

Mineral oil hydrocarbons

A new challenge for the oils
and fats processing industry

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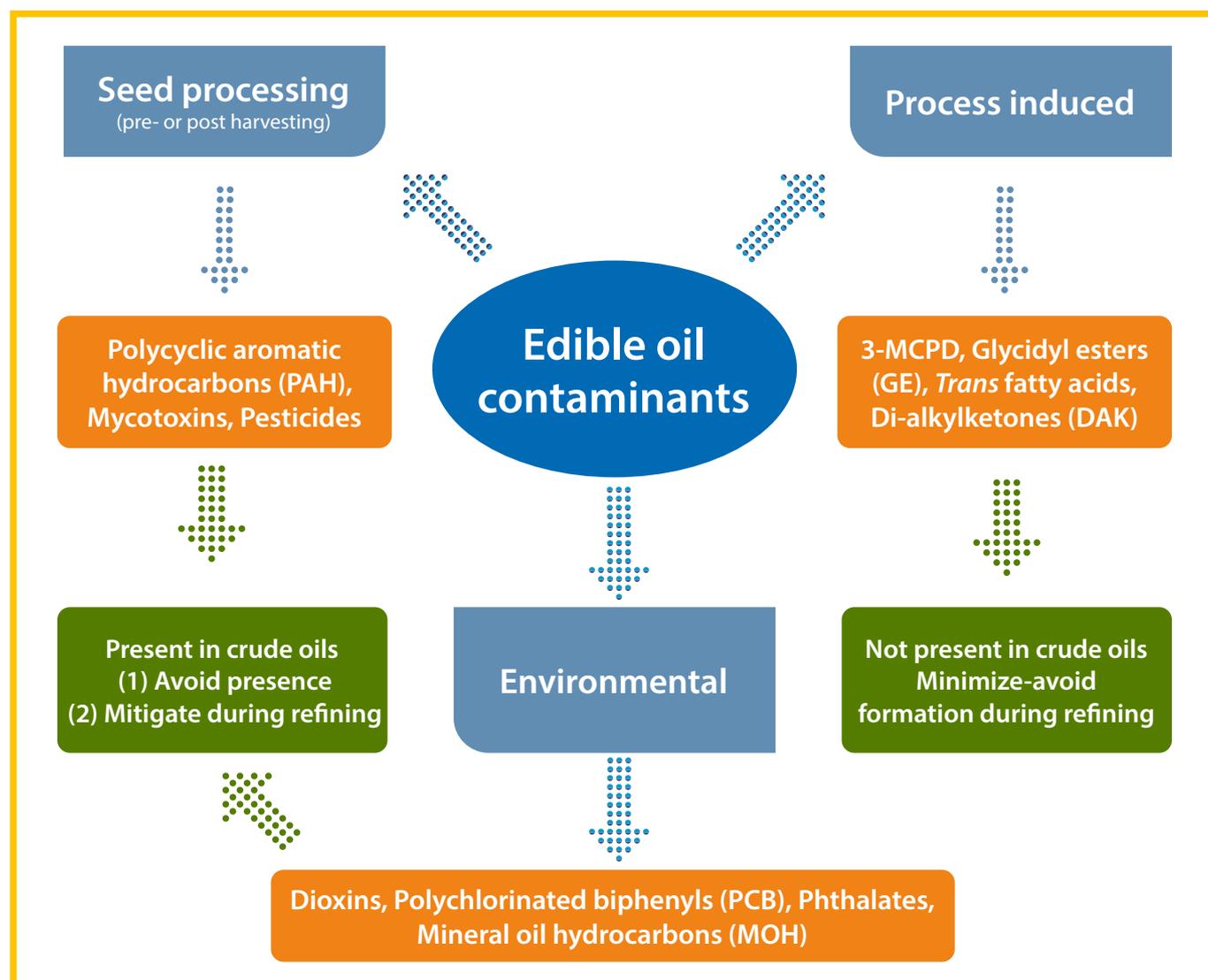
A new challenge for the oils and fats processing industry

