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Review Article

Recent advances in sample preparation for the determination of mineral oil saturated (MOSH) and aromatic hydrocarbons (MOAH) in food matrices

Ana Jano [a], Ana M. Ares [a], Floriatan Santos Costa [b], Jorge A. Custodio-Mendoza [c], José Bernal [a,*], Adrián Fuente-Ballesteros [a,*]

[a] *Analytical Chemistry Group (TESEA), I. U. CINQUIMA, Faculty of Sciences, University of Valladolid, 47011 Valladolid, Spain*
[b] *Department of Chemistry, Federal University of Paraná, Curitiba 81531-980, Brazil*
[c] *Instituto de Agroecoloxía e Alimentación (IAA) – Food and Health Omics, Universidade de Vigo, Campus Auga, 32004, Ourense, Spain*

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ABSTRACT

The analysis of mineral oil hydrocarbons (MOH), particularly the saturated (MOSH) and aromatic (MOAH) fractions, has gained importance due to their widespread occurrence in food and associated health concerns. Human exposure mainly occurs through diet, with toxicological outcomes strongly dependent on the compounds' molecular structure, ranging from rapid metabolic clearance to potential carcinogenicity. Accurate quantification relies on efficient sample preparation prior to chromatographic analysis. Typical procedures involve analyte extraction, saponification to remove triglycerides, and clean-up strategies such as treatment with activated aluminium oxide or epoxidation, sometimes followed by enrichment. Nonetheless, their applicability depends on the target analytes and matrix interferences. This review summarizes recent advances (2020–2025) in sample preparation strategies for MOH analysis reported over the last five years. In addition to analytical performance, the sustainability and practicality of these methods is evaluated using green (AGREEprep) and blue (BAGI) metrics yielding scores ranging from 0.11 to 0.43 and from 50 to 70, respectively, thereby highlighting key opportunities for improvement in MOH analysis and providing insights into the applicability of current sample preparation methods.

1. Introduction

Mineral oil hydrocarbons (MOH) are a complex blend derived from petroleum, consisting of numerous structural isomers (see Fig. 1). These substances are generally categorized into two principal groups: mineral oil saturated hydrocarbons (MOSH), which consist of linear and branched paraffins, as well as alkylated cycloalkanes, and mineral oil aromatic hydrocarbons (MOAH), composed of alkylated (poly)aromatic hydrocarbons containing varying numbers of fused aromatic rings [1]. Mineral oils are commonly employed across a wide range of applications. They can be used as emollients in cosmetics, formulation aids in food products, and serve as release agents for baked goods and confectionery, as well as components of food packaging materials like wax paper and waxed cardboard [2]. The primary source of human exposure to MOH is food products [3], and in this category, the most studied matrices are edible oils. However, they can also be found in other food matrices such as dairy products, crisps, ready-to-eat meals or salads, cereals, baby food, legumes, nuts, spices, canned fish, and chocolate [4]. For example, Liu et al. [5] analysed ten commercial milk powders and reported total MOH concentrations between 0.61 and 5.46

Abbreviations: **AGREEprep**, Analytical Greenness Metric for Sample Preparation; **Alox**, Activated Aluminium Oxide; **BAGI**, Blue Applicability Grade Index; **FID**, Flame Ionization Detection; **GC**, Gas Chromatography; **GC×GC-FID**, Comprehensive Two-Dimensional Gas Chromatography with Flame Ionization Detection; **GC×GC–MS**, Comprehensive Two-Dimensional Gas Chromatography coupled to Mass Spectrometry; **GC-FID**, Gas Chromatography coupled to Flame Ionization Detection; **GC×GC**, Comprehensive Two-Dimensional Gas Chromatography; **LC**, Liquid Chromatography; **LC-GC-FID**, Liquid Chromatography–Gas Chromatography coupled to Flame Ionization Detection; **LOQ**, Limit of Quantification; **MAE**, Microwave-Assisted Extraction; **MAS**, Microwave-Assisted Saponification; **m-CPBA**, meta-Chloroperoxybenzoic Acid; **MNs**, Methylnaphthalenes; **MOAH**, Mineral Oil Aromatic Hydrocarbons; **MOH**, Mineral Oil Hydrocarbons; **MOSH**, Mineral Oil Saturated Hydrocarbons; **MS**, Mass Spectrometry; **PAC**, Polycyclic Aromatic Compounds; **PFA**, Performic Acid; **POSH**, Polyolefin Oligomeric Saturated Hydrocarbons; **RGB**, Red-Green-Blue; **TBB**, Tri-tert-butylbenzene; **WAC**, White Analytical Chemistry.

[*] Corresponding authors.

E-mail addresses: jose.bernal@uva.es (J. Bernal), adrian.fuente.ballesteros@uva.es (A. Fuente-Ballesteros).

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mg/kg, with both MOSH and polyolefin oligomeric saturated hydrocarbons (POSH) identified. A comparison between total and surface MOSH/POSH suggested that most of the contamination originated from pre-packaging sources. Biedermann et al. [6] analysed rice and infant formula. Rice contamination was frequently associated with batching oils derived from crude mineral oil distillates, meaning that rice may contain MOAH with three or more aromatic rings. In contrast, the source of contamination in infant formula remained unclear.

Given that MOH cover a wide molecular weight range from relatively volatile diluents to heavier lubricating oils, they can migrate to food in different ways. These include environmental contamination from air and water, contact with machinery during harvesting and processing, and migration from food contact materials, mostly packaging [7]. Among the different exposure pathways to humans, ingestion, inhalation, and skin contact are the most studied for both MOSH and MOAH fractions [7].

Due to the large number of structurally similar compounds, MOSH and MOAH are difficult to separate and typically appear as unresolved complex mixtures, forming characteristic broad “humps” in LC–GC–FID chromatographic analysis (see Fig. 2) [1,8]. From a toxicological perspective, neither MOSH nor MOAH can be treated as uniform groups, given that variations in their composition can lead to significantly different biological effects [9–11]. Studies on MOSH have emphasized their bioaccumulation, although not all exhibit the same behaviour: compounds with fewer than 20 carbon atoms are typically absorbed and metabolized rapidly, while those with more than 35 carbon atoms have a more limited ability to cross cell membranes, reducing their bioavailability. For this reason, the MOSH fraction most likely to accumulate in the human body falls within the C20 to C35 range [11]. MOAH compounds, on the other hand, are generally not bio-accumulative and are quickly catabolized through oxidation pathways. However, during this process, reactive intermediates such as epoxides can form, which is concerning for non- or weakly alkylated polycyclic aromatic compounds (PAC) with 3 to 7 fused rings [8]. In contrast, mono- and diaromatic compounds, especially those that are highly alkylated, are considered less hazardous given that their structure limits metabolic activation [9].

