long chain fatty acid esters displayed higher protection of target lipids from oxidation. Moreover, compared to ascorbic acid, synthesized derivatives exhibited an antibacterial effect. Furthermore, ascorbyl derivatives were evaluated, for the first time, for their antileishmanial effects against visceral (*Leishmania infantum*) and cutaneous parasites (*Leishmania major*). Among all the tested compounds, only Asc-C<sub>10</sub>, Asc-C<sub>12</sub>, and Asc-C<sub>18:1</sub> exhibited antileishmanial activities. The interaction of ascorbyl esters with a phospholipid monolayer showed that only medium and unsaturated long chain (Asc-C<sub>10</sub> to Asc-C<sub>18:1</sub>) derivative esters were found to interact efficiently with mimetic membrane of leishmania. These properties would make ascorbyl derivatives good candidates to be used in cosmetic and pharmaceutical lipophilic formulations.

## Studying the chemistry of cationized triacylglycerols using electrospray ionization mass spectrometry and density functional theory computations

Grossert, J.S., *et al.*, *J. Am. Soc. Mass Spectrom.* 25: 1421–1440, 2014, <http://dx.doi.org/10.1007/s13361-014-0917-9>.

Analysis of triacylglycerols (TAGs), found as complex mixtures in living organisms, is typically accomplished using liquid chromatography, often coupled to mass spectrometry. TAGs, weak bases not protonated using electrospray ionization, are usually ionized by adduct formation with a cation, including those present in the solvent (e.g., Na<sup>+</sup>). There are relatively few reports on the binding of TAGs with cations or on the mechanisms by which cationized TAGs fragment. This work examines binding efficiencies, determined by mass spectrometry and computations, for the complexation of TAGs to a range of cations (Na<sup>+</sup>, Li<sup>+</sup>, K<sup>+</sup>, Ag<sup>+</sup>, NH<sub>4</sub><sup>+</sup>). While most cations bind to oxygen, Ag<sup>+</sup> binding to unsaturation in the acid side chains is significant. The importance of dimer formation, [2TAG + M]<sup>+</sup> was demonstrated using several different types of mass spectrometers. From breakdown curves, it became apparent that two or three acid side chains must be attached to glycerol for strong cationization. Possible mechanisms for fragmentation of lithiated TAGs were modeled by computations on tripropionylglycerol. Viable pathways were found for losses of neutral acids and lithium salts of acids from different positions on the glycerol moiety. Novel lactone structures were proposed for the loss of a neutral acid from one position of the glycerol moiety. These were studied further using triple-stage mass spectrometry (MS<sup>3</sup>). These lactones can account for all the major product ions in the MS<sup>3</sup> spectra in both this work and the literature, which should allow for new insights into the challenging analytical methods needed for naturally occurring TAGs.

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## High resolution MALDI imaging mass spectrometry of retinal tissue lipids

Anderson, D.M.G., et al., *J. Am. Soc. Mass Spectrom.* 25: 1394–1403, 2014, <http://dx.doi.org/10.1007/s13361-014-0883-2>.

Matrix assisted laser desorption ionization imaging mass spectrometry (MALDI IMS) has the ability to provide an enormous amount of information on the abundances and spatial distributions of molecules within biological tissues. The rapid progress in the development of this technology significantly improves our ability to analyze smaller and smaller areas and features within tissues. The mammalian eye has evolved over millions of years to become an essential asset for survival, providing important sensory input of an organism's surroundings. The highly complex

sensory retina of the eye is comprised of numerous cell types organized into specific layers with varying dimensions, the thinnest of which is the 10  $\mu\text{m}$  retinal pigment epithelium (RPE). This single cell layer and the photoreceptor layer contain the complex biochemical machinery required to convert photons of light into electrical signals that are transported to the brain by axons of retinal ganglion cells. Diseases of the retina, including age-related macular degeneration (AMD), retinitis pigmentosa, and diabetic retinopathy, occur when the functions of these cells are interrupted by molecular processes that are not fully understood. In this report, we demonstrate the use of high spatial resolution MALDI IMS and FT-ICR tandem mass spectrometry in the *Abca4* (-/-) knockout mouse model of Stargardt disease, a juvenile onset form of macular degeneration. The spatial distributions and identity of lipid and retinoid metabolites are shown to be unique to specific retinal cell layers.