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REVIEW

Sensorial, cultural and volatile properties of milk, dairy powders, yoghurt and butter: A review

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Countries with an established dairy tradition consume milk, milk powder, yoghurt and butter directly or as an ingredient; however, in countries without this tradition the lack of familiarity and unknown expectations can be challenging to overcome. Therefore, having a better understanding of the volatile properties that influence their sensory appeal can aid overcoming these challenges. This review focusses on traditional and novel sensory methods used to research milk, milk powders, yoghurt and butter as well as the extraction techniques used in gas chromatography mass spectrometry and gas chromatography olfactometry to identify volatiles in these products that influence sensory perception.

Keywords Milk, Dairy powders, Yoghurt, Butter, Sensory, Volatile organic compounds.

INTRODUCTION

Sensory analysis is an important part of dairy product development and manufacture, providing answers to specific flavour, visual and textural characteristics and hedonic consumer responses amongst others. Affective tests incorporating preference and hedonic testing use subjective criteria of untrained consumers to provide important market information cost effectively (Stone et al. 2020). Combinations of affective and analytical techniques (threshold, discrimination and descriptive tests) are applied to take advantage of each technique's convenience for specific purposes providing important sensory information that can be used for example to improve product quality and/or market share. New sensory methodologies have been developed with the aim of rapidly providing sensory data more cost effectively, but doing so relatively simply in comparison to traditional techniques (Ruiz-Capillas et al. 2021). Such sensory methods include check-all-that-apply (CATA), flash profiling (FP) and rate-all-that-apply using trained panels or semi-trained panels.

Cross-cultural sensory and consumer research is becoming increasingly important, as it involves both consumer psychology and the dynamic interaction between the consumer, the context and the food (Lee et al. 2010). Culture is one of the significant factors underlying consumers' food choices, influencing attitudes and beliefs about food (Rozin 1988). Different food environments and dietary experiences across cultures influence both sensory perception and consumer preferences (Prescott and Bell 1995). Cross-cultural studies aid in the understanding of how consumers from different cultures perceive foods and assists in achieving market penetration, especially for new products or for unfamiliar products in new markets (Ares 2018).

As food aroma is such a significant factor in flavour, it is a widely researched topic, with over 10 000 volatile organic compounds (VOC) known to exist with less than 3% thought to contribute to the aroma of any given food (Dunkel et al. 2014). VOCs must be present at a

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concentration above their odour threshold in order to be perceived, this is typically measured as their odour activity value (OAV), which is the ratio of the concentration to the odour threshold. In most cases, the presence of multiple VOCs is essential for the characteristic aroma of a product, rather than a single VOC. It is also thought that many other factors not just the OAV of VOCs impact sensory perception, such as gustatory and trigeminal components and genetic differences between individuals (Spence 2021). However, understanding the VOC profile of a food gives an important insight into the relationship between VOC and multisensory flavour perception, but determining the true VOC profile of any product is difficult due to the many factors that can impact analysis.

A key aspect of VOC research that is often not addressed in dairy research is the actual relationship between VOC and sensory perception. This can be achieved to some extent using multivariate statistical analysis of VOC and sensory data (ideally descriptive sensory data) where some plausible associations can be implied; however, it is much more beneficial to undertake gas chromatography-olfactometry (GC-O) where individual aroma active VOC can be identified (Cadwallader and Singh 2009; Sarhir et al. 2021) as well as their potential significance to the overall aroma. There are several factors involved in the processing of dairy products that impact VOC; such as oxidative stability, thermal treatments, high pressure, ultrasound and addition of processing aids, ingredients, cultures or enzymes (Vazquez-Landaverde et al. 2005; Cadwallader and Singh 2009; Riener et al. 2009; Serra et al. 2009; Liu et al. 2022). Table 1 summarizes the composition and common thermal treatments applied to, milk, dairy powders, butter and yoghurt during processing.This review will focus on the key aroma active VOC in milk, dairy powders, yoghurt and butter and their relationship to product quality from a flavour perspective, incorporating cross cultural sensory analysis and new trends in sensory science applicable to these products. This review does not include cheese due to the added complexity of the product, and the fact that so many studies have been undertaken that it requires a separate independent review.

SENSORY ANALYSIS

Sensory techniques

Sensory science is used to assess, study and explain the response of the peculiarities of food that are observed by panellists using their senses of sight, smell, taste, touch and hearing (Stone et al. 2020). Sensory analysis is used to obtain a better understanding of the relationship between aroma and sensory perception. Different types of sensory analyses, from conventional methods (Consumer Acceptance Testing and Quantitative Descriptive Analysis) to novel rapid sensory techniques (Check All That Apply, Flash Profile, Temporal Dominance of Sensations, etc.) are used to understand more about key sensory attributes and/or preferences of dairy products (Drake 2007; Andrewes et al. 2021).

Consumer acceptance testing is easy to preform using hedonic scales without sensory training. The hedonic scale assumes that participants' preferences exist on a continuum and that their responses can be categorized into the degree of liking or disliking of sensory attributes, such as appearance, odour, taste, aroma, texture (O'Sullivan 2016). The most widely used scale for measuring food acceptability is the nine-point hedonic scale, which has ruler-like and equal-interval properties with 'dislike extremely' on the left and 'like extremely' on the right (Wichchukit and O'Mahony 2015). Previous sensory studies have employed between 18 and 310 consumers for hedonic testing of milk, butter, yogurt and dairy powders (Hoppert et al. 2013; Potts et al. 2017; Cheng et al. 2020; Garvey et al. 2020; Clarke et al. 2020a; da Silva et al. 2021).