In terms of sample preparation for their analysis, the most applied approach begins with a solvent-based extraction, typically using hexane or mixtures of this solvent with isopropanol [5] or dichloromethane [12]. This is followed by additional purification steps such as saponification, treatment with activated alumina, or epoxidation, when necessary [13]. The selection of these steps will generally depend on the amount and kind of interferents. Clean-up strategies are chosen to eliminate specific interferences, such as triglycerides, olefins, and n-alkanes, each removed by saponification, epoxidation, and alumina treatments, respectively. For example, Albert et al. [14], used 15 mL of n-hexane and 15 mL of ethanol for the initial extraction of MOAH from coconut oil. A 10 mL aliquot of this solution was saponified using aqueous KOH and shaking at 60 °C for 30 min. After saponification, 5 mL of n-hexane and 5 mL of a 1:1 water and ethanol mixture were added, and then the aqueous phase was discarded. For MOSH determination, this extract was passed through a glass column filled with 10 g of activated alumina, 3 g of silica gel, and 1 g of sodium sulphate. The column was eluted with 25 mL of n-hexane, and the eluate was concentrated under nitrogen and reconstituted to 1 mL. For MOAH determination, a similar column containing 3 g of silica gel and 1 g of sodium sulphate was employed. The extract was eluted with 15 mL of a mixture of hexane and dichloromethane (7:3, *v*/v), concentrated, and then epoxidized with 1 mL of ethanolic meta-chloroperoxybenzoic acid (m-CPBA) at 40 °C for 20 min. The reaction was quenched with 2 mL of aqueous solution of sodium thiosulfate and sodium carbonate and 500 μL of ethanol. After shaking and separation, the hexane phase was mixed with sodium sulphate and injected into the chromatographic system.

Given the typically high solvent usage, elevated temperatures, use of non-green solvents, and the overall complexity of many sample preparation procedures in terms of the number of steps, it becomes relevant to evaluate these methods using some metrics. Assessing them using both green [15] and blue [16] analytical tools is important to identify their strengths and weaknesses, with the ultimate goal of guiding the development of more sustainable and applicable alternatives for future applications.

To the best of our knowledge, only three reviews published up to 2020 have addressed sample preparation for MOH in food matrices [3,7,17]. In other cases, the focus has been placed on chromatographic techniques [18], but no recent works specifically addressing sample preparation procedures for the determination of MOH in food matrices are available. The relevance of our work lies in the fact that the study of these contaminants is a hot topic nowadays. For this reason, having up-to-date information on sample preparation for MOH analysis is important to improving food safety. Despite the fact that no major changes have been made in the last five years regarding the analysis of MOH, there is a growing scientific and regulatory interest in developing safer and faster workflows that comply with the European recommendations. Therefore, a review of current sample preparation strategies is timely and necessary to identify methodological gaps, highlight opportunities for greener alternatives, and discuss the current lack of harmonized analytical workflows. The wide variability in sample preparation and clean-up approaches across studies hinders reliable cross-comparison of results and emphasizes the need for greater standardization in MOH analysis.

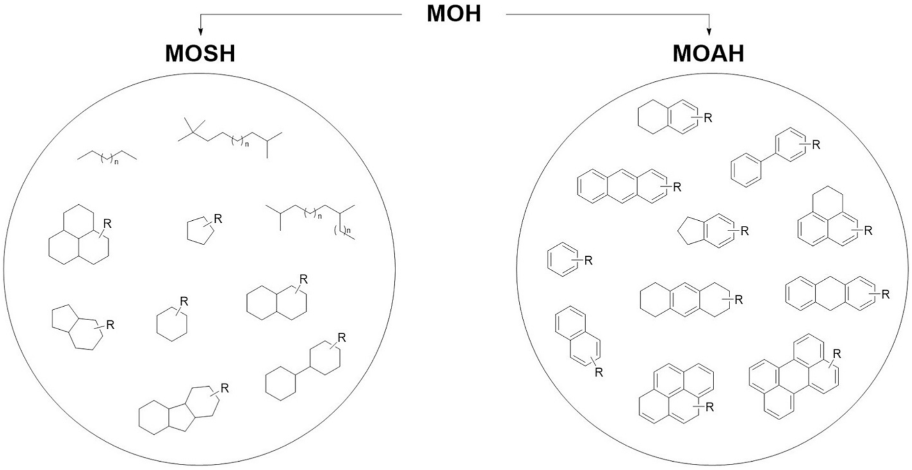

Fig. 1. Representative structures of MOSH and MOAH compounds.

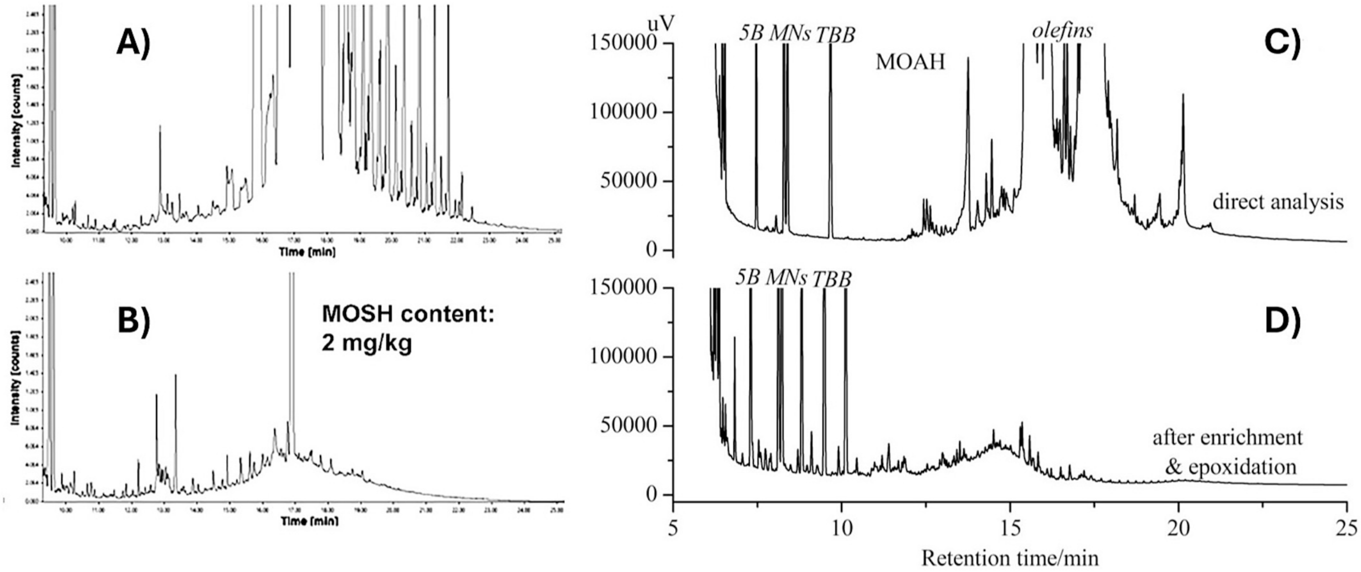

Fig. 2. LC-GC-FID chromatograms showing: A) MOSH hump in sunflower oil; B) MOSH hump after Alox treatment in previous sunflower oil (reprinted with permission from [14], copyright 2022 ACS). C) MOAH hump with olefin interference in milk powder product; D) MOAH hump after enrichment and epoxidation in previous milk powder product (reprinted with permission from [5], copyright 2020 Elsevier).