- Mineral oils are widespread food contaminants, and edible oils, like many other foodstuffs, are often contaminated.
- The lack of robust analytical methods and proper toxicological evaluation make it difficult to set a tolerance level.
- The best way to avoid mineral oil contamination is to prevent it by complying with good manufacturing practices, and the best solution to reducing mineral oil contamination in edible oils is through refining, especially during deodorization.
- This paper gives an overview of contamination sources, levels in some edible oils, regulatory aspects, analytical methods, and strategies for mitigation during refining.



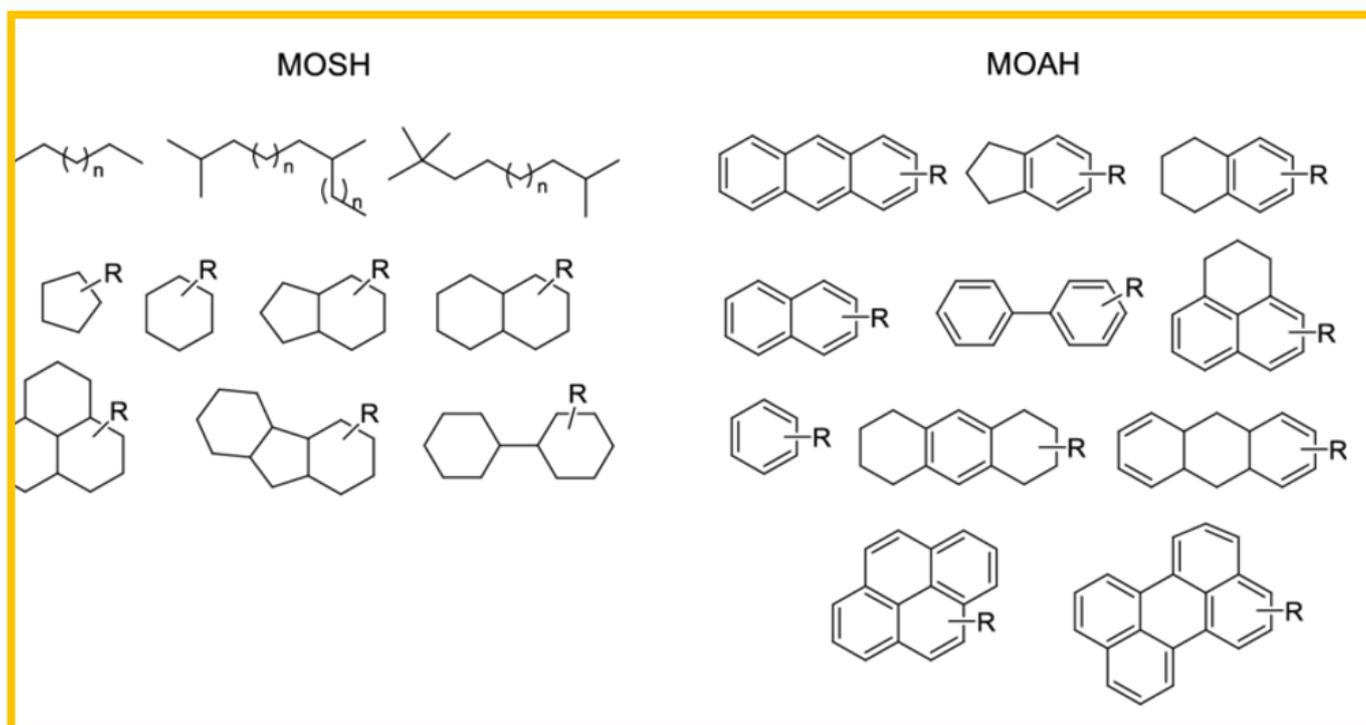
Refined oil quality is primarily evaluated by traditional parameters, such as free fatty acid content, oxidative stability, color, odor, and taste. In addition, high-quality food oils will contain low amounts of trans fatty acids, high amounts of natural antioxidants (tocopherols), and very low or no “process” contaminants, such as polycyclic aromatic hydrocarbons (PAH), mycotoxins, pesticides, dioxins, polychlorinated biphenyls (PCB), phthalates, 3-monochloropropane-1,2-diol (3-MCPD), glycidyl esters (GE), trans fatty acids, di-alkyl ketones, and mineral oil hydrocarbons (Fig. 1). Contamination with mineral oil hydrocarbons (MOHs) is not new but has attracted more attention recently.