Descriptive tests consist of a full sensory description of the products and require fewer panellists, but the panellists must be highly trained to distinguish between attributes previously selected through focus groups (using selected sensory attributes from product references or standards) that best describe the product, and to evaluate their perception with quantitative values (O'Sullivan 2016). Quantitative descriptive sensory analysis is one of main descriptive analysis techniques in sensory evaluation. Clarke et al. (2020b) used 12 trained (60 h) descriptive sensory panellists to assess milk samples from different diets and the results of full descriptive sensory analysis provided a reliable insight into the differences of milks based on cows feeding system. However, operating traditional descriptive trained panels is expensive and time consuming, and therefore other methods have been developed in order to obtain sufficient sensory information, but more rapidly and cost effectively.

Optimized descriptive profiling (ODP) is a rapid method for obtaining sensory descriptions utilizing semi-trained judges that has the potential to quantitatively evaluate sensory attributes (da Silva et al. 2012). Cheng et al. (2020) used ODP method to identify the sensory attributes of skim milk powder (SMP) produced from different cows diets with trained assessors from Ireland and China. Irish and Chinese trained assessors had different preferences for many attributes, and both found it more difficult to discern differences between SMP derived from cows outdoors fed perennial ryegrass or perennial ryegrass with white clover, than SMP produced from cows indoors on a concentrate diet.

CATA is another sensory approach to rapidly assess products. Consumers are presented with the sample and a versatile multiple-choice questionnaire, then asked to indicate which words or phrases appropriately describe their sensory experience (Ares et al. 2015). The terms might include sensory attributes, hedonic responses, emotional responses, purchase intentions, potential applications, product positioning

Table 1 Summary of the processing method and compositional in milk, milk powder, yogurt and butter

Dairy products	Processing method	Typical thermal treatments	Compositional information (%)	References
Milk	UHT	135°C for 2–5 s		a
	High temperature short time (HTST)	72°C for 15 s	Moisture: 86; protein: 4.2; Fat: 3–4.6	b,c,d
	Low temperature long time (LTLT)	63°C for 30 min		d
	High hydrostatic pressure processing (HPP)	40°C, 600 MPa for 5 min		d
	Ultrasound (US)	400 W, 45°C for 2.5–20 min		e
SMP	Low heat spray drying	71°C for 20 min	Moisture: 3–4; protein: 34–37; Fat: 1.2	f,g
	Medium heat spray drying	71–79°C for 20 min		
	High heat spray drying	90°C for 30 min		
WMP	Medium-heat	65°C for 20 min	Moisture: 3–4; protein: 24–27; Fat: 26–29	h,i
Yogurt	High temperature short time (HTST)	72°C for 15 s	Moisture: 88; protein: 3–5; Fat: 3–4	j,k
	Ultra-high pressure homogenized (UHPH)	300 MPa, 90°C for 90 s		
Butter	High temperature short time (HTST)	72°C for 15 s	Moisture: 15; protein: 1; Fat: 80–83	l

The data adapted from (a) Vazquez-Landaverde et al. (2005), (b) Faulkner et al. (2018), (c) O'Callaghan et al. (2019), (d) Liu et al. (2020a), (e) Riener et al. (2009), (f) Karagül-Yüceer et al. (2001), (g) Turner et al. (2002), (h) Clarke et al. (2020b), (i) Lloyd et al. (2009), (j) Tian et al. (2017), (k) Serra et al. (2009), (l) O'Callaghan et al. (2016).

or other terms that the consumer might associate with the sample. Harwood and Drake (2020) used a list of 22 features in a CATA format to identify what features typically influenced panellists purchase of milk. The results demonstrated that consumers generally expressed preferences that aligned with their explicit beliefs, and flavour considerations appeared to be a secondary differentiator of preference.

FP is another rapid low cost technique where untrained panellists select their own terms to describe and evaluate a set of products simultaneously, and then rank the products for each attribute that they individually create. Panellists are forced to generate discriminative attributes of the whole sample set but not on a hedonic term (Delarue 2015). Yao et al. (2018) used FP with 17 sensory attributes developed by 10 panellists for yoghurts produced by pasteurisation or by thermisation. FP was able to discriminate yoghurts based on the heat-treatment applied.

Temporal dominance of sensations (TDS) is dynamic descriptive sensory technique that involves repeatedly assessing, until the sensations end, and determining which sensation is dominant in scoring its intensity (Pineau et al. 2009). Compared to time intensity, this method considers the multidimensionality of the perceptual space over time. Hutchings et al. (2017) used TDS to analysis milk protein hydrolysates using 20 consumers over six training sessions. Similar TDS results were obtained by the panellists from three levels of training session (untrained, familiarized and trained) for each product, but training also increased panel consensus and the ability to discriminate between milk protein hydrolysates. As the training session increased, the number of attributes selected decreased and the time spent on a given attribute increased.

Cross-cultural sensory analysis

The familiarity of food products plays an important role in acceptability and preference because it decreases the uncertainty about the safety and suspense associated with a novel product by generating a better match between expectations and sensory characteristics (Methven et al. 2012; Borgogno et al. 2015). For several studies, familiarity has had a positive effect on the liking scores of the food items and demonstrates a products' palatability and safety (Prescott 1998; Torrico et al. 2019). Liem et al. (2016) noted that Chinese consumers who had repeated exposure to the taste of ultra-high temperature (UHT) milk preferred UHT milk over pasteurised milk, highlighting that familiarity is a powerful driver of consumer liking. Cross-cultural differences exist in that familiarity may even influence trained panellists' perception of an attribute, e.g to be more or less intense than it actually is when responding to unfamiliar products (Lee et al. 2010). Tu et al. (2010) also concluded that the French panels who were less familiar with soya yoghurts needed twice as many attributes to describe the product's aroma than a Vietnamese panel who were more familiar with these products. Garvey et al. (2020) investigated the liking and perception of salted butters, produced from milk derived from different diets (perennial ryegrass or perennial ryegrass and white clover, or concentrate) by consumers in Ireland, Germany and the USA. The results demonstrated that familiarity contributed to sensory differences in Irish butter identified by German, Irish and USA consumers and assessors. Irish consumers preferred the appearance and flavour of butters produced from milk derived from cows outdoors on perennial ryegrass or perennial ryegrass and white clover, than German and USA

consumers. German consumers found the salt intensity highest in butter produced from cows milk derived from the perennial ryegrass or perennial ryegrass and white clover, which was thought to relate to the softer texture of these butters and their more rapid melting properties due to changes in fatty acid content, as the salt contents were similar. Familiarity was also postulated to contribute to differences in 'appearance liking' and 'colour liking' of these butters by USA consumers, where the butter produced from milk derived from cows indoors fed on concentrate scored highest, as this is the most widespread type of this butter available in the USA.