2. Methodology of literature review

This review explores the latest trends in sample preparation methods for the analysis of MOH in food matrices, a “hot topic” of growing interest in analytical chemistry since the first studies on these compounds were reported in 1989 [19]. A comprehensive literature search was conducted using several databases, including Scopus, Web of Science, ScienceDirect, and Google Scholar, using the following keyword combinations: (1) “mineral” AND “oil” AND “saturated” AND “hydrocarbons” AND “MOSH” AND “food”; (2) “mineral” AND “oil” AND “aromatic” AND “hydrocarbons” AND “MOAH” AND “food”. A three-step selection approach was applied based on predefined inclusion and exclusion criteria. Only studies published in English between 2020 and 2025 were considered. In step A, records were screened based on title, abstract, and keywords. In step B, duplicate records and review articles were excluded. In step C, the remaining publications were assessed through full-text evaluation to determine their relevance, adequacy, and the availability of sufficient sample preparation data in relation to the scope of this work. Based on this workflow, the distribution of the selected studies was analysed according to the type of food matrix investigated. As summarised in Table 1, MOH analysis has been reported for a wide range of food matrices; however, edible oils such as olive, sunflower, and rapeseed oils are by far the most frequently studied. These are followed by other fat-rich matrices like milk-derived products (including infant formula and powdered milk), as well as butters and animal fats. This preference for fatty foods is linked to the chemical nature of MOH, which are highly lipophilic. Their strong affinity for lipid phases means they are more likely to accumulate and persist in fat-containing products. Therefore, fatty matrices not only pose a higher contamination risk but also serve as critical points for monitoring MOH exposure in the food chain.

As mentioned in the introduction, MOAH are generally considered to pose higher toxicological risks compared to MOSH. PACs with three to seven fused aromatic rings have been associated with carcinogenic effects and potential harm to prenatal development [9–11]. Due to these significant health concerns, the accurate detection and quantification of MOAH in food matrices becomes especially important. According to current European regulations, MOAH must be detected at the limit of quantification (LOQ), which highlights the need for adequate and optimized sample preparation procedures [9–11]. MOAH are consistently examined in all reviewed studies, while MOSH are analysed less frequently, appearing in only 73% of the included publications. This prevalence of studies focusing on MOAH can be attributed to their higher toxicological relevance and stricter regulatory attention. In contrast to MOSH, which are generally regarded as less harmful and tend to accumulate in human tissues without immediate toxic effects, MOAH include polyaromatic compounds with potential genotoxic and carcinogenic properties. Consequently, regulatory bodies such as EFSA and the European Commission have prioritized the monitoring of MOAH to ensure that their presence in food is kept as low as reasonably achievable, driving a stronger analytical focus on this fraction [9].

In terms of detection, flame ionization detection (FID) is by far the most extensively used approach in MOH analysis, due to its uniform mass response across a broad range of hydrocarbons. This characteristic makes it advantageous over mass spectrometry (MS), which often faces calibration challenges [20]. However, FID lacks both sensitivity and selectivity, requiring a robust sample preparation step to ensure that only the target fractions can reach the detector without interference. Furthermore, the separation power of current liquid chromatography–gas chromatography coupled to flame ionization detection (LC-GC-FID) setups remains inadequate for detailed compound-level characterization, especially in complex mixtures. This is causing an increase in the use of advanced chromatographic techniques like comprehensive two-dimensional gas chromatography (GC × GC) [9].

3. Sample preparation strategies for MOH analysis

The sample preparation workflow prior to chromatographic analysis can vary depending on the sample and the type of analysis required. Sometimes a straightforward hexane extraction is sufficient [21], but in other cases, more complex steps are required. The effectiveness of these depends on various factors, such as the ability to fully extract compounds from dry samples, which is influenced by their permeability and their ability to swell and disperse in said solvent. This situation often involves a multi-step sample preparation that, besides the initial extraction, may include enrichment steps, and/or selective removal of interferents from both the MOSH and MOAH fractions, making the procedure more labour-intensive [2,3]. Regarding these sample preparation issues, the latest report published by the European Commission [22] includes a decision tree (see Fig. 3), establishing when additional

Table 1
Representative works for the analysis of MOSH and MOAH in food matrices.

Article ID	Matrix	Target analytes	Extraction*	Saponification	Alox	Epoxidation	Enrichment	Detection	Ref.
1	Vegetable oils	MOSH & MOAH	10 mL n-Hx	10 mL KOH in MeOH (MAS)	Cartridge	500 uL m-CPBA in EtOH	Reconcentration	LC-GC-FID & GC × GC-FID/MS	[36]
2	Vegetable oils	MOSH & MOAH	10 mL n-Hx	10 mL KOH in MeOH (MAS)	No	500 uL m-CPBA in EtOH	No	LC-GC-FID	[46]
3	Batching oil, rice, infant formula	MOAH	20 mL n-Hx (rice), 5 mL EtOH (infant formula), 10 mL n-Hx (olive oil)	5 mL KOH in H_2O, ultrasound (infant formula)	No	3 mL m-CPBA in EtOH (infant formula)	Reconcentration (rice and infant formula)	LC-GC-FID	[27]
4	Vegetable oils and cocoa butter	MOSH & MOAH	10 mL n-Hx:EtOH (1:1, *v*/*v*)	2 mL KOH in H_2O	No	PFA	Reconcentration	LC-GC-FID	[25]
5	Vegetable oils and fats	MOAH	IPA, vortex and ultrasound	No	No	No	Low temperature	LC-FLD	[26]
6	Vegetable oils	MOSH & MOAH	15 mL n-Hx, 15 mL EtOH	3 mL KOH in H_2O	Column	1 mL m-CPBA in EtOH	Reconcentration	LC-GC-FID	[14]
7	Vegetable oils	MOSH & MOAH	15 mL n-Hx, 15 mL EtOH (MAE)	10 mL KOH in MeOH	Cartridge	500 uL m-CPBA in EtOH	No	LC-GC-FID	[30]
8	Cocoa powder	MOSH & MOAH	10 mL n-Hx	10 mL KOH in MeOH (MAS)	No	500 uL m-CPBA in EtOH	No	LC-GC-FID	[47]
9	Vegetable oils	MOAH	2 mL n-Hx	No	No	No	No	LC-GC × GC-FID/MS	[21]
10	Vegetable oils	MOAH	10 mL Hx	10 mL KOH in EtOH:H_2O (1:1, v/v) (MAS)	No	1 mL m-CPBA in EtOH	No	LC-GC-FID	[32]
11	Milk powder products	MOSH & MOAH	60 mL n-Hx:IPA (3:1, *v*/*v*)	No	No	0.5 mL m-CPBA	Silica gel glass column	LC-GC-FID	[5]
12	Basil pesto and its ingredients	MOSH & MOAH	10 mL n-Hx	10 mL KOH in MeOH (MAS)	Cartridge (basil and basil pesto)	0.5 mL m-CPBA in EtOH	No	LC-GC-FID	[34]
13	Vegetable oils	MOSH & MOAH	2 mL n-Hx:DCM (70:30, v/v)	No	Column	1 mL CPBA	Silica gel column	LC-GC-FID	[12]
14	Vegetable oils	MOSH & MOAH	700 μL n-Hx	No	Column	0.5 mL m-CPBA (MOAH)	No	LC-GC-FID	[37]
15	Vegetable oils	MOSH & MOAH	1.3 mL n-Hx	No	No	1 mL m-CPBA in EtOH	Reconcentration	GC-FID	[48]
16	Vegetable oils	MOAH	10 mL n-Hx	10 mL KOH in MeOH (MAS)	No	500 uL m-CPBA in EtOH	No	LC-GC-FID	[49]
17	Vegetable oils	MOSH & MOAH	n-Hx	Yes	Column	1 mL mCBPA in EtOH or DCM	Silica gel column (MOAH)	LC-GC-FID	[40]
18	Vegetable oils	MOAH	15 mL n-Hx	15 mL KOH in EtOH:H_2O (1:1, v/v)	No	No	No	LC/GC × GC-FID/MS	[43]
19	Rice, infant formula	MOAH	20 mL Hx (rice)	5 mL KOH in H_2O (infant formula)	No	0.5 mL m-CPBA in EtOH (infant formula)	Silica gel column (infant formula)	GC × GC-FID	[6]
20	Vegetable oils	MOSH & MOAH	No	1 mL 5% KOH in EtOH:H_2O (1:9, v/v), (MOAH)	Cartridge	No	Reconcentration (MOSH)	Ag–pSPE–UV/FLD & GC-FID	[35]
21	Meat	MOSH & MOAH	10 mL n-Hx	10 mL KOH in EtOH:H_2O (1:1, v/v) (MAS)	No	1 mL m-CPBA in EtOH	No	LC/GC × GC-FID/MS	[50]
22	Complementary food for infants	MOSH & MOAH	20 mL n-Hx (pureed or paste foods, noodles, chewing stick and biscuit), 30 mL n-Hx (rice flour)	No	Column	0.5 mL m-CBPA	silica gel column (chewing stick and biscuit), reconcentration (all)	LC-GC-FID	[39]
23	Infant formula	MOSH & MOAH	100 mL Hx	50 mL KOH in EtOH:H_2O (1:1, v/v)	No	No	Reconcentration	GC-FID/MS	[29]
24	Vegetable oils	MOSH & MOAH	10 mL n-Hx	No	No	1 mL m-CPBA in EtOH	No	GC × GC–MS	[51]
25	Vegetable oils, rice, flours, sugar, salt, infant formula, powdered milk, infant biscuits	MOSH & MOAH	1–50 mL n-Hx	No	No	No	No	LC-GC-FID	[52]
26	Infant formula, vegetable oils	MOSH & MOAH	30 mL n -Hx:EtOH (1:1, v/v), 10 mL n-Hx (vegetable oil), 30 mL n-Hx (infant formula)	Yes	Column	1 mL m-CPBA (infant formula), m-CPBA (vegetable oils)	Reconcentration	LC-GC × GC-FID/MS	[38]

