

Review

Analysis of Volatiles in Food Products

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Abstract: The evaluation of volatiles in food is an important aspect of food production. It gives knowledge about the quality of foods and their relationship to consumers' choices. Alcohols, aldehydes, acids, esters, terpenes, pyrazines, and furans are the main chemical groups that are involved in aroma formation. They are products of food processing: thermal treatment, fermentation, storage, etc. Food aroma is a mixture of varied molecules. Because of this, the analysis of aroma composition can be challenging. The four main steps can be distinguished in the evaluation of the volatiles in the food matrix as follows: (1) isolation and concentration; (2) separation; (3) identification; and (4) sensory characterization. The most commonly used techniques to separate a fraction of volatiles from non-volatiles are solid-phase micro-(SPME) and stir bar sorptive extractions (SBSE). However, to study the active components of food aroma by gas chromatography with olfactometry detector (GC-O), solvent-assisted flavor evaporation (SAFE) is used. The volatiles are mostly separated on GC systems (GC or comprehensive two-dimensional GCxGC) with the support of mass spectrometry (MS, MS/MS, ToF-MS) for chemical compound identification. Besides omics techniques, the promising part could be a study of aroma using electronic nose. Therefore, the main assumptions of volatolomics are here described.

Keywords: aroma profile; electronic nose; food chemistry; food aroma; gas chromatography; GCxGC; mass spectrometry; microextraction; olfactometry; volatolomics



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1. Introduction

Aroma (odor) is an important aspect in designing/formulating novel food products [1]. Thereby, the characterization of aroma is the main goal in many studies. Aroma characterization can give data in the area of food products' quality and their relationship to consumers' choices [2]. The aroma evaluation is often used to identify key molecules accounting for the aroma characteristics of specific food products. Most aroma molecules are volatile organic compounds. These compounds come from, e.g., the ripening of plants, the development of oils or during natural processes such as fermentation [3–6], but also food contamination [7]. Moreover, the volatile compounds are likely to form during long exposure to high temperatures in the reaction of sugars with amino groups of either amino acids or peptides/proteins, which is called the Maillard reaction [8]. The Maillard-derived volatile components are responsible for the characteristic odor of bakery or roasted products, e.g., meat, nuts, and coffee. The listing of naturally-occurring aroma chemicals is very broad, ranging from acids, alcohols, aldehydes, amines, esters and ketones, heterocyclic compounds. About 10k of diverse volatile chemicals have been recognized in foodstuffs plus beverages; e.g., coffee aroma is a mix of over 1k volatiles. Lots of these compounds are frequent in several varied food products. For example, methional is determined in almost every food sample in an olfactometry study. Aldehydes were detected as quality markers of edible oils during their storage at ambient temperature [9]. Additionally, Zhou et al. [10] determined aldehydes and alcohols as significant volatiles in oil quality changes during storage, and the formation is negatively correlated with poly-unsaturated fatty acids amount. As was described by Ahn et al. [11] in their study, turkey meat is also exposed

to the oxidation process and as a result, off-flavor aldehydes could be formed. According to the authors' recommendations, negative volatiles formation could be limited by the addition of vitamin E with antioxidant activity. The example of chemical components in charge of the odor of different foodstuffs is presented in Table 1. However, the contribution of the individual compound to the food aroma depends on several factors, like aroma description, adsorption to the food matrices, content in the food samples, threshold of odor, vapor pressure, chemicals interactions, and co-reaction with other volatiles [12]. Certain volatile compounds do not contribute to food aroma, whereas others define the flavor of food products; for instance, benzaldehyde is a characteristic compound in the aroma of almonds, and citral is a defining aroma of lemons [13]. However, in most cases, the aroma depends on the contribution of a mix of volatile components from various chemical classes.

Table 1. The most frequent volatile compounds from different family groups, their characteristic aroma and their occurrence in food products.