Consumers may also rely on their memory associative structure created from past personal experiences to influence acceptance, which is heavily influenced by culture. The multidimensional experience (sensory perception, memory, culture and emotions) by consumers may increase acceptance for products (Corredor et al. 2010). The appearance is the first attribute evaluated by consumers and the visual information of the samples strongly influences the hedonic scores (Zampini et al. 2007). Satisfaction of these extrinsic aspects can influence overall liking, and thus purchase intent and even willingness to pay a premium, particularly for dairy products (Bir et al. 2020; Scozzafava et al. 2020). Hay et al. (2021) investigated consumer sensory preferences for drinkable yoghurt and the impact of provenance using Chinese, European and New Zealand consumers. In terms of sensory drivers 'sweetness', 'sourness', 'strawberry flavour', 'dairy flavour', 'creamy flavour' and 'creamy texture' and 'thickness' were correlated with culture. Chinese consumers had a cultural expectation for higher levels of sweetness compared to New Zealand and European consumers, while New Zealand consumers expected higher level of sourness, but not too sour. Dairy flavour was an important sensory attribute for Chinese consumers, and expectations concerning 'strawberry flavour' and 'thickness' also differed between the cultural groups.

Novel or unfamiliar food products are usually rejected by consumers and consistently score lower liking scores for all sensory attributes regardless of the cultural group (Pingali 2007). Tan et al. (2015) contrasted two groups of potential consumers with and without cultural exposure to specific foods and found that rejection of unfamiliar foods was greater than familiar foods, which can be considered a big factor in product development of novel food items. Ethnic food in a cultural community is often regarded as novel food by another community (Bell et al. 2011). Cheng et al. (2020) assessed consumer perceptions of SMP produced from milk derived from cows outdoors fed perennial ryegrass, or perennial ryegrass and white clover, or cows indoors fed concentrate in Ireland, China and USA. Chinese consumers could not discern a difference between the three SMP produced from the different diets, but rated 'aftertaste liking' and 'aftertaste intensity' differently than Irish and USA consumers, which may relate to the fact that some attributes were difficult to categorise with ambiguous cultural meanings. Moreover, Chinese consumers and trained assessors scored many attributes quite differently than their Irish or USA counterparts, likely again reflecting a lack of familiarity with dairy products. USA consumers had preference for SMP produced from milk derived from cows on a concentrate diet, while Irish consumers generally preferred SMP produced from cows on a pasture diet (either perennial ryegrass or perennial ryegrass and white clover diets), which reflects the main sources of cow diet used in both geographical locations.

Situational interpretations and meanings can also differ across languages and cultures. This can be a problematic for panels (consumers) only measuring the momentarily blinded sensory perception for preference, liking and acceptance, by the fact that anchors have also been shown to influence cultural differences (Yeh et al. 1998; Ares 2018). Sensory attributes do not necessarily have a direct relationship with a single ingredient and have not a direct translation across languages, and therefore can cause problems for consumers with dissimilar cultures and languages (Prescott 1998). Cheng et al. (2020) suggested that differences in the 'aftertaste liking' attribute for Chinese consumers in relation to their perception of SMP may have more to do with the verbalisation of sensory perception and linguistic representation, rather than the Western definition of the term. A similar result was found by Zhi et al. (2016), where a high 'aftertaste intensity of thickness and sweetness' is often used as a positive term to describe better quality milk in China and thus the concept of 'aftertaste' may be cultural dependent, because the underlying conceptual elements and words used to describe its features may be dissimilar. Pingali (2007) identified that creamy attributes would not be considered a common descriptor to delineate the characteristics of dairy products in the Chinese and Korean language. Chinese and Korean groups would use goso/xiāng (fragrancy) instead to describe their perception of dairy products.

It is necessary to validate scales (especially the meaning and psychological properties of scale labels) and any questions within the cultures of interest properly before conducting any cross-cultural sensory evaluation. Instructions to participants and questions should be accurately translated from one language to the other by a bilingual person to ensure that they hold the same meaning across all the cultural groups under consideration and to minimize differences in cultural interpretation and familiarity of any words (Helms 1992; Arnold and Smith 2013). Preference mapping can potentially allow the interpretation of preference data from another culture to be related to trained panel descriptions and measurements conducted in one's own language (Prescott 1998). Ares (2018) also suggested that the behavioural measurements such as the Ranking or Best–Worst

scaling becomes an alternative to hedonic scaling, which could decrease the mistranslation in scale-usages styles/response styles between Asian and Western consumers. Lee and Lopetcharat (2017) highlighted that using a combination of behavioural measurements and sensometrics improved both the validity and reliability of cross-cultural sensory and consumer studies by both stabilizing the subjects' evaluative process and quantifying the effects of cultures. Kim et al. (2018) processed the verbal definition in conceptual elements of nutty with a sensory approach that correlates structured sensory space with cross-cultural sensory elements driving nuttiness perception. Their results revealed that each cultural group (Korean, Chinese and English-speaking-Western consumers) evaluated nuttiness in soymilk based on similar criteria, which avoided misunderstandings in sensory attributes caused by conceptual differences across culture. Köster and Mojet (2015) recommended the use of non-verbal methods, such as PrEmo (a tool used to measure the emotions evoked by materials), in cross-cultural research in order to overcome language differences in the use of emotional terms.