[*] *Note*: extraction and saponification are presented in separate columns to indicate to the reader the solvent used and its amount during the extraction step; however, both steps are often combined [30,36]. **DCM**: dichloromethane; **EtOH**: ethanol; **H_2O**: water; **Hx**: n-hexane; **IPA**: isopropanol; **KOH**: potassium hydroxide; **MAS**:

microwave-assisted saponification; **m-CPBA**: meta-chloroperoxybenzoic acid; **MeOH**: methanol; **MOAH**: mineral oil aromatic hydrocarbons; **MOSH**: mineral oil saturated hydrocarbons; **PFA**: performic acid.

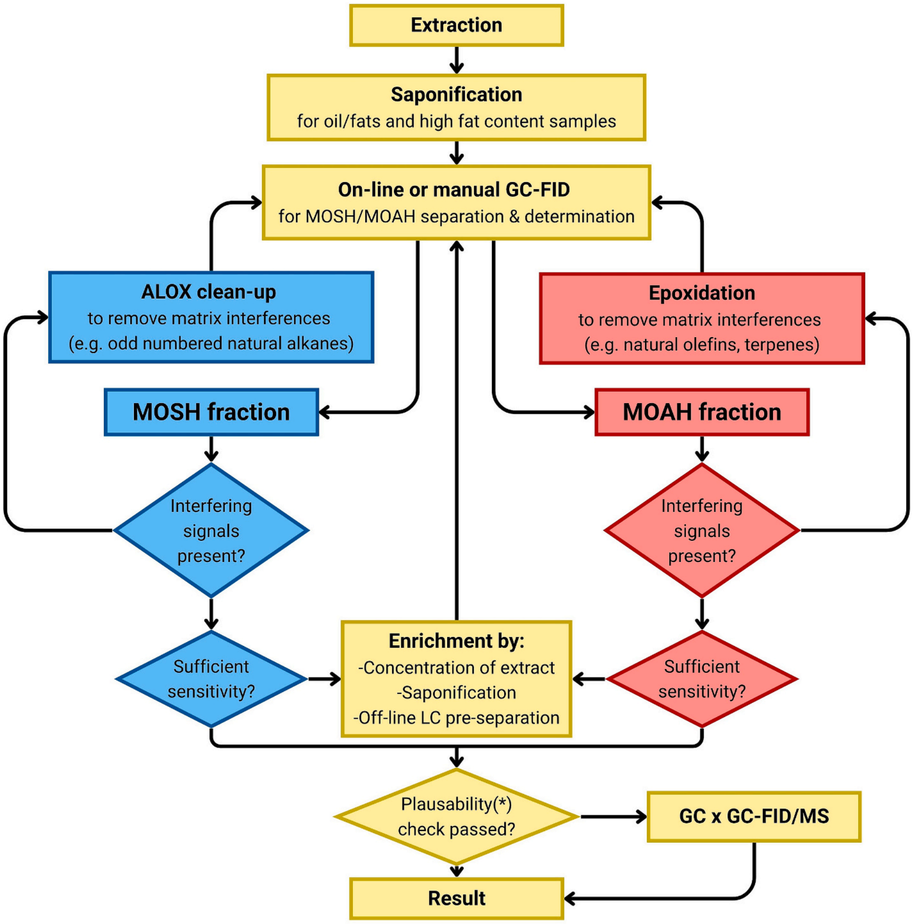

Fig. 3. Decision tree on the use of additional procedures for MOH analysis. Adapted from [22].

procedures should be applied. These include clean-up using aluminium oxide to eliminate naturally occurring n-alkanes from the MOSH fraction, epoxidation to remove biogenic olefins from the MOAH fraction and other enrichment steps to meet the required LOQ. These auxiliary techniques are further explained in their respective subsections, following the order established in the aforementioned decision tree. Accordingly, the articles selected for this review have been compiled in Table 1. For each publication, the presence of auxiliary steps, the food matrix analysed, the target analytes, and the type of detection employed are indicated.

The key stages of sample preparation for the analysis of MOH in food matrices are presented below: (a) initial extraction, (b) saponification, (c) activated alumina clean-up, (d) epoxidation, and (e) other enrichment steps. As has been mentioned before, it is important to note that not all of these steps are mandatory; their inclusion depends on the specific analytes targeted for analysis (MOSH and MOAH) and the interferences associated to each.