Figure 1. Major contaminants of edible oils and fats



They are categorized into two main groups: mineral oil saturated hydrocarbons, or MOSH (paraffins and naphthenes), and mineral oil aromatic hydrocarbons, or MOAH (aromatics). MOSH correspond to straight and branched open-chain alkanes and largely alkylated cyclo-alkanes; MOAH are alkylated and non-alkylated polyaromatics (Fig. 2). MOHs exclude hydrocarbons naturally occurring in food and hydrocarbons from synthetic origin. They are usually classified according to their carbon number: total MOSH refer to the sum of C10-C16, C16-C20, C20-C25, C25-C35, C35-C40 and C40-C50 fractions, and total MOAH to the sum of C10-C16, C16-C25, C25-C35 and C35-C50 fractions.

Figure 2. Chemical structures of representative compounds found in MOSH and MOAH (1)



Source and level of contamination

MOHs are contaminants that can be present in food due to environmental pollution (air, soil), use of lubricants for machinery during harvesting and food production, food contact materials, processing aids, food additives, and other factors. They often come from malpractices in process operations. A contamination of walnut oil with a food-grade lubricant during refining was highlighted in 2009, followed in 2011 by a contamination of milk fat during production (2). In most cases, proper monitoring of good manufacturing practices is the best solution to avoiding such problems. The application of benchmark levels to identify unusually high loads of MOHs is informative (3). These levels are oriented to the market and allow manufacturers to respond appropriately; they are an effective tool for minimization. It is also important to monitor MOHs contents to better understand their relative presence

in food commodities; variable levels of MOSH (up to 50 mg/kg) can still be detected in large food categories.

Oil seeds, fats, and oils can accumulate MOHs from all kinds of sources along the production chain. A general overview is given in (4) for vegetable oils like sunflower oil, olive oil, palm oil, sesame oil, walnut oil, rapeseed oil, argan oil, poppy seed oil, olive oil, olive pomace oil, cottonseed oil, and grapeseed oil; contamination is also reported in oils for feed production. A specific survey is described in (5, 6) for olive oils. Contents depend on the variety: 8 to 90 mg/kg for MOSH and 1 to 13 mg/kg for MOAH, mostly in the C25-C35 fractions. Highly contaminated oils contain heavier MOSH (C35-C50).

The average MOSH and MOAH contents (C10-C50) in a series of oils are presented in (7). The highest amounts are found in coconut oils (MOSH up to 40 mg/kg and MOAH above 15 mg/kg); the contents largely depend on suppliers, and some special grades do not have MOAH. In MOSH, the major fractions are C16-C40; in MOAH the major ones are C16-C35. MOSH in soybean are also high (up to 35 mg/kg), although MOAH are usually low (< 2 mg/kg). Rapeseed, palm kernel, and sunflower oils, are also reported to have low MOAH and MOSH levels below 5–10 mg/kg.

Higher contents in MOSH (> 25 mg/kg) and in MOAH (> 2.5 mg/kg) are detected in palm oil; however, palm oil from sustainable sources with low MOH contents is preferred for infant formula applications. A pilot study on MOSH status in crude palm oil was conducted by Sime Darby Plantation Palm Oil Mills and published in 2019 (8). Results revealed that MOSH in Hazard Analysis Critical Control Point (HACCP)-certified palm oil mill was at the range of 10–17 mg/kg, while non-HACCP certified-mill had an average of 44.8 mg/kg. Premium CPO showed lower MOSH, with 10.7 mg/kg compared to standard CPO at 21.9 mg/kg. The study did not establish any correlation between the free fatty acid and the MOSH contents.