VOLATILE PROFILING BY GAS CHROMATOGRAPHY MASS SPECTROMETRY

Volatile extraction techniques

The VOC profile of dairy products can be influenced by animal diet, heat treatments, processing and storage conditions (Baldwin et al. 1991; Birchal et al. 2005; Kilcawley et al. 2018). As the composition of the dairy products varies extensively, this can have a significant impact on VOC extraction due to differences in VOC solubility in polar and non-polar phases within the product, and from interferences from other elements present, especially salts. These factors need to be taken into account to determine the most suitable method of extraction for their isolation and subsequent analysis (Jeleń et al. 2012). Many dairy products also contain active microbes that are dynamically undergoing enzymatic and or chemical changes that both directly and indirectly impact on the VOC profile.

A wide array of extraction techniques have been employed to isolate and concentrate VOCs from different dairy products, including, for example, solid-phase microextraction (SPME; Coppa et al. 2011; Clarke et al. 2019; Cheng et al. 2020), solvent-assisted flavour evaporation (SAFE; Evans et al. 2009), dynamic headspace extraction (DE; Ciccioli et al. 2004), thermal desorption (TD; Faulkner et al. 2018), stir bar sorptive extraction (SBSE; Schiano et al. 2019) and simultaneous distillation extraction (SDE; Kobayashi et al. 2008). However, reliable detection and complete quantification of VOCs in dairy products remains challenging (Schiano et al. 2019), as every technique has a degree of bias towards the extraction of certain chemical classes based aspects of the process itself, such as type of solvent or sorbent phase used (Ning et al. 2011). Therefore, it is best to utilise multiple extraction techniques if possible in an attempt to get the as true a volatile profile as possible for untargeted analysis. Most volatile extraction techniques are used in tandem with gas chromatography mass spectrometry (GC-MS), although other options exist, such as GC-FID (flame ion detection), SIFT-MS (selected ion flow tube mass spectrometry) and PTR−MS (proton-transfer reaction mass spectrometry) (Mariaca and Bosset 1997; Aprea et al. 2009; Olivares et al. 2011).

Microextraction methods that have a minimal amount of extractant phase enable fast sample preparation, high sensitivity and are more easily automatable, and are thus becoming more widely favoured for VOC characterization. In addition as 'green chemistry' techniques that are seen as more environmentally friendly which require little or no solvents are becoming increasingly favoured. Figure 1 (a) shows the results of Web of Knowledge search for extraction methods used in milk, SMP, whole milk powder (WMP), yoghurt and butter between 2000 and 2021. In total, ~44 publications were identified, dominated by headspace solid-phase microextraction (HS-SPME), with solvent-assisted flavour evaporation (SAFE) also widely used (Figure 1a). When looking into the types of dairy products in which multiple extraction techniques were used for analysis of VOC, the biggest group was for milk, followed by SMP, WMP, then butter and finally yoghurt (Figure 1b).

A summary of the volatiles identified in milk, dairy powders, butter and yoghurt from four common extraction methods by GC-MS are provided in Table 2. A total of 303 VOCs were identified by various extraction methods including alcohols (59), aldehydes (50), esters (38), ketones (30), organic acids (23), lactones (20), terpenoid compounds (17), carbonyl compounds (14), sulphur compounds (8) and furans (8).

Solid-phase microextraction

SPME is a widely used extraction technique, in part due to its relative simplicity (no extensive sample preparation) when compared to other techniques such as dynamic headspace extraction or solvent extraction. It is a manual or fully automotive technique (in conjunction with a robotic autosampler) and offers high reproducibility and is relatively inexpensive as SPME fibres can be used multiple times. SPME can be performed as a direct immersion procedure (DI-SPME) by exposing a phase-coated fibre into a liquid sample (Mallia et al. 2005), or as a headspace procedure (HS-SPME; Pawliszyn 1997; Januszkiewicz et al. 2008). DI-SPME is not that widely practiced for dairy products, due to the fact that it can adversely impact on the longevity of the fibres due to repeated swelling and drying, and fouling of the fibre can also occur which also adversely impacts on the ability of low-molecular-weight VOCs interaction with fibre phases (Heaven and Nash 2012). A main

advantage of SPME is that a wide range of sorbent phases are available; from single phases; polydimethylsiloxane (PDMS) and polyacrylate (PA); dual phases; carboxen (CAR)/PDMS, PDMS/divinylbenzene (DVB), carbowax polyethylene glycol (CW-PEG), CW/DVB, CW/TR (templated resin), or as triple phases; DVB/CAR/PDMS (Mondello et al. 2005; Yu et al. 2008; Heaven and Nash 2012; Jeleń et al. 2012). The type of fibre coating and thickness determine the properties in terms of polarity and retention, which affects the extraction efficiency, selectivity, reproducibility and discrimination of the extraction (Spietelun et al. 2010). A range of film thicknesses are also available; PDMS at 100, 30 and 7 µm, PA at 85 µm, PDMS/DVB at 65 and 60 µm, CAR/PDMS or CW/DVB at 75 and 65 µm and CW/TR at 50 µm.

The most convenient way to discuss the coating capacity independent of its characteristics as a solid or liquid sorbent is to consider the fibre constant as defined by Rivellino et al. (2013), where

$$Fc = KfsVf$$

Fc = fiber constant, Kfs = the, fiber coating/sample matrix distribution constant of the analyte, Vf = volume of the extraction phase.