3.1. Challenges in sample preparation for MOH analysis

MOH analysis is a hard nut to crack as many challenges have been reported by authors in terms of sample preparation. If interfering substances are not effectively eliminated, they must be excluded from the integration of the chromatographic hump. However, there is no universally standardized approach for doing so, which can result in inconsistencies in interpretation. Conversely, inadequate pre-treatment may inadvertently remove a portion of the target analytes, potentially causing an underestimation of their actual concentration [3]. In addition, the detection of MOH-like substances that may migrate from food contact materials can further complicate data interpretation [8]. These interfering substances may include natural n-alkanes which are present in plant-derived materials and foods. They are predominantly odd-numbered, typically ranging from C23 to C35, and generate well-defined, isolated peaks during chromatographic analysis. Nonetheless, when found in high concentrations, these compounds can overload the gas chromatography (GC) column, hindering accurate MOSH detection. Another group of potential interferences are POSH, which originate from certain food contact materials, such as heat-sealable layers in aluminium packaging. Because they are composed mainly of branched hydrocarbons, POSH often co-elute with the MOSH fraction during liquid chromatography (LC) separation [2] but, in some cases, it is possible to distinguish them from MOSH based on their chromatographic behaviour [23]. Additionally, the use of plastics derived from olefins (such as polypropylene and polyethylene) can appear in both the MOSH or MOAH fraction due to their wide range of polarity. In particular, edible oils rich in squalene may contain levels of it so high that they surpass the detection thresholds for MOAH by several orders of magnitude [24]. Another example are lipids like triglycerides, that although typically elute after the MOAH region, when present in large amounts, they can overload the LC column, thereby reduce the efficiency of the stationary phase and compromising the separation of MOSH and MOAH fractions. Finally, aromatic oligomers, which are commonly found in adhesives, can also interfere with the analysis due to their structural similarities to MOAH compounds [2].

According to the previously stated, to ensure the highest accuracy in MOH analysis, it is essential to minimize the extent of sample handling. Regular blank tests should be conducted, and solvent purity must be verified. Additionally, strict protocols should be followed to confirm the cleanliness of any reused glassware, and the use of plastic equipment should be avoided due to the risk of contamination [3]. As for the analysis, fully integrated LC-GC-FID systems are favoured over offline methods. These hyphenated platforms reduce the risk of cross-contamination, offer complete automation, and ensure the entire target fraction is transferred to the gas chromatography coupled to flame ionization detection (GC-FID) for analysis [8].

3.2. Initial extraction

As shown in Table 1, in most articles, an initial extraction of the analytes is required, typically through either liquid-liquid or solid-liquid extraction techniques, depending on the food matrix. The use of a single extraction solvent, most commonly n-hexane, has been demonstrated to be effective for a wide range of both dry and wet food matrices. However, in certain works, a more exhaustive extraction has been implemented using either alternative solvents or mixtures. Some examples include combinations of hexane with ethanol [25], isopropanol [5], or dichloromethane [12], or single solvents such as isopropanol [26] or ethanol [27]. MOSH and MOAH are highly non-polar substances, a fact which makes the use of a solvent like n-hexane appropriate. The widespread use of *n*-hexane and other non-green solvents should therefore not be interpreted as a disregard for sustainability considerations, but rather as a consequence of the stringent analytical requirements imposed by MOH analysis. Efficient extraction of broad, unresolved hydrocarbon mixtures at trace levels, together with the need to meet low LOQs and ensure compatibility with downstream LC, GC-FID analysis, currently limits the applicability of greener solvent systems in routine workflows.

These solvents can be assessed in terms of greenness using the CHEM21 solvent selection guide, which classifies them based on their environmental impact and hazard profile [28]. Hexane, the most widely used solvent for these analyses and often in large quantities (up to 100 mL [29]), is classified as hazardous, and so is dichloromethane. In contrast, isopropanol and ethanol are recommended by the guide due to their safer environmental and health profiles.

In order to enhance the extraction process, some authors have explored the role of including various types of equipment in the sample preparation. While most studies report the use of agitation at either ambient temperature or around 60 °C, some authors have specifically indicated the use of microwave-assisted extraction (MAE) or ultrasound in samples that needed it after vortexing. This is particularly relevant for complex or solid matrices (e.g. processed meats), where mineral oils may be more strongly embedded within the matrix or tightly bound to lipids and proteins. MAE improves extraction efficiency by heating the sample, causing matrix disruption and enhancing solubilization of MOH. For example, Menegoz Ursol et al. [30] analysed extra-virgin olive oils employing saponification at 120 °C for 20 min but simultaneous extraction with hexane. Four different extraction protocols were tested, three of which included MAE. Other work [26] proposed, ultrasonication as it helps disintegrate the matrix and improves solvent penetration; however, the authors stated that they used to improve solubilization.

3.3. Saponification

Saponification involves the hydrolysis of triglycerides by hydroxide ions, breaking the ester bond and resulting in glycerol and free fatty acids [31]. In MOH analysis, this step has a dual purpose: it enables the simultaneous extraction of both MOSH and MOAH (which stay in the unsaponifiable fraction) while effectively eliminating triglycerides, which otherwise can constrain the sensitivity of the analysis. By removing triglycerides through saponification prior to injection, the permissible sample load can be increased by up to fivefold, leading to a proportional reduction in the LOQ. Despite its advantages, this step introduces an added source of variability. Specifically, it can lead to inconsistencies in the distribution of internal standards used for MOAH quantification, such as tri-tert-butylbenzene (TBB) and 1- and 2-methylnaphthalenes (MNs), as they can be lost at different rates [32].

In the reviewed cases where saponification was employed, KOH was applied as the base, most often dissolved in methanol or ethanol-water. The reaction was facilitated by agitation, ultrasonic treatment and/or elevated temperatures ranging from 60 to 120 °C. While saponification is sometimes performed after the initial dissolution commented in the previous subsection, several studies presented in Table 1 integrated both steps simultaneously. This has led to the development of microwave-assisted saponification (MAS) [33].

3.4. Activated aluminium oxide “Alox”

Following the previously described procedures, which are generally standard for the analysis of both MOSH and MOAH, additional purification steps may be required. For MOSH, the principal issue found is the presence of naturally occurring n-alkanes, typically characterised by a dominance of odd-numbered chains within the C21 to C33 range. These compounds may interfere with accurate MOSH quantification when present in high concentrations, particularly when their peaks mask the MOSH hump or overload the GC column. In such instances, the auxiliary clean-up step using activated aluminium oxide, commonly referred to as “Alox” becomes necessary. Otherwise, the n-alkanes' contribution can be excluded during hump integration. In several of the reviewed articles, aluminium oxide was packed into glass cartridges [34–36], pre-treated by overnight activation at 500 °C and conditioned with n-hexane prior to use. Elution was performed with 5 mL of n-hexane, and the resulting extract was injected into the chromatographic system. In other cases, glass columns were employed instead, including setups in online configurations [37]. Additionally, some authors incorporated additional materials such as silica gel [12,14,38–40] and sodium sulphate [12,14,38,40] alongside Alox to enhance the purification process in said column. The silica layer is mainly employed to retain remaining sterols and traces of polar substances such as alkaline soaps. This step is crucial because the ability of activated alumina to retain long-chain n-alkanes is compromised by the presence of more polar compounds. Substances such as dichloromethane, ethanol, or even minor amounts of oxidised n-hexane can adversely affect the retention efficiency of Alox, reducing its effectiveness in purifying the MOSH fraction [40]. As shown in **Fig. 2A** and **B**, alumina pre-treatment can reduce the content of n-alkanes, particularly those with carbon numbers above C24. However, the retention is not entirely selective to linear alkanes, and as molecular weight increases, a notable portion of the MOSH fraction may also be held back [2]. It is important to note that polar substances such as lipids or moisture can irreversibly compromise the adsorptive performance of the stationary phase, and so it is critical to ensure their removal prior to the application of this treatment of the MOSH fraction. When analysing both MOSH and MOAH fractions, some authors separated aliquots of the same sample with distinct purification steps, such as applying Alox clean-up exclusively for the MOSH fraction and epoxidation for the MOAH fraction [14,38]. In other cases, Alox treatment is applied selectively, and when necessary, as highlighted in the decision tree (see Fig. 3). For example, it must be used when the concentration of naturally occurring n-alkanes is sufficiently high to interfere with the proper visualisation of the MOSH hump in the chromatogram [30,39]. Additionally, online configurations, such as columns are sometimes utilised to streamline the purification process [37].