Family Group	Compounds Example	Aroma Description	Examples of Food Sample	References
alcohols	hexanol	bitter, floral	watermelon	[14,15]
terpenes	myrcene	earthy, fruity, and clove-like	lemongrass, clove, bay leaves, basil and thyme	[15]
	α - and β -pinene	woody, green, pine-like ¹	black pepper	[16,17]
	limonene	citrus-like	pistachio, nutmeg, gin	[18–21]
	linalool	floral	saffron, oregano, basil	[22–24]
aldehydes	hexanal	freshly cut grass	almond, chestnut	[10,25–27]
	octanal	fruity	peanut, walnut, lamb meat orange juice	[28]
esters	ethyl octanoate	sweet, fruity, brandy-, apple-, apricot- and banana-like	fermented and dried fish; wine; Xiaoqu Liquor	[29,30]
	ethyl hexanoate	floral, fruity, pineapple-like	alcoholic beverages	[30,31]
furans	furan	sweet, woody, almond-like, baked bread	coffee	[32]
	furfural	almond-like, sweet	cookies, bread	[33–35]
pyrazines	2,5-dimethyl-4-hydroxy-3(2H)-furanone (furanol)	fruity, strawberry-like	kiwifruit, strawberry	[36,37]
	2,5-dimethylpyrazine	roast, coffee-line, peanut-like ²	peanut, cookies	[3,26,33]
	ethylpyrazine methylpyrazine	nutty, buttery, peanut nutty, cocoa, roasted meat	cookies, roasted beef cocoa, cookies	[33,38]
acids	acetic acid	acidic ¹	hazelnut; bread sourdough	[35,39]

¹ [40]; ² [41].

The complexity of food aroma makes the preparation of the sample an essential stage prior to the final investigation. In a matter of technology, the certain detection and categorization of aroma components is a demanding part. Moreover, volatile molecules can naturally interact with the food matrix and also with other food components. The interactions can be classified into three groups. The first is synergism, the second compensation and the third one is masking. Following the definitions created by Mottram and Elmore [12]: “*Synergism* is defined as the interaction when two or more distinct substances produce a mutual scent which is stronger than those of individual components. *Compensation* is the case when one component counteracts another constituent. *Masking* is the combination of one pleasant odor with an unpleasant.” Therefore, as was mentioned before the challenging part, the proper preparation of food samples. Generally, the preparation of food samples prior to analysis could be divided into three main phases. These phases are named as follows: sample pretreatment, preparation and analysis by adequate apparatus [42,43].

The review presents the key concerns and examples of sample pretreatment and preparation that are obligatory in the analysis of food volatiles. The gathered information covers also aspects of methods of identification in the new branch of analysis called volatolomics [44–46]. The un- and targeted analysis are very frequent in the determination of volatile metabolome [47,48]. Rodriguez-Campos et al. [49] studied the volatile profile of cocoa beans to determine the best conditions of their fermentation and drying process, whereas Caprioli et al. [50] and Marseglia et al. [51] examined the fingerprint of volatile compounds of cocoa according to their different geographical origin. Profile of volatiles metabolome was also helpful in the evaluation of the production process of the most popular cocoa product—chocolate [52]. The sample preparation could be divided into four main steps (Figure 1). These steps are: (1) isolation and concentration; (2) separation; (3) identification; and (4) sensory characterization. The isolation and concentration of volatiles could be carried out from head-space, adsorbed on the fiber and then desorbed in the port, distilled or extracted from the matrix using an adequate solvent. In the second step, volatilized molecules are mostly separated using gas chromatography (GC) or sometimes preparative liquid chromatography (prep-LC). After separation on GC, volatile compounds could be identified by their retention times (rt), by calculating their linear retention indices (LRI) or Kovats index, or by comparing their mass spectrum (MS) with the available data. Moreover, nuclear magnetic resonance (NMR) and infrared spectroscopy (IR) could be applied to obtain knowledge about some of the identified compounds, as a separate analysis. To the identification of aroma compounds their references could be also used or if it is not available, their chemical synthesis is preferable. As the last part, the sensory characterization gives information about the characteristic aroma of the compounds/products by gas chromatography coupled with olfactometry port and by professional judges during sensory sessions.