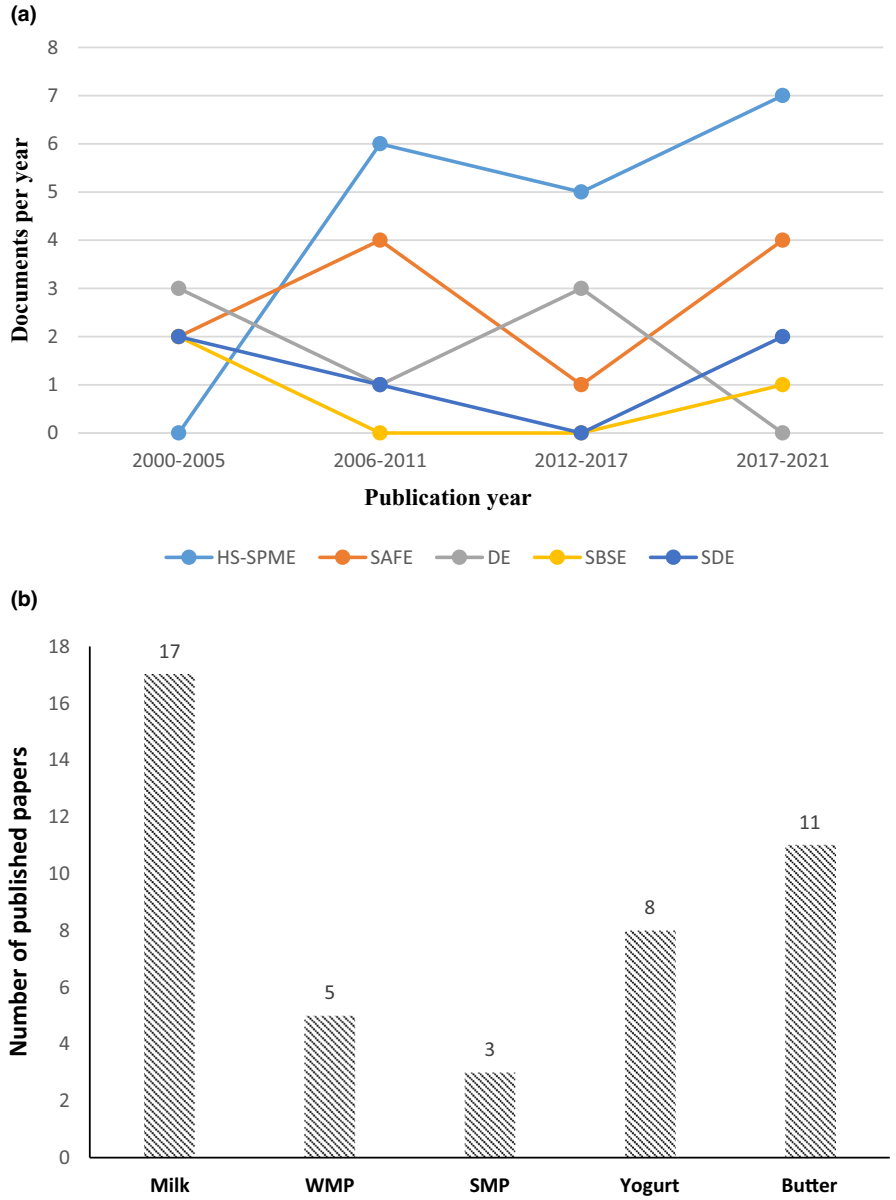

Figure 1 Applications of microextraction methods in selected dairy products. (a) Application of all extraction methods (HS-SPME, SAFE, DE, SBSE and SDE) used (b). Number of published studies based on Web of Knowledge search for years 2000–2021 incorporating all extraction techniques

Table 2 Volatile compounds found in milk (milk powders), butter, and yoghurt using GCMS by four common extraction methods

Compound	CAS no.	Extraction methods				Ref
		SPME	SAFE	DE	SBSE	
Alcohols						
(E)-2-Hexen-1-ol	928-95-0		✓			a
(E)-2-Nonen-1-ol	31502-14-4		✓			a
(E)-2-Octen-1-ol	18409-17-1		✓			a
(E)-2-Octenal	2548-87-0		✓			a
1,3-Butanediol	24621-61-2		✓			b
1,4-Butanediol	110-63-4	✓				c
1-Butanol	71-36-3	✓	✓	✓		a,d,e
1-Dodecanol	112-53-8	✓				f
1-Heptanol	111-70-6	✓	✓	✓		a,g,h,i
1-Hexadecanol	36653-82-4				✓	j
1-Hexanol	111-27-3	✓	✓	✓		a,e,i,j,l
1-Nonanol	143-08-8	✓	✓			a,i,k,l
1-Octadecanol	112-92-5		✓		✓	j,m
1-Octanol	111-87-5	✓	✓	✓	✓	a,h,i,j,l
1-Pentanol	71-41-0	✓	✓	✓		a,d,e,f,h,k,n,o
1-Penten-3-ol	616-25-1		✓			a
1-Phenylethanol	98-85-1				✓	j
1-Propanol	71-23-8		✓	✓		a,e,h
1-Tetradecanol	112-72-1				✓	j
2-(Methylthio)-ethanol	5271-38-5		✓			a
2,3-Butanediol	513-85-9	✓	✓			a,k
2-Butanol	78-92-2	✓	✓			b,i
2-Ethyl-1-hexanol	104-76-7	✓	✓			d,l
2-Furanmethanol	98-00-0	✓	✓	✓	✓	a,f,j,p,q
2-Heptanol	543-49-7		✓			b
2-Hexanol	626-93-7			✓		e
2-Methyl-1-butanol	137-32-6	✓				n
2-Methyl-1-propanol	78-83-1		✓	✓		a,e
2-Methyl-2-propanol	75-65-0			✓		e
2-Methyl-3-furanthiol	28588-74-1	✓	✓			q,r
2-Methyl-3-pentanol	565-67-3	✓				i
2-Nonanol	628-99-9	✓				i
2-Octanol	123-96-6	✓				f
2-Pentanol	6032-29-7		✓	✓		b,e
2-Phenethanol	60-12-8		✓			r
2-Phenoxyethanol	122-99-6		✓			b
2-Propanol	67-63-0	✓				i
3-Hexanol	623-37-0	✓		✓		h,i
3-Methyl-1-butanol	123-51-3		✓			a,b
3-Methyl-2-butanol	598-75-4	✓				b,k
3-Methyl-2-hexanol	2313-65-7		✓			b
3-Methyl-3-buten-1-ol	763-32-6	✓	✓			a,k
3-Octanol	589-98-0		✓			a
3-Pentanol	584-02-1	✓				k
3-Penten-2-ol	3899-34-1	✓				k
4-Methyl-1-pentanol	626-89-1	✓				k
4-Methyl-2-pentanol	108-11-2			✓		e
4-Methyl-2-pentanol	108-11-2		✓			b

(continued)

Table 2 (Continued).