3.5. Epoxidation

Epoxidation involves the transformation of an alkene, acting as the nucleophile, into an epoxide through its reaction with an oxidising agent, the electrophile, such as a peracid, hydrogen peroxide, or an organic hydroperoxide [41]. In the context of MOH analysis, this reaction serves as an auxiliary method specifically aimed at purification. These olefins may originate from the food matrix itself, including compounds such as carotenoids, squalene, sterenes, and their derivatives, or be formed during fat and oil refining processes (see Fig. 4). Indeed, squalene and carotenoids are almost completely removed by epoxidation; however, residual interferences may still persist. These have been attributed to sterenes and isomerised squalene formed under the conditions of oil refining. Such interferences are typically absent in non-refined oils, while they become evident in refined matrices (see Fig. 4). Although in certain cases their impact can be minimized by excluding their peaks from integration, again, complications arise when these compounds overload the GC column, in some cases forming humps that overlap with the MOAH one [8] (see Fig. 2C). This is equivalent to how high levels of alkanes interfere in the MOSH fraction, as explained in the previous subsection. The reaction converts the original alkene into an epoxide, increasing the compound's polarity. As a result, the newly formed derivative exhibits greater retention during LC, causing it to elute later than the MOAH fraction and thereby reducing potential interferences in the target region, as shown in Fig. 2D in comparison to Fig. 2C. It must be mentioned that this separation of the olefins from the MOAH fraction becomes feasible once at least one double bond within the olefin molecule has been epoxidized, complete or multiple epoxidations is not needed [25]. In addition to this, other chemical strategies such as hydroboration and the bromohydrin reaction have been explored as potential methods for eliminating this kind of interferences, but both reactions failed for removal of polyunsaturates [42].

As shown in Table 1, epoxidation is commonly applied, appearing in more than 75% of the studies reviewed. The decision to use it or not is not based on matrix type, nor on loss of MOAH as it cannot be known before analysis. This depends on the level of interferences relative to the degree of contamination.

The most typical epoxidizing agent for this procedure is m-CPBA dissolved in ethanol. Due to the instability of the peracid in said solvent, the solution should be kept refrigerated, and new solutions need to be regularly prepared. Over prolonged automated sample preparation runs, variations in performance may arise and can require correction by refining the integration parameters to minimize interference effects [24]. This ethanolic solution is typically added to a pre-existing hexane extract, resulting in a hexane and ethanol mixture. While dichloromethane was initially used as the solvent [1], it was later replaced by Nestola et al. [42] who used the previous mixture to slow down the reaction kinetics. This adjustment enabled the reaction to proceed at room temperature without requiring the addition of polyunsaturated oils, and it simplified the solvent exchange process, greatly facilitating full automation of the step. Despite these practical improvements, the epoxidation process still has analytical challenges as it can result in a loss of 20–35% of MOAH content, and the removal of the interfering material may remain incomplete, particularly for refined oils/fats [24]. This behaviour has also been reported in comparative studies showing that both protocols provide comparable MOAH recoveries and interference removal efficiencies despite their different reaction conditions [24]. Even the hexane and ethanol system performs similarly to the initial dichloromethane one in terms of MOAH losses and the effectiveness of interference removal. It should be noted that one of the works [25] included in the review uses a different epoxidizing agent, namely performic acid (PFA) generated in situ by the reaction of formic acid and hydrogen peroxide and it was more effective than m-CPBA.

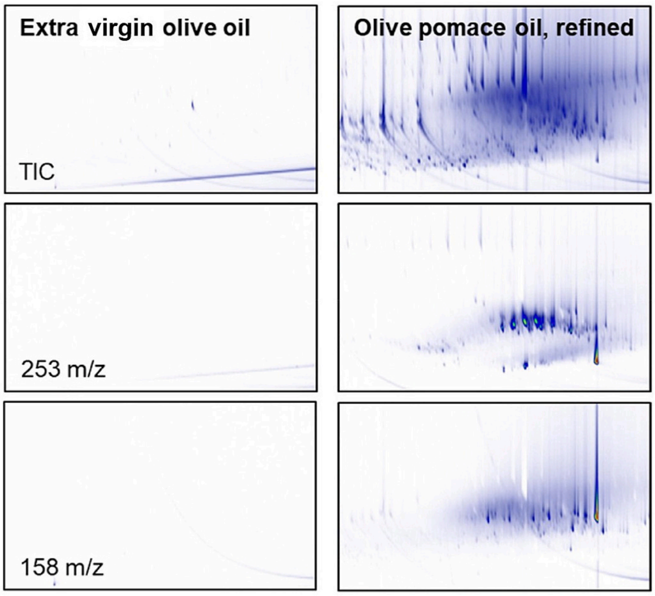

Fig. 4. GC × GC–MS plots comparing non-refined extra virgin olive oil (left) and strongly refined olive pomace oil (right), highlighting the presence of interfering material formed during the refining process. Reprinted with permission from [24], copyright 2020 Elsevier.

With the objective of avoiding epoxidation steps, a recent study by Gorska et al. [43] proposed a novel clean-up approach based on LC fractionation on silica. They stated that MOAH losses are particularly pronounced when using epoxidation protocols with faster reaction kinetics, such as those involving PFA, and that they fluctuate depending on the composition, which remains difficult to predict. Additionally, common internal standards as mentioned in the saponification step (e.g. 2-MN and TBB) exhibit differing degrees of loss during epoxidation. In the study, the sample was divided into three aliquots after saponification. Two were subjected to "traditional" epoxidation methods (using m-CPBA and PFA), and one underwent the LC purification. This chromatographic method successfully removed interferences such as squalene, carotenes, and their derivatives, while avoiding the inconsistent losses observed with epoxidation. The LC method yielded a MOAH recovery of 94% across various edible oils, including coconut, palm, sunflower, and olive oils, regardless of the MOAH source or concentration.

In this regard, it is recommended by EFSA to reserve epoxidation for situations where it is required, due to the associated loss of MOAH content [9]. A more cautious approach would involve initially analysing the food products without applying epoxidation, in order to determine whether it is truly needed, paying special attention in cases of refined edible oils or fats. When uncertainty remains, comprehensive two-dimensional gas chromatography with flame ionization detection (GC × GC-FID) can be employed to confirm their presence, with additional help of comprehensive two-dimensional gas chromatography coupled to mass spectrometry (GC × GC–MS) [24].