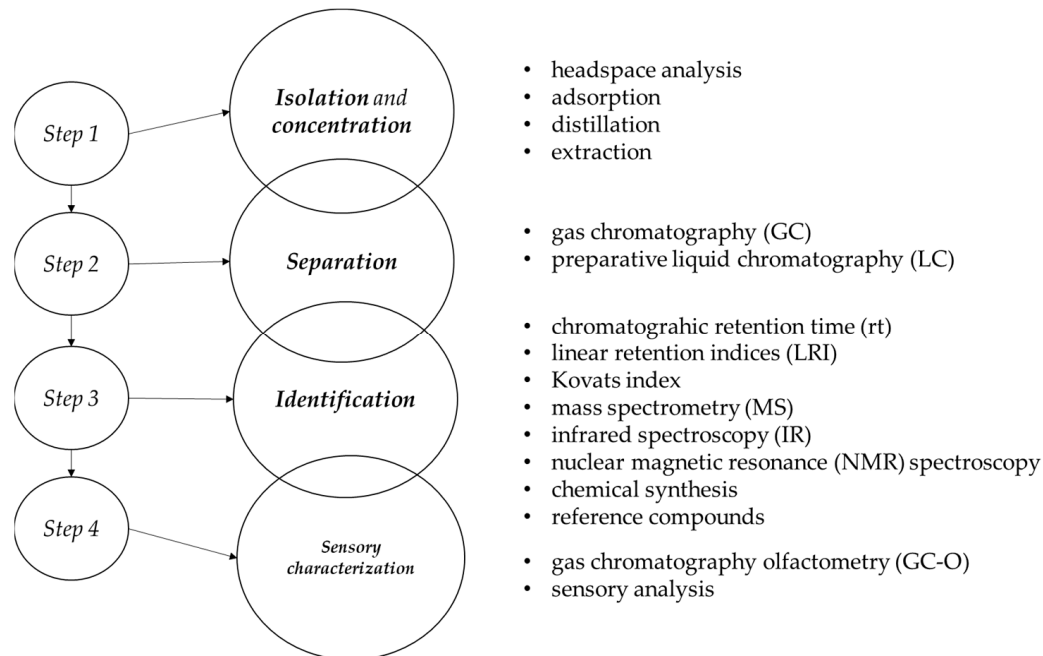


Figure 1. Four stages of the aroma compounds analysis.

2. Isolation and Concentration as a First Step to the Analysis of Volatile Components

These days scientists are searching for novel, simple, and rapid preparation strategies to determine different compounds in complex food matrices. Particularly for fast monitoring of food available on the market [53]. Rapid techniques of sample preparation and furthermore its automatically sampling allow for the analysis of numerous extracts in a short timeframe [54]. The contemporary trend in sample preparation is also the minimalization of the amount of the analyzed sample. Moreover, assessment of extracts preparation

techniques is related to some assumptions of green chemistry for example limiting the usage of organic solvents, decreasing the scale of analytical procedures and minimalizing the exposure to toxic reagents by analytical chemists [55–57]. The extraction technique should be also sufficient to significantly intensify the isolation of volatiles.

Several techniques are implemented to isolate aroma components, including liquid/liquid extraction, solid-phase extraction (SPE), supercritical fluid extraction (SFE), stir bar sorptive extraction (SBSE), solvent assisted (SAFE) and micro-extraction to the stationary phase (SPME) [8,58,59]. According to several publications from over the last decade, the main isolation techniques of volatile compounds from food samples are: stir bar sorptive extraction (SBSE, 2308 items), and solid-phase microextraction—SPME (12465 research items in Science Direct in years: 2012–2022) [60]. Agreeing with the opinion of Liberto et al. [61] that the headspace (HS) is the best technical mode to picture the volatile compounds profile because it is an equilibrium between the vapor phase and food matrix. Therefore, the most widely used in food analysis is the HS-SPME.

2.1. SPME as a Method of Extraction

According to the description, the SPME technique is based on the divide of equilibrium of the analytes between the extracted matrix and extraction phase (in this case, fiber) [62]. The SPME fiber can be exposed directly to the media by direct immersion (DI) or in its head-space (HS) [42]. At the GC injection port, the analytes are thermally desorbed and then transferred to the chromatographic column using a carrier gas for their separation [63]. The universal schema of the SPME technique is shown in Figure 2. Concerning polarity of chemicals, different sorts of coatings might be implemented for extraction, such as polyacrylate coating (PA), carboxen (CAR), polydimethylsiloxane (PDMS), polydimethylsiloxane-divinylbenzene (PDMS/DVB), divinylbenzen-carboxen-polydimethylsiloxane (DVB/CAR/PDMS), or with another type of sorbents. The success of the SPME method might be connected with its timesaving feature and possible options for automatization, as well as non-solvents usage [58]. The positive aspect of the SPME usage is that it can cover multiple steps of analysis such as sampling, separation of compounds of interest from the other matrix compounds, transfer of analytes from outside to the laboratory, and transport of analytes to the instrument. Despite these advantages offered by SPME, Souza-Silva et al. [64] enumerated few limitations of SPME use in analysis of food, e.g., (1) a narrow amount of commercially available coatings, (2) the relatively low temperature to operating due to coatings' poor thermal stability, (3) de-stability and probable swelling of the coating by organic solvents; and (4) the quiet short period of the coating physical usage.