Compound	CAS no.	Extraction methods				Ref
		SPME	SAFE	DE	SBSE	
Benzyl alcohol	100-51-6	✓	✓			a,d
Ethanol	64-17-5	✓	✓	✓		d,h,k,s,t
Ethyl furaneol	27538-10-9	✓				u
Furaneol	3658-77-3		✓			q
Heptanol	111-70-6	✓				i
Hexanol	111-27-3	✓				c
Isomaltol	3420-59-5				✓	j
Maltol	118-71-8		✓			w
Methionol	505-10-2		✓			a
Phenethyl alcohol	60-12-8		✓			a,l
Tetradecanol	112-72-1		✓			r
Aldehydes						
(E)-2-Heptenal[a]	18829-55-5	✓	✓			a,f
(E)-2-Hexanal	6728-26-3		✓			y
(E)-2-Nonenal	18829-56-6	✓	✓	✓	✓	a,g,j,r,w,x
(E)-2-Octenal	2548-87-0		✓			w
(E)-2-Undecenal	53448-07-0		✓			r
(E,E)-2,4-Decadienal	25152-84-5		✓			a,r
(E,E)-2,4-Heptdienal	4313-03-5	✓	✓			a,f,m
(E,E)-2,4-Nonadienal	5910-87-2	✓	✓			f,r
(E,Z)-2,4-Decadienal	25152-83-4		✓			r
(E,Z)-2,4-Nonadienal	5910-87-2		✓			m,w
(E,Z)-2,6-Nonadienal	557-48-2		✓			a,r,w,x
(Z)-2-Decenal	2497-25-8				✓	j
(Z)-2-Nonenal	60784-31-8		✓			w,x
(Z)-3-Hexenal	6789-80-6		✓			y
(Z)-4-Heptenal	6728-31-0		✓			m,r,w,x,y,δ
2,4-Decadienal[a]	2363-88-4	✓		✓		g,p
2,4-Hexadienal[a]	142-83-6			✓		e
2,4-Nonadienal[a]	6750-03-4			✓		p
2-Decenal[a]	3913-71-1	✓	✓	✓		a,f,p
2-Dodecenal	4826-62-4		✓			r
2-Ethyl-4-pentenal	5204-80-8	✓				f
2-Heptenal[a]	18829-55-5			✓		p
2-Methyl-2-propenal	78-85-3			✓		e
2-Methyl-butanal	96-17-3	✓	✓	✓		n,r,x,y,z
2-Methyl-propanal	78-84-2			✓		h
2-Nonenal	18829-56-6			✓		p
2-Octenal	2363-89-5			✓		p
2-Undecenal	2463-77-6			✓		p
3-Methyl-butanal	590-86-3	✓	✓	✓		i,r,y,α
4-Ethyl-benzaldehyde	4748-78-1	✓				p,q
6-Decenal	147159-48-6	✓				f
Acetaldehyde	75-07-0	✓				i,k,η
Benzaldehyde	100-52-7	✓	✓	✓		b,c,d,h,i,n,o,s,t
Benzeneacetaldehyde	122-78-1	✓				f
Butanal	123-72-8	✓				I,c
Decanal	112-31-2	✓	✓	✓	✓	a,d,i,j,k,m,o,r,t,w,y,α
Dodecanal	112-54-9				✓	j
Heptanal	111-71-7	✓	✓	✓	✓	a,d,g,j,n,o,r,t,w,y,α

(continued)

Table 2 (Continued).

Compound	CAS no.	Extraction methods				Ref
		SPME	SAFE	DE	SBSE	
Hexadecanal	629-80-1		✓			a
Hexanal	66-25-1	✓	✓	✓	✓	c,g,i,j,m,n,p,r,t,w,x,y,β,γ,δ
Methional	3268-49-3	✓	✓			r,s,w,y,δ
Nonanal	124-19-6	✓	✓	✓	✓	a,c,d,h,i,j,k,m,n,o,p,r,u,w,x,z,β,δ
Octanal	124-13-0	✓	✓	✓	✓	g,j,m,p,r,z,δ
Pentadecanal	2765-11-9		✓			a
Pentanal	110-62-3	✓	✓	✓	✓	d,g,j,n,r,t,α
Phenylacetaldehyde	122-78-1		✓			r
Pyruvaldehyde	78-98-8		✓			y
Tetradecanal	124-25-4	✓	✓			a,β
Tridecanal	10486-19-8		✓			δ
Undecanal	112-44-7	✓				g
Carbonyl compounds						
4-Ethylphenol	123-07-9		✓			m,y
Benzene	71-43-2	✓		✓		e,f
Ethyl benzene	100-41-4	✓		✓		g,h
Ethyl ether	60-29-7			✓		h
Guaiacol	90-05-1		✓			m,r,δ
m-Cresol	108-39-4		✓			b
m-Xylene	108-38-3	✓		✓		e,f,n
o-Xylene	1330-20-7	✓				n
Phenol	108-95-2	✓	✓			d,k,s,y
p-Xylene	106-42-3	✓				i
Styrene	100-42-5		✓			x
Toluene	108-88-3	✓		✓		c,d,e,h,f,k,n,t,z,α
p-Cresol	106-44-5	✓	✓		✓	f,w,y
Dehydro-p-cymene	1195-32-0		✓			c
Ketones						
1-Hexene-3-one	1629-60-3		✓			m,r,x
1-Hydroxy-2-acetone	116-09-6	✓	✓	✓		c,b,d,p
1-Nonen-3-one	24415-26-7		✓			r
1-Octen-3-one	4312-99-6		✓			w,x,y,δ
2,3-Pentanedione	600-14-6		✓			y
2-Aminoacetophenone	551-93-9		✓			m
2-Butanone	78-93-3	✓		✓		d,e,i,k,n,t,z,α
2-Decanone	693-54-9		✓			a
2-Heptanone	110-43-0	✓	✓	✓	✓	b,e,g,i,j,k,n,x,z,α,β
2-Hexadecanone	544-76-3		✓			a
2-Hexanone	591-78-6	✓		✓		e,k,z
2-Nonanone	821-55-6	✓	✓	✓		b,c,g,k,n,s,t,z,β
2-Octanone	111-13-7	✓	✓	✓		a,i,z
2-Pentadecanone	2345-28-0	✓		✓	✓	j,s
2-Pentanone	107-87-9	✓		✓		e,g,i,k,n,t,z,α
2-Tridecanone	593-08-8	✓				i,k,s,β
2-Undecanone	112-12-9	✓	✓			a,d,f,i,k,q,s,β
3-Hexanone	589-38-8			✓		e
3-Octanone	106-68-3		✓			o
3-Octen-2-one	1669-44-9	✓				g
3-Pentanone	96-22-0			✓		e
4-Decanone	624-16-8		✓			g