3.6. Other enrichment steps

After completing the previously described preparation steps, most authors proceed with an additional enrichment step, which typically involves total or partial evaporation and then redissolving the extract prior to injection into the chromatographic system. This is a common analytical practice to increase the concentration of analytes in a sample, thereby enhancing sensitivity and allowing for accurate quantification, especially when dealing with trace levels or low-abundance compounds. Evaporation is often performed under a nitrogen stream to control solvent removal and reduce exposure to ambient laboratory air; however, this step may also represent a potential source of contamination if gas purity and system cleanliness are not carefully controlled.

When considering food matrices with varying fat content, the clean-up strategy must be adapted accordingly. In samples with low fat content, triglycerides can be efficiently removed simply using silica, without the need for additional pre-treatment steps. However, in high-fat matrices, it becomes necessary to significantly reduce the triglyceride content to achieve the desired LOQ before LC–GC–FID analysis. For instance, Liu et al. [39] reported that in samples such as chewing sticks and biscuits, the fat content exceeded 4%, so a chromatographic column containing silica gel had to be employed for clean-up. The silica was prepared as a solid slurry using 10 mL of a solvent mixture composed of n-hexane and dichloromethane (8:2, *v*/v), which was later used to elute the sample for its subsequent concentration by rotary evaporation.

In this work, enrichment (understood mainly as solvent evaporation and concentration of the extract) and saponification are discussed in different sections; however, it is important to note that saponification is indeed used as an enrichment step to decrease the LOQ.

3.7. Discussion

Although a wide range of sample preparation strategies for MOH analysis has been reported, recent advances are primarily associated with improvements in workflow design rather than the introduction of entirely new extraction concepts. Traditional solvent-based extraction remains the cornerstone of MOH analysis; however, recent studies focus on optimizing solvent composition, extraction conditions, and clean-up sequences to better address complex and fat-rich matrices. Compared with earlier methodologies, recent sample preparation approaches show a differentiation between mandatory and auxiliary steps. Decision-tree-based workflows and selective application of clean-up procedures have reduced unnecessary treatments, thereby minimizing analyte losses and improving reproducibility. In particular, the integration of extraction and saponification, especially through microwave-assisted protocols, represents an improvement in terms of extraction efficiency and processing time when compared to conventional multi-step procedures, albeit at the cost of increased sensitivity to experimental conditions.

Another notable improvement concerns the treatment of matrix-derived interferences. While activated aluminium oxide and epoxidation remain widely used, recent studies place greater emphasis on understanding their limitations, such as non-selective retention of MOSH fractions or partial losses of MOAH. This has led to the exploration of alternative clean-up strategies, including chromatographic fractionation on silica, which offers improved selectivity and recovery in certain matrices while avoiding chemical derivatization.

From a comparative perspective, recent workflows tend to favor automation and online configurations, particularly LC, GC–FID systems, over offline procedures. These approaches reduce manual handling, lower the risk of contamination, and improve interlaboratory reproducibility. However, solvent consumption and the use of hazardous chemicals remain unchanged, indicating that progress in terms of greenness has been more gradual and matrix-dependent.

4. Green and blue analytical evaluation of sample preparation methods

The increasing importance of white analytical chemistry (WAC) has driven the development of tools to assess the performance, eco-friendliness and practicality of the methods following the red-green-blue (RGB) model, respectively [44]. As this review is focused on the sample preparation step, the analytical greenness metric for sample preparation (AGREEprep) [15] and the blue applicability grade index (BAGI) [16] were selected. AGREEprep is a green metric used to evaluate how environmentally friendly sample preparation steps are, with a score that ranges from 0 to 1, the higher indicating a greener approach. BAGI focuses on the practical aspects of analytical methods, with 60 points considered the minimum threshold for a method to be classified as blue. Both tools are supported by freely available software and rely on pictograms and numerical scores. All the reviewed articles were evaluated using the previously mentioned metrics, and the resulting scores were used to construct a comparative graph (see Fig. 5). The IDs for each article are specified in Table 1 for reference. It is important to note that in some cases, it was not possible to obtain specific information from the available data. In such situations, specific conventions were adopted, which are thoroughly detailed in **Table S1** to ensure transparency and facilitate reference. Although these efforts aim to provide the most accurate estimates possible, minor variations in scores and results may occur if calculations are performed with full access to data covering all experimental steps.

The scores obtained with AGREEprep (see Fig. 5A and **Table S2**) show the need for significant improvements in analytical procedures, particularly concerning the complexity of the steps involved, the intensive use of toxic organic solvents, and the generation of waste. AGREEprep scores ranged from 0.11 (ID-26) to 0.43 (ID-5). The best-performing methods were those that used small sample amounts and employed simpler experimental procedures. Although the low AGREEprep scores demonstrate the non-green character of current methods, several underlying reasons explain why these workflows remain largely unchanged. Beyond solvent choice, the properties of MOH limit the adoption of greener approaches. Their broad unresolved chromatographic distribution, lack of selective chemical handles, and frequent co-elution with matrix-derived hydrocarbons require exhaustive extraction and clean-up steps, which are difficult to reconcile with solvent-free or low-impact methodologies. As a result, greenness in MOH analysis is constrained not only by methodological inertia but by fundamental analytical requirements imposed by the target analytes themselves.

For BAGI, score values ranged from 50 to 70 (see Fig. 5B and **S1**), further emphasizing the need to improve the practical aspects of the evaluated methods. Factors such as the requirement for additional solvent evaporation and reconstitution steps, the use of sophisticated and high-cost techniques, and the need for large sample volumes negatively impacted most approaches. In contrast, simpler methods based on sample dilution followed by detection, or those employing more accessible instrumentation (as in the cases of ID-5 and ID-14), demonstrated superior performance. However, the operator skills required to manage the instruments is a key issue to consider [38]. Accordingly, BAGI metric favours methods that emphasize operational efficiency, simplicity, and reduced resource consumption. Key attributes for making MOH analyses in food more sustainable, accessible, and reproducible.

The combined AGREEprep and BAGI evaluation provides a complementary perspective on the environmental and operational weaknesses of current sample preparation workflows for MOH analysis. Low AGREEprep scores reflect a dependence on hazardous organic solvents and limited opportunities for waste reduction or solvent substitution. Although solvent consumption is fairly comparable across the articles, the green assessment can focus on the number of analyses required to provide both quantification and confirmation of the contamination [38]. At the same time, moderate BAGI scores show that improvements in greenness are often constrained by practical requirements, such as achieving low LOQs, handling complex lipid-rich matrices, and relying on established regulatory workflows. These results suggest that future developments should prioritize strategies that simultaneously reduce solvent consumption and procedural complexity without compromising analytical robustness. In particular, approaches based on miniaturization and greater automation appear as key strategies to improve both green and blue performance. The metrics also highlight that replacing individual solvents alone is unlikely to yield substantial gains unless accompanied by broader workflow redesign. Therefore, AGREEprep and BAGI not only quantify current limitations but also serve as diagnostic tools to identify where meaningful progress toward green and blue analytical workflows can be achieved.