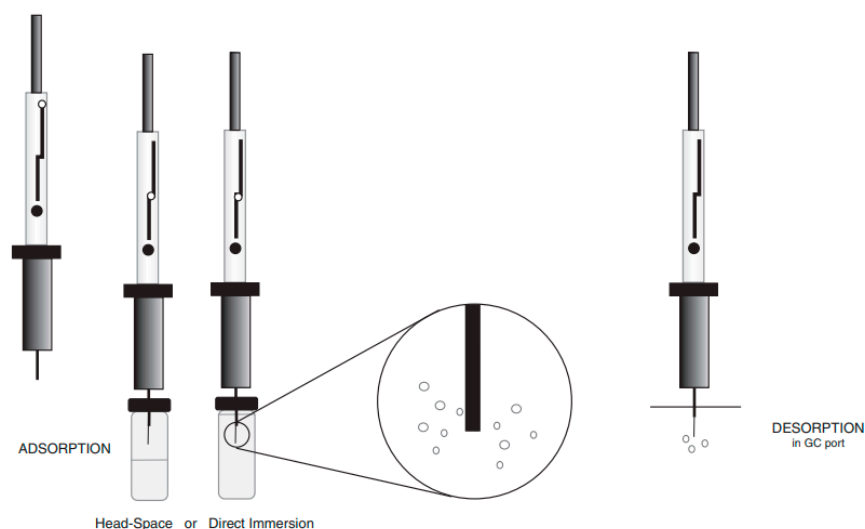


Figure 2. The scheme of the SPME technique.

The SPME was introduced into numerous applications in cannabis, environmental, forensic, and food analysis. As said by Jeleń et al. [65], the main five classes of food products that the SPME was implemented for aroma analysis are: beverages, dairy, fruits/vegetables, honey, meat, seafood, and wine [66–70]. Usage of SPME in the determination of volatile chemicals extracted from various food samples is presented in Table 2. Furthermore, the main problems that were solved by using SPME are: studies of volatiles in different varieties of food products, profiling individual molecules reliable for food aroma, categorization of foodstuffs, also proof their authenticity, analysis of particular compounds responsible for food quality, to screen the technological process influences on aroma properties, screening chemical and biochemical processes related to transformations of aroma molecules, and to linked SPME with gas chromatography-olfactometry (GC-O) to describe the characteristic aroma of volatiles extracted by the coating of the fiber.

Over thirty years on the market, the SPME technology is still developing. Herrington et al. [71] presented the SPME arrow as a new concept of SPME device that can limit the low inter-device reproducibility, as well as a little volume of mobile phases. Vazquez et al. [72] suggested also miniaturization of the SPME device to make it portable and to allow sampling also not in a laboratory environment. Moreover, Starowicz et al. [73] demonstrated that volatile compounds analysis with SPME is a favorable tool to determine also the quality of functional food and designing the product for consumer’s needs. Xu et al. [9] found out SPME as an appropriate method to separate volatile components from the oil matrix and afterward to determine compounds responsible for oil quality during storage. Furthermore, SPME coupled with GC/MS, and with other techniques of analysis, e.g., ATR-FTIR could be a promising possibility to prove the authenticity of food products [23]. Aceña et al. [74] admitted that in comparison to other extraction methods SPME is useful to determine more aroma-active regions of analyzed products whereas Berrou et al. [75] showed a high, 90% recovery of volatiles and semi-volatile using SPME technique. Berrou et al. [75], as well as Ruvalcaba et al. [76], compare SPME with another popular extraction method that is stir bar sorptive extraction, abbreviated as the SBSE method.

Table 2. Identification of volatiles in various food products using the SPME technique.