(continued)

Table 2 (Continued).

Compound	CAS no.	Extraction methods				Ref
		SPME	SAFE	DE	SBSE	
4-Methyl-2-hexanone	105-42-0			✓		e
4-Methyl-2-pentanone (methyl isobutyl ketone)	108-10-1	✓	✓	✓		e,k,o
5-Methyl-2-hexanone	110-12-3			✓		e
Acetoin	513-86-0	✓	✓			a, b,c,d,i,k,y,β
Acetone	67-64-1	✓		✓		c,d,g,i,k,n,t,α
Acetophenone	98-86-2			✓	✓	h,j
2,3-Butanedione (diacetyl)	431-03-8	✓	✓			b,c,i,k,r,t,u,w,x,y,ε,η
Sulcatone	110-93-0	✓		✓		i,z
Lactones						
Geranylactone	689-67-8	✓				i
Sotolone	28664-35-9		✓			r
γ-Butyrolactone	96-48-0		✓			l
γ-Crotonolactone	497-23-4				✓	j
γ-Decalactone	706-14-9	✓	✓			w,y,β
γ-Dodecalactone	2305-05-7	✓	✓			w,y,β
γ-Hexadecalactone	695-06-7		✓			β
γ-Nonalactone	104-61-0	✓	✓			r,x,ε
γ-Octalactone	104-50-7	✓	✓			r,β
γ-Tetradecalactone	2721-23-5		✓			y
γ-Undecalactone	104-67-6		✓			a
ε-Caprolactone	502-44-3	✓				d
σ-Decalactone	706-14-9	✓	✓		✓	f,j,l,o,r,s,t,w,x,y,β,ε
σ-Dodecalactone	713-95-1	✓	✓		✓	a,b,d,f,j,m,o,q,β
σ-Hexalactone	695-06-7	✓	✓			o,t,w,y
σ-Octalactone	104-50-7	✓	✓			o,t,u,w,x,y
σ-Tetradecalactone	2721-22-4		✓			y
σ-Undecalactone	104-67-6		✓			w
σ-Valerolactone	542-28-9		✓		✓	j,l
5-Ethyl-2(5H)-furanone	2407-43-4	✓				d,z,β
Sulphur compounds						
Dimethyl sulfide	75-18-3	✓	✓			k,r,t,x,η
Dimethyl sulfoxide	67-68-5		✓	✓		a,z
Dimethyl trisulfide	3658-80-8	✓	✓	✓		q,r,w,x,y,z,δ
Dimethyl disulfide	624-92-0	✓	✓	✓		m,r,s,z,α,δ
Dimethyl sulfone	67-71-0	✓	✓			d,f,i,o,l
Dimethyl tetrasulfide	5756-24-1		✓			t,δ
Dipropyl disulfide	629-19-6	✓				q
2-Methylthiophene	554-14-3		✓			y
Terpenoid compounds						
2-Carene	554-61-0	✓				v
3-Carene	3466-78-9	✓		✓		d,e,v
4-Terpineol	562-74-3			✓		e
Camphene	79-92-5			✓		e
Limonene[a]	5989-27-5	✓	✓	✓		b,k,o,e,h,v,y,α,β,λ
p-Cymene	99-87-6	✓	✓	✓		f,o,v
Sabinene	3387-41-5			✓		e
Squalene	111-02-4				✓	j
Styrene	100-42-5			✓		z
Terpinolene	586-62-9	✓				v
α-Pinene	7785-70-8	✓	✓	✓		d,e,t,v

(continued)

Table 2 (Continued).