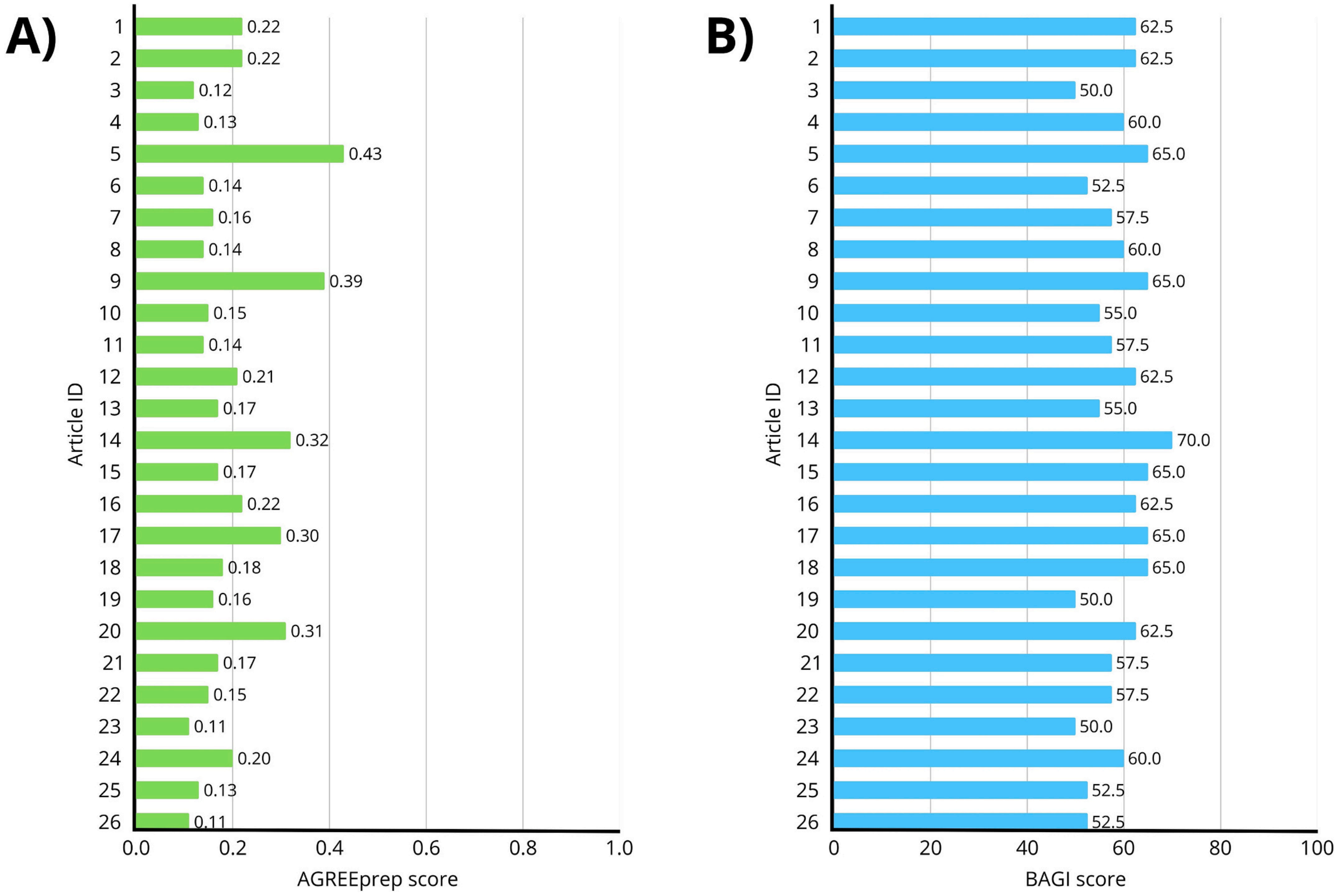

Fig. 5. Scores obtained for each reviewed article using A) AGREEprep and B) BAGI tools. The IDs for each article are specified in Table 1 for reference.

5. Perspectives

The analysis of these contaminants is a relatively recent field, leaving room for further improvement and innovation. Two key concepts in modern analytical chemistry are miniaturization and automation, particularly relevant when dealing with contaminants and their derivatives in food matrices. However, in the case of MOH analysis, miniaturization remains challenging due to the highly lipophilic nature of MOSH and MOAH, their typically low concentration levels, and the need for clean-up to remove structurally similar matrix-derived interferences. These constraints limit the direct transfer of microscale approaches commonly applied in other areas of food analysis.

Current methodologies for MOH analysis are often complex and time-consuming, often involving multiple extraction, clean-up, and concentration steps. For this reason, integrated all-in-one systems that combine extraction, purification, and preconcentration are expected to gain prominence, particularly as methods incorporating in-line or online purification are already emerging. Beyond efficiency gains, automation plays a critical role in improving reproducibility, reducing operator-dependent variability, and supporting method validation. These aspects are especially relevant for MOH analysis, where the lack of harmonized protocols has hindered interlaboratory comparability and regulatory alignment.

In terms of miniaturization, future efforts will likely focus on reducing the use of hazardous solvents and reagents by working with smaller volumes and fewer processing steps, rather than eliminating solvents altogether. Following the framework of the 12 principles of green analytical chemistry, this approach addresses principles related to reduced reagent consumption, waste minimization, and enhanced operator safety. In this regard, commonly used reagents such as m-CPBA may be replaced by less hazardous alternatives or avoided entirely through chromatographic strategies that eliminate the need for chemical derivatization, provided that analytical performance is not compromised.

The challenges associated with sample preparation, such as the removal of MOAH interferences through epoxidation, are actively driving the search for alternative procedures that achieve comparable selectivity with fewer limitations. Chronologically, m-CPBA was the first widely adopted option for olefin removal, followed by the exploration of alternative reagents (e.g., PFA) and, more recently, chromatographic LC-based separation strategies. Similarly, the initial adoption of LC, GC–FID has been complemented by comprehensive GC × GC techniques, aiming to improve the resolution of complex MOH mixtures while reducing reliance on extensive chemical pre-treatment.

In the context of food analysis, although most current studies focus on edible oils such as olive, sunflower, or rapeseed oils, it is expected that future research will address other lipid-rich food matrices (e.g., meat products, dairy products, nuts, and complex processed foods). Despite the availability of several guidelines and technical reports addressing MOH analysis, stronger regulatory and scientific collaboration is still required to harmonize criteria for chromatographic integration, interference exclusion, and data reporting [45]. Such harmonization is essential to ensure consistency, reproducibility, and reliability across laboratories and analytical workflows.

6. Conclusions

Recent advances in sample preparation for MOSH and MOAH analysis in food matrices are mainly driven by refinements in existing workflows rather than by disruptive methodological changes. Solvent-based extraction combined with selective clean-up steps remains the dominant approach, with its application strongly dependent on matrix composition and interference profile. Automation and online configurations have improved robustness and reproducibility, but challenges persist related to solvent consumption, procedural complexity, and MOAH losses during clean-up. The combined green and blue assessment reveals environmental and practical limitations of current methods, highlighting the need for more harmonized and efficient analytical workflows for routine MOH analysis.

CRediT authorship contribution statement

Ana Jano: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation. **Ana M. Ares:** Writing – review & editing, Visualization, Supervision, Investigation, Conceptualization. **Floriatan Santos Costa:** Writing – review & editing, Visualization, Methodology. **Jorge A. Custodio-Mendoza:** Writing – review & editing, Visualization, Methodology. **José Bernal:** Writing – review & editing, Visualization, Supervision, Methodology, Conceptualization. **Adrián Fuente-Ballesteros:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.microc.2026.118083.

Data availability

Data will be made available on request.

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