Food Sample		Equilibration				Extraction		Identification	References
		Sample Amount [mL/g]	Agitation [yes/no]	Time [min]	Temperature [°C]	Time [min]	Temperature [°C]		
Coffee	Arabica and Robusta	0.1 g	no	10	40	20	-	RI, mass spectra	[66]
	Arabica and Robusta	1.5 g	yes	40	50	20	50	semi-quant. with IS	[32]
	Arabica	5.0 mL	yes	1	25	15	25	RI, mass spectra	[77]
Alcoholic beverages	white wines	20.0 mL	no	-	-	15	50	mass spectra	[58]
	beer <i>Baijiu</i>	10.0 mL	yes	5	40	40	40	RI, mass spectra	[20]
Bee products	honey	0.2 g	no	2	-	30	-	semi-quant. with IS	[72]
	honey/bee bread/bee pollen/beeswax	2.0 g	yes	50	40	15	50	RI, mass spectra	[67]
Nuts	hazelnuts	1.5 g (grinding)	no	20	50	20	50	IS ¹	[39]
	almond	5.0 g (grinding)	no	40	24	30	24	semi-quant. with IS	[24]
	almond	1.0 g (oil)	yes	5	60	60	60	retention times and mass spectra	[23]
	almond	5.0 g	no	40	24	30	24	IS	[78]
	chestnut	250.0 g	-	-	-	24	-	RI, mass spectra	[25]
	peanut	3.0 g (grinding and oil extraction)	yes	10	50	40	50	semi-quant. with IS	[59]
	pistachio	15.0 g (grinding)	yes	15	50	120	50	- ²	[79]
Meat	walnut	0.5 g (grinding and oil extraction)	yes	15	50	30	60	semi-quant. with IS	[80]
	beef	4 g (cooked)	no	-	-	50	80	RI, mass spectra	[20]
	beef meatballs	5 g	no	20	60	40	60	semi-quant. with IS	[68]
	fish <i>miso</i>	3 g	-	-	-	40	40	RI, mass spectra, standards	[81]
	pork	5 g	no	-	-	30	60	RI, mass spectra	[82]
yellowfin tuna	6 g	no	15	70	40	70	RI, mass spectra, IS	[83]	
Oil	peanut/soybean/rapeseed/linseed oil	5 g	yes	30	50	30	50	RI, mass spectra	[9]

¹ IS: internal standard; ² without quantification.

2.2. The SBSE Extraction

SBSE is supposed to be the second most mentioned technique of extraction in volatiles analysis. Same as SPME, SBSE is a green extraction technique that does not require the usage of organic solvent, but only a small amount of samples. It could be useful in the

evaluation of aroma components of varied foodstuffs. SBSE has a more effective extraction capacity in comparison to the SPME method. The volume of coating used for SBSE is from 50 to 250 times higher than in SPME [84]. The magnetic bar of SBSE is mainly coated by PDMS fiber, whereas SBSE is dedicated to extracting semi- and volatile compounds in aqueous foods' matrices. Thus, the stir bar is inserted into the medium directly, rotated and then molecules are captured by bars, and then desorbed by gas or liquid phase [8]. The other option developed by Bicchi et al. [85] is that the volatile compounds are adsorbed onto the PDMS stirring bar in the headspace of the sample for some time (headspace sorptive extraction—HSSE). The main advantage of using a coating bar in both extractions is the possibility to extract low polarity molecules. SBSE has a very low limit of detection (LoD) and this was the reason why it was applied to describe, for example, wine off-flavors [86]. The SBSE technique is also coupled with GC equipment, however, SBSE is a not-automated technique, due to that it is not common like extraction by SPME fiber.

Franc et al. [86] showed high reproducibility and repeatability, and a good correlation of SBSE in contrast to SPME and liquid–liquid extraction (LLE), in the wines' off-flavors profiling. With SBSE, Franc et al. [86] successfully extracted and quantified eight domain volatiles, which is likewise highly responsible for the unpleasant aroma of wines for instance geosmin with characteristic muddy and musty odor. SBSE and HSSE were also exploited to develop the aroma composition of beer samples by Ruvalcaba et al. [76]. The SBSE was successfully validated to determine the volatiles profile of Sherry brandy [87], to established Spanish brandy authenticity. Moreover, this technique of extraction allowed researchers to determine safral as a main volatile compound in cured ham with saffron addition [22]. Moreover, Berrou et al. [75] optimized the method of SBSE to extract bacterial metabolites and established the 90% recovery of volatiles. Expect alcoholic beverages and meat, SBSE was similar to Guerrero et al. [88], evaluate to the determination of volatile components of vinegar samples with low detection limits. In another study, validation of SBSE with GC-MS allowed determining limonene as a major aroma compound in squeezed orange juices as well as commercial samples [89]. Usage of SBSE simultaneously with SPME could be important to determine the wide range of volatiles that could be aroma-active compounds [90].