A survey of MOSH and MOAH in different vegetable oils is shown in a 2020 article (9). Cocoa butter had the highest contents: up to 162 mg/kg for MOSH and 55 mg/kg for MOAH. MOSH were mostly observed in the \leq C35 fraction, and MOAH in the \leq C24 fraction; presence of benzothiophene, dibenzothiophene, and alkylated naphthalenes was detected. Palm oil contained up to 124 mg/kg of MOSH (in \leq C35 and in > C35 fractions, in lower extent in \leq C16 and \leq C24 fractions) and 39 mg/kg of MOAH (mostly in \leq C35 and > C35 fractions). Sunflower oil showed lower contents: less than 17 mg/kg of MOSH (in \leq C35 fraction) and no MOAH. In 2009, other authors (10) had already described similar levels for sunflower oil and concluded that environmental contamination and harvesting were critical factors for accumulation in the seed oil.

All new data will help fill the missing gaps needed for proper risk assessment and make it possible to establish a tolerable daily intake (TDI) for MOSH in the next EFSA (European Food Safety Authority) opinion due December, 2022.

Risk assessment

MOHs pose potential health risks in animal studies; Sprague Dawley rat seems to be a good model for humans, but it is not yet confirmed. Scientific understanding of MOHs has advanced significantly, but knowledge gaps remain, particularly regarding the risk to humans.

The risks associated with MOSH are different from those of MOAH. MOSH are neither carcinogenic nor mutagenic; there is no direct indication of harmful effects during accumulation - even though formation of granulomas in organs of Fischer rats is reported. Critical effect was nevertheless identified by EFSA, with NOAEL (No Observed Adverse Effect Level) value of 19 mg/kg body weight/day.

MOAH are carcinogenic, mutagenic, and hormone-disruptive. Carcinogenic potential seems to correlate with increasing number of aromatic ring systems; MOAH are suspected to contain bio-accumulative genotoxic compounds, mainly related to 3- to 7-ring aromatic substances. Today, no safe thresholds are clearly defined, and presence in foods is a concern.

Regulatory aspects

There is currently no EU legislation regulating the limits of MOHs in vegetable oils—only a recommendation (2017/84) for their monitoring in food. However, large companies are setting their own standard for maximal levels in vegetable oils and fats.

In 2012, EFSA released a scientific opinion on MOHs in foods: all MOH mixtures are mutagenic unless they are treated specifically to remove MOAH, and total MOAH must be monitored with analytical methods able to separate different structural subclasses (11). In October 2015, food watch tests were organized on 120 dry products; 52 of 120 products tested were contaminated with the potentially carcinogenic MOAH. The introduction of functional barriers became mandatory, and EFSA started to consider specific limits for MOSH and MOAH in foods. In 2017, an EC (European Community) recommendation (2017/84) was released for MOHs monitoring in food. In April 2019, the JRC (Joint Research Centre of the European Commission) published a guidance on sampling, analysis, and data reporting for the monitoring of MOHs in food and food contact materials. In October 2019, a food watch test on infant formula was organized based on analytical methodology following JRC guidance; in half of the products, MOAH contamination was detected up to 3 mg/kg.

Industry publicly denied MOAH contamination and EC immediately commissioned EFSA to rapidly assess the methodology/results published. In December 2019, the food watch test was proved to use inadequate methodology with false negative results. In February 2020, *Reuters* Malaysia published an article claiming that Nestlé was putting pressure on the palm oil industry to reduce MOHs contamination. In June 2020, the European Standing Committee on Plants, Animals, Food and Feed (SCOPAFF) defined a harmonized risk management approach with a maximum level of 1 mg/kg of MOAH per C-fraction.

Contamination with MOSH is almost avoidable, and with MOAH is nearly completely avoidable; these compounds represent an unjustifiable health risk for all consumers. An ongoing collaboration between industry and health and regulatory authorities is a promising solution with multiple benefits. There have already been many advances in analytical techniques which still need to become fully routine in the service laboratories; qualified ring trials and harmonization will drive further progress.

In anticipation of a specific EU legislation, the German Food Society has put a “benchmark level” of max. 13 ppm C10-C50 for MOSH in edible oils, while MOAH should be below the LOQ (the limit of quantification).

Analytical methods

Standardized analytical methods are useful in establishing internationally validated methods with precision data, and to verify product compliance with regulations. The first systematic study on the contamination of food by MOHs appeared in 1991; the LC–GC (liquid chromatography-gas chromatography) technique experienced success in the 1990s. The topic was not considered for almost 20 years, but re-emerged in 2008, with the discovery of highly contaminated Ukrainian sunflower oil (12); this led to the need for proper MOAH quantification. A method to separate the MOSH and MOAH was developed.

In 2015, the ISO 17780: 2015 method (13) was used as reference for MOSH, with a LOQ of 50 mg/kg. In 2017, the EN 16995: 2017 method (14) was released for MOSH and MOAH, with a LOQ of 10 mg/kg. From 2019 to 2021, the focus was on introducing additional clean-up steps in the EN 16995 method to lower the LOQ, for both MOSH and MOAH, from 10 mg/kg to 1–2 mg/kg. The DGF-C-VI 22 (20) method (15) was proposed in 2020, with an improved precision and comparability with a LOQ of 1 mg/kg. The general approach of the method consists in: (1) extraction of the mineral oil and enrichment, (2) separation of the MOSH and MOAH by liquid chromatography, and (3) quantification by gas chromatography.

Chromatograms are generally complex with many interferences, since food products contain varieties of liposoluble components, such as waxes, squalene, steradienes, terpinoids, and carotenoids. Sample preparation is essential, and clean-up with aluminum oxide makes it possible to retain long-chain natural n-alkanes which interfere with the quantification of MOSH. Epoxidation is used to eliminate natural compounds like squalene, terpinoids, and carotenoids, which interfere with the quantification of MOAH. Special epoxidation treatments or additional purification steps after epoxidation may be necessary in the case of important disturbances due to matrix components.

The need to understand the source of the Ukrainian sunflower oil contamination brought into play another key hyphenated technique, GC × GC, which allows a detailed characterization of the MOSH and MOAH fractions to be obtained. GC × GC provides a sensitivity gain and a burst in separation power, along with a well-ordered 2D plot. This permits a group type separation (16). GC × GC-FID (flame ionization detection) enables a rough separation and quantification of paraffins and naphthenes in the MOSH fractions; it also appears to be the most effective method to distinguish the different MOAH subclasses.

In May 2020, a co-authored paper by two recognized institutions, the Joint Research Centre (JRC) of the European Commission and the Official Food Cantonal Laboratory of Zürich, clearly stated that GC × GC is the best technique for verification purposes (17), confirming the previous suggestion reported by the EFSA in 2012. The combination of GC × GC with TOF-MS (time-of-flight–mass spectroscopy) permits the separation of condensed aromatics and consequently helps to identify with more confidence the occurrence of carcinogenic or mutagenic constituents, namely the 3–7 ring MOAH. The use of multidimensional GC × GC with parallel dual detection (FID and TOF-MS) was recently proposed as a confirmatory method to better distinguish MOHs fractions from interferences. The method was optimized and published (18); it shows completely overlapped 2D plots from the two detectors and helps to correct any quantification issue due to coelution with naturally present components.