2.3. Solvent-Assisted Flavor Evaporation (SAFE)

The SAFE was established in the 1990s as a novel extraction technique, that admits to separate the volatile part from nonvolatile compounds [91]. SAFE is devoted to the extraction of the profile of aroma of foodstuffs to control aroma-active molecules and follows the molecular concept strategy. The key benefits of the SAFE technique are firstly inhibiting the formation of artefacts, secondly forbid the thermal degradation of nonstable volatiles' composition. The major disadvantage of this method is the time-consuming practice, mainly dealing with products with fats. Additionally, in contrast to SPME, SAFE needs a greater volume of samples for analysis. Though, the SAFE extraction is a very suitable extraction technique for sample preparation to GC-O analysis [92]. The technique of SPME could be too selective to capture the whole profile of aroma reported in the foodstuffs. Therefore, the SAFE methodology was mostly used to verify particular molecules responsible for the aroma of varied foodstuffs like orange juice, cocoa, products from cocoa, nuts, alcoholic beverages and also raw fruits, e.g., apricots [28,38,93–99].

SAFE is an important preliminary part of the molecular sensory concept, which allows separating volatile from non-volatile compounds to further analysis by GC-O and/or other GC instruments. The molecular sensory concept allowed to pointed out chemicals responsible for the off-flavor of stored orange juice prepared from concentrate [28], and to aroma reconstruction of apricots [99]. Kiefl and Schieberle [93] involved SAFE to prepare samples to further differentiate hazelnut cultivars, whereas Burdack-Freitag et al. [95] pointed out differences in aroma composition between raw and fried Italian hazelnuts. Fractionation of aroma compounds from raw peanuts and panned fried peanut meal was also carried out using SAFE [90]. Moreover, Franitza et al. [96] and Langos et al. [97]

prepared samples by SAFE to determine aroma-active compounds of alcoholic beverages such as rums and beers, respectively, to distinguish the quality of the lower and higher-priced market-available products. Additionally, SAFE was used to prepare samples of Chinese alcohol based on sesame to prove their authenticity by determination of aroma profile [98].

3. Methods of Volatile Compounds Separation and Identification

The second important step of volatiles analysis is their separation and identification. SPME has been broadly employed in a sequence of several analytical techniques: capillary electrophoresis (CE), gas chromatography (GC), inductively coupled plasma MS (ICP-MS), ICP-optical emission spectrometry (ICP-OES), and mass spectrometry (MS) [49,100]. Not frequently liquid chromatography (LC) was applied to analyze volatile compounds [101]. Moreover, various detectors are applied, e.g., universal flame ionization (FID) or selective detectors, e.g., nitrogen-phosphorus (NPD), flame photometric (FPD), electron capture (ECD) detectors. However, the most frequently used detector in the analysis of volatile compounds is the mass detector (MS) [49]. MS is a powerful tool to identify compounds. By the MS it is possible to maintain screening of volatile compounds and then the comparison of obtained mass spectrums with the literature and/or available databases.

For example, Chin et al. [102] optimized the GC-MS technique to aroma evaluation of most popular beverages like wine and coffee. Additionally, Jordán et al. [103], who evaluated aroma profiles of food products, based on compounds' molecular masses. In the latest research, Singh et al. [104] presented the usefulness of GC-MS in the determination of aroma-active regions of protein-based products. Research about the aroma of protein-rich products is nowadays an important aspect because a lot of vegan products are not always formulated with high consumer acceptability. Therefore, the knowledge in this area needs a lot of improvement. Moreover, based on GC-MS signals, Abou-el-Karam et al. [105] built the database of the volatile profile, which can be used in the food authenticity. Nicolai et al. [106] reconstruct the aroma of citrus fruits using chemical standards and then analyzed its profile on GC-MS. Miniaturization is not only proposed in extraction practice but also as it was presented by Beck et al. [107] in the case of analytical devices. Beck et al. [107] constructed portable GC-MS equipment and compare their possibilities with benchtop devices. In their opinion usage of a portable GC-MS was satisfactory and allowed to analyze in any place. The time-consuming methods are replaced with the newer ones. Mayr et al. [108] employed Proton Transfer Reaction Mass Spectrometry to do fast screening of volatiles profile of meat from the market and therefore, avoid the spoilage of meat. Instead of a time-consuming method that needs a few days to be performed, they applied a methodology that requires only a few minutes.