Laboratory discrepancies

The JRC guidance does not properly clarify how to report the LOQ, resulting in different approaches taken by laboratories (19). Some laboratories provide LOQs that may be different for each individual C fraction; others consider that the same LOQ applies whatever the C fraction. Some laboratories calculate the result for “total C10-50” by summing the values for the individual C fractions which are greater than LOQ. This does not take into consideration the complete signal as indicated in the JRC guidelines. In some commercial laboratories, LOQs are

depicted only when a value of the corresponding C fraction is below the LOQ; in cases where the value exceeds the LOQ, the LOQ is not shown. The LOQs are therefore not always known by the company requesting the analysis. The need for confidence in laboratory performance is essential; such confidence can be demonstrated through laboratory participation in proficiency testing schemes (20).

Mineral oil mitigation during refining

There is little information available in the literature on mineral oil mitigation during edible oil refining, and data are sometimes contradictory.

In 2001, Wagner, *et al.* (21) reported the paraffin content in some refinery by-products and edible oils. By-products (fatty acids from the deodorizer condensate) showed an average concentration of 650 mg/kg. Crude, semi-, and fully refined vegetable oils from diverse origins were investigated; with few exceptions, most of the levels detected were below 50–100 mg/kg. The authors reported the influence of press versus solvent extraction; differences observed were not related to paraffins present in the hexane migrating into the crude oil, but rather to better extractability of the paraffins from the seeds by the solvent. Removal of mineral paraffins during chemical refining of peanut oil was monitored. The crude oil contained two types of paraffins: one centered at C22 and another reaching up to C28 to beyond C40. Deodorization completely removed the paraffins up to C23 and about 50% of C25; harder conditions from physical refining were necessary to remove the longer-chain fractions. However, neutralization and bleaching did not modify the paraffin content (50 mg/kg), which only decreased after deodorization (14 mg/kg).

On the other hand, in a 2006 article (22), van Duijn claimed that much of the mineral oil that does find its way into edible oils is removed during the refining process. Gasoline and diesel can be removed during neutralizing and bleaching, and mid-fraction mineral oil (C20–C35) is stripped during deodorization. However, heavy-fraction mineral oil, such as grease and hydraulic oil, are not removed during refining.

In a 2020 article (9), crude and deodorized cocoa butters from different refineries were analyzed; the MOSH and MOAH in \leq C24 fraction were reduced by 10 to 75%, depending on the process conditions applied. In coconut oils, the MOSH \leq C24 fraction was also diminished by the refining process to levels below LOQ, while ranges of higher carbon number were reduced to less extent. The entire MOAH fraction from C10–C50 was removed, resulting in MOAH levels below LOQ. For MOSH, only single iso-alkanes were present after refining; for MOAH, naphthalenes, pyrenes, and fluoranthenes, predominantly present in crude oils, were eliminated by the refining process. The

influence of deodorization temperatures (140°C to 240°C) on MOSH/MOAH reductions was investigated on laboratory scale using spiked crude cocoa butter. Significant reduction of $\leq C_{24}$ in MOSH and MOAH fractions was observed at 210°C; both desired effects of free fatty acids and MOSH/MOAH reduction could be achieved.

In a 2021 survey (5), crude olive pomace oils were shown to contain MOSH ranging from 30 to 300 mg/kg and MOAH from 2 and 30 mg/kg. To better understand these important deviations, MOSH and MOAH were analyzed in both solvent and press-extracted oils. MOSH and MOAH were much higher in the solvent-extracted oils (210 mg/kg versus 65 mg/kg for MOSH, and 65 mg/kg versus 3 mg/kg for MOAH). It was shown that the hexane used in solvent-extracted olive pomace oil had a neutral effect, and that the higher content observed was only related to better oil extractability of the solvent.

The MOSH and MOAH contents were analyzed at different stages of the chemical refining. Neutralization, water washing, and bleaching did not significantly decrease the contents, but some C10-C35 MOAH were slightly reduced after bleaching. Most of MOSH and MOAH still remained in the deodorized oil apart from the lighter



C10-C16 MOSH fraction which was fully removed; the C17-C24 MOSH fraction and, to a lower extent, the C25-C35 fraction were slightly decreased. The heavy fractions of both MOSH (C36-C40 and C41-C50) and MOAH (C36-C50) were almost untouched.