The volatile profile is a complex mixture, which is why, often, the usage of GC-MS is not enough to unravel the aroma of food products. Therefore, a set of equipment is needed to recognize the active regions responsible for odor. Cialì Rosso et al. [48] used two-dimensional GC to describe the potent odorants of hazelnuts, whereas Stilo et al. [109] prepared the quality fingerprint of virgin olive oil due to its volatiles profile. Interestingly, the GCxGC approach was established by Giri et al. [110] to evaluate the profile of aroma as well as compounds with toxicology properties. Besides the demand for advanced analytical equipment, Lubes and Goodarzi [78] highlighted the importance of the statistical analysis of obtained data. An important aspect is also the determination of food odor at the time of its consumption. In this case, the APCI-MS, named as Atmospheric-Pressure Chemical Ionization Mass Spectrometry, is often used. Elmasry et al. [111] applied APCI-MS to evaluate honey authenticity. Additionally, other techniques of analysis have been adopted to the determination of volatiles profile, e.g., ion mobility spectrometry [112], or infrared spectroscopy [113].

Several approaches have been adopted to establish volatile profiles in quantitative and semi-quantitative terms and, therefore, the results have been expressed in different ways. Sometimes authors expressed their results only as an integrated peak area, or as a percent

contribution of each compound in the total amount of volatiles. This method could be used only in the case of profiling the volatiles in food samples, to determine general differences between samples [67]. The proper study of volatiles content should be carried out using an internal standard, that might be useful in the calculation of the volatiles content in the semi-quantitative way [32]. Furthermore, the quantitative analysis could be performed after preparing the calibration curve of standards of each main volatile, as was carried out in the research of Starowicz, Koutsidis and Zieliński [33] and Cialiè Rosso et al. [48]. In both cases, either reference compounds (from the stock/synthesized in the laboratory) or stable labelled isotopes reference compounds (e.g., ^2H , ^{13}C , ^{15}N) could be used. Moreover, if the food matrix plays an important role in aroma molecules release, Sgorbini et al. [114] propose multiple headspace extraction as the exhaustive extraction of an analyte from a sample by the HS-SPME method. It allows to sum up the peak areas of each compound after series of extraction to quantify its content.

4. Sensory Characterization

4.1. Olfactometry Study

It is well-known that food staples usually contain several hundreds of molecules responsible for their aroma. However, ~20–30 of these compounds are important to generate its aroma. Therefore, to perform an important analysis of aroma, it is important to recognize these compounds with a significant impact on the aroma. In this case, gas chromatography and olfactometry (GC-O) are the best selection [115]. The GC-O technique uses the human nose as a detector that elutes the aroma from the chromatography column. Further, when an odor is sensed, the time and characteristic aroma quality, and from time to time the intensity are written down. There are different methods to evaluate the relative odor potential of the individual aroma-active molecule, as aroma extraction dilution assay (AEDA), CharmAnalysis, Osme, and some others [116–118]. Interestingly, Feng et al. [119] combine AEDA with the SPME technique and achieved satisfactory results that allowed to determine the aroma with sensory impact in Japanese soy sauce. Afterward, detection and the quantification of the odor-active molecules by analysis of GC-O, the odor activity value (OAV) (ratio between the concentration of one compound in the food and its threshold concentration) could be determined. Commonly, if one of the compounds shows an OAV value higher than 1, then this molecule might have an aroma impact on the overall odor of a food product. The presented procedure of analysis is called the molecular sensory concept. According to this procedure, Averbek and Schieberle [28] pointed out the off-flavor formation with a significant amount of dimethyl sulphide and 2-methoxy-4-vinylphenol in stored orange juice. Moreover, using this methodology allowed to differentiate the quality of market-available food products [96]. To proper identification of aroma, deuterated standards and/or molecules with isotopic labelled carbon could be used [120].