MOSH and MOAH contents were analyzed in crude sunflower oil obtained from spiked and un-spiked seeds as well as spiked crude sunflower oil in the framework of a recent German FEI research project (23). The contents were determined at different stages of physical refining: after filtration, water washing, degumming, bleaching, and deodorization. MOSH and MOAH significantly decreased after deodorization only.

For rapeseed oil, the influence of deodorization time (60 -> 150 min.) and temperature (220°C a real arrow of 280°C) on MOSH removal were evaluated. Removal was time and temperature dependent, with the best removal at 260°C and 280°C; however, excessive trans fatty acids were formed. The optimal conditions for low-trans content (< 1%), with a residual MOSH content of 25–30% was 60 minutes at 240°C. Several absorption materials were tested but did not achieve significant MOSH and MOAH removal, except for active carbon (to remove PAHs). Further, cold treatment (winterization) did not show any significant removal of long-chain MOHs.

MOSH and MOAH contents in crude and fully refined soybean oil were reported in (7). In this example, MOSH in the crude oil (95 mg/kg) were principally C16-C20, C20-C25 and C25-C35. Very low MOAH contents (1.2 mg/kg) were observed (C35-C50). The C16-C20 and C20-C25 MOSH were almost completely removed in the refined oil which contained residual amounts of C25-C35; the high molecular weight fractions of both MOSH and MOAH were untouched.

Pilot trials were recently conducted using a tray-type deodorizer (24). Bleached high-oleic sunflower oil was spiked with relatively high amounts of commercial lubricants to reach 232.9 mg/kg of MOSH and 32.5 mg/kg of MOAH. Spiked oil was deodorized at 220°C, 245°C, and 260°C under varying amounts of stripping steam, and samples were analyzed (25) using the DGF-C-VI 22 (20) method (LOQ 1 ppm) (15). Almost 65% of MOSH were stripped at 245°C. The short-chain fractions (C10-C16 and C16-C20) were fully removed; ~90 and ~77 % of the C20-C25 and C25- C35 fractions were also removed, but the long-chain fractions C35-C40 and especially C40-C50 remained mostly untouched. Almost 50% of the MOAH were removed at 260°; the C16-C25 fraction was fully stripped regardless of deodorization temperature.

To sum it all up

The quantity and relative contribution of different mineral oil fractions depends on the source of contamination, generally differ from one oil to another, and may vary over time for each oil. Removing MOSH/MOAH during the refining process is mainly possible during deodorization and is temperature dependent. The relative removal and final content depend on the initial contamination. Under process conditions, the short chains of MOSH (C10-C16 and C16-C20) are generally stripped easily. The medium ones (C20-C25 and C25-C35) can be efficiently stripped, but the heavy ones (C35-C40 and C40-C50) usually remain in the oil. Similarly, the C16-C25 fraction of MOAH is easily distilled, removal of the C25-C35 is possible, but the C36-C50 fraction is more difficult to remove



Figure 3. General overview of a packed column stripper and low pressure ice condensing system (24)

In industrial practices, mineral oil can best be stripped over a packed column stripper operating at higher temperature and at low pressure with an ice condensing system (Fig. 3). The efficient countercurrent stripping and short residence time in the packed column will provide the most efficient mineral oil stripping, with minimal formation of trans fatty acids and glycidyl esters. Mineral oil can also be stripped via molecular or short path distillation, but use of these technologies will be difficult to justify due to high costs and higher oil losses. Higher stripping efficiency to remove MOH will also lead to more removal of essential components like tocopherols and sterols, which is disadvantageous for oil stability and nutritional quality. The best solution to mitigate MOH in edible oils is therefore to avoid contamination at the source.

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