Mostly, results realized during the methodology for GC-O are associated with sensorial analysis. The recombination or omission analysis is the last part of the molecular sensory concept, during that test the aroma overall is judged by a professional sensory panel. This methodology allowed Kiefl and Schieberle [93] to differentiate three hazelnut cultivars, whereas Żołnierczyk and Szumny [121] established the basic composition of volatiles in edible insects, and compared it with their sensorial properties, and their possible acceptance by consumers. The chemical and sensory connection was also helpful in the elevation of new products [122]. In these cases, sensory panelists are the main “analytical tool”, who need to be specially trained.

4.2. Electronic Nose (E-Nose)

The electronic nose is not strictly used to prepare sensory characteristics of food products according to a definition it is a matter of sensory analysis with trained panelists. Therefore, it can be said that e-nose is an instrumental way to reveal some aroma discriminates of foods. The e-nose is a group of chemical sensors, associated with a pattern-

recognition system that is responsible for odors transport through it. Various aromas affect different responses in the sensor system, and these reactions provide a signal pattern characteristic to a particular aroma [123]. The computer system recognizes the pattern of signals and then is able to compare the character of the aroma of varied foods' extracts by pattern recognition system, e.g., artificial neural network. This artificial olfaction could be helpful in the characterization of hundreds of samples in a relatively short time [124]. For instance, a producer who uses coffees from around the world, can analyze coffee samples by the e-nose, and the coffees' odors can be putting together on a multidimensional response map (Figure 3). The analysis of numerous coffee samples, using the best conditions, can result in a group of points for each sample, that furthermore can be grouped. As the number of samples increases, the differences among the groups ought to also grow. Mapping the sensor responses of an unknown sample on the same scheme could allow its detection, by its proximity to one of the known samples.

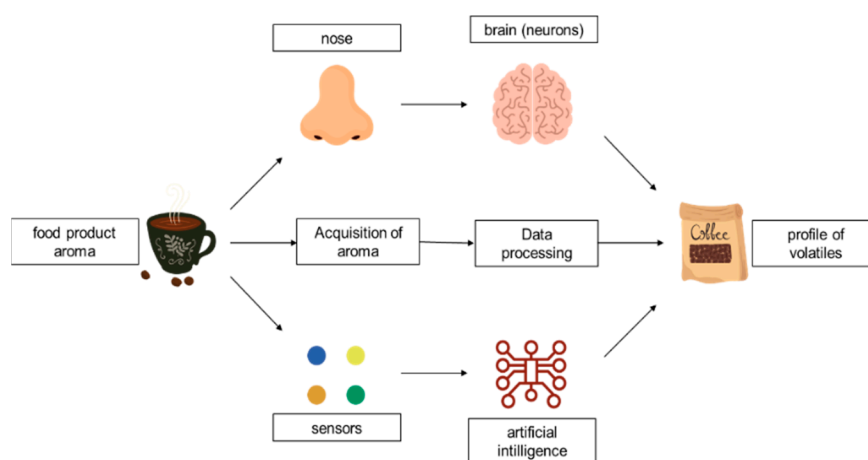


Figure 3. Analogy between the biological olfactory system and the e-nose technology.

Electronic noses have been already applied in the food industry for quality control and detection of contaminants and off-odors. Wojnowski et al. [125] also described the advantages of portable electronic nose usage. However, the human nose is often more sensitive to odorous compounds than are instruments used to detect odors. The study of Lindinger et al. [126] about the aroma of espresso coffee presented a higher correlation of sensory panel results with aroma characteristics obtained from on-line proton-transfer-reaction mass spectrometry (PTR-MS). The important innovation was using a combination of HS-SPME with a mass spectrometer detector and to determine the aroma of in-cup coffee [127]. The proposed method allows to receive the fingerprint of volatiles in tested food material and predict the sensory quality of other coffee beans; however, the limitations are a high number of samples that need to be analyzed to prepare a proper model and, in the next step, the insufficient access to data mining program.

5. Conclusions

This review was prepared to gather the most recent knowledge about analytical aspects of volatile compounds determination in varied food products. Starting from extraction, then describing the separation and identification step, and finally presented the olfactometry and e-nose approaches. The general improvement in laboratory equipment and miniaturization of analytical devices is being observed. According to the presented information, the most popular are rapid and solvent-free extractions, like SPME and SBSE.

From the future perspectives, the efficiency of extraction methods especially from problematic food matrices should be still a point of discussion. Besides the extraction method, particular studies could be focused on the maximum shortening of the instrumental analysis procedure. All of these efforts should be taken into consideration to processed routine verifications of food quality, e.g., during production.

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