Compound	CAS no.	Extraction methods				Ref
		SPME	SAFE	DE	SBSE	
α-Terpinene	99-86-5	✓		✓		e,v
α-Thujene	2867-05-2		✓	✓		e, y
β-Caryophyllene	87-44-5		✓			y
β-Pinene	127-91-3	✓	✓	✓		e,f,n,t,y
γ-Terpinene	99-85-4		✓			m
(E,E)-Farnesyl acetate	4128-17-0		✓			r
Esters						
2-Methyl-butyl-acetate	624-41-9		✓			r,δ
3-Phenylpropionate	2012-28-5	✓				k
Butyl acetate	123-86-4	✓		✓		k
Butyl benzoate	136-60-7	✓				a,b
Ethyl acetate	141-78-6	✓	✓	✓		c,h,n,t,x,y
Ethyl butanoate	105-54-4	✓	✓			b,f,r,x
Ethyl decanoate	110-38-3		✓			f
Ethyl heptanoate	106-30-9			✓		h
Ethyl hexadecanoate	628-97-7	✓	✓			a,β
Ethyl hexanoate	123-66-0		✓	✓		a,b,h
Ethyl lactate	97-64-3		✓			b
Ethyl nonanoate	123-29-5		✓	✓		a,h
Ethyl octanoate	106-32-1	✓	✓			a,b,n,r
Ethyl pentanoate	539-82-2			✓		h
Ethyl tetradecanoate	124-06-1	✓				β
Ethyl tridecanoate	28267-29-0		✓			a
Ethyl undecanoate	627-90-7			✓		h
Ethyl-2-hydroxy-3-methyl-butanoate	2441-06-7		✓			a
Ethyl-2-hydroxy-hexanoate	52089-55-1		✓			y
Ethyl-2-hydroxy-propanoate	687-47-8		✓			a
Ethyl-2-methyl-butyrate	7452-79-1		✓			y
Ethyl-3-hydroxly-butanoate	5405-41-4		✓			a
Ethyl-3-methyl-butyrate	108-64-5		✓			a
Ethyl-9-decenoate	67233-91-4		✓			a
Heptyl hexanoate	6378-65-0	✓				f
Hexyl acetate	142-92-7			✓		h
Isopropyl hexadecanoate	142-91-6				✓	j
Linalyl acetate	115-95-7		✓			l
Methyl butanoate	623-42-7		✓			μ
Methyl dodecanoate	111-82-0	✓				a
Methyl heptanoate	106-73-0			✓		h
Methyl hexadecanoate	112-39-0	✓			✓	f,j
Methyl isobutyrate	547-63-7			✓		h
Methyl octanoate	111-11-5	✓				f
Methyl tetradecanoate	124-10-7	✓				β
Phenylethyl acetate	103-45-7		✓			a,r
Diisobutyl phthalate	84-69-5	✓				F
Propyl benzoate	2315-68-6	✓				c
Furans						
2-Furan-methanol	98-00-0		✓			l
5-Hydroxymethyl-furfural	67-47-0				✓	j
5-Methyl furfural	620-02-0		✓			m
2,3-Dihydro-2methyl-benzofuran	1746-11-8	✓				f

(continued)

Table 2 (Continued).

Compound	CAS no.	Extraction methods				Ref
		SPME	SAFE	DE	SBSE	
2-Methyl furan	534-22-5	✓				f
Furfural	98-01-1	✓	✓	✓	✓	a,c,j,p
Homofuraneol	27538-09-6		✓			m
Hydroxy-2(5)H-furanone	14032-66-7				✓	j
Acids						
2-Methylbutanoic acid	116-53-0		✓			l
2-Methylpropanoic acid	79-31-2		✓			a,b,l,w
3-Methylbutanoic acid	503-74-2		✓			b,l,w,x,y
4-Methyloctanoic acid	54947-74-9		✓			r
9-Decenoic acid	14436-32-9		✓			l,w
Acetic acid	64-19-7	✓	✓	✓	✓	a,b,c,d,h,i,j,k,m,n,q,r,s,w,x,y,ε,λ
Benzoic acid	65-85-0	✓	✓			c,f,k,l,j,β
Butanoic acid	107-92-6	✓	✓		✓	a,c,d,f,j,k,l,i,n,q,r,s,w,x,y,ε,λ,β
Cyclohexylcarboxylic acid	98-89-5		✓			l
Decanoic acid	334-48-5	✓	✓		✓	a,b,c,f,j,k,i,l,m,o,r,s,w,β,δ
Dodecanoic acid	143-07-7	✓	✓			a,f,l,o,w,β
Formic acid	64-18-6		✓			l,w
Heptanoic acid	111-14-8	✓	✓			a,b,c,k,l,w,i
Hexadecanoic acid	57-10-3	✓	✓			o,β
Hexanoic acid	142-62-1	✓	✓	✓	✓	a,b,c,f,j,k,l,m,n,p,s,t,x,β,ε
Lauric acid	143-07-7		✓			a
Nonanoic acid	112-05-0	✓	✓		✓	a,b,c,f,j,k,l,o,t,β,ε
Octanoic acid	124-07-2	✓	✓		✓	a,c,f,I,j,l,k,o,s,w
Pentanoic acid (Valeric acid)	109-52-4	✓	✓	✓		a,b,i,h,l,m,r,w,y
Phenylacetic acid	103-82-2		✓			l,y
Propanoic acid	79-09-4		✓			a,b,d,w,l
Tetradecanoic acid	544-63-8	✓	✓			a,f
Undecanoic acid	112-37-8	✓	✓			a,f,l,β

Abbreviation: DE, Dynamic extraction; SAFE, Solvent-assisted flavour evaporation; SBSE, Stir bar sorptive extraction; SPME, Solid-phase microextraction.

The data adapted from (a) Ning et al. (2011), (b) Sarhir et al. (2021), (c) Su et al. (2017), (d) Cheng et al. (2020), (e) Ciccioli et al. (2004), (f) Coppa et al. (2011), (g) Clarke et al. (2019), (h) Rabaud et al. (2003), (i) Tian et al. (2017), (j) Faulkner et al. (2018), (k) Tian et al. (2019), (l) Miyaji et al. (2021), (m) Smith et al. (2016), (n) O'Callaghan et al. (2016), (o) High et al. (2019), (p) Francesca et al. (2015), (q) Mallia et al. (2014), (r) Evans et al. (2010), (s) Dadali and Elmaci (2019), (t) Garvey et al. (2020), (u) Abilleira et al. (2010), (v) Mahajan et al. (2004), (w) Lozano et al. (2007), (x) Bendall (2001), (y) Jansson et al. (2014), (z) Contarini and Povolo (2002), (α) Sarrazin et al. (2011), (β) Panseri et al. (2011), (γ) Evans et al. (2009), (ε) Martin et al. (2011), (η) Salum and Erbay (2019), (λ) Guneser and Yuceer (2011).

[a]Compounds are unidentified isomers.

HS-SPME extraction is considered complete when the analyte concentration has reached a distribution equilibrium between the sample, headspace and the fibre (Mondello et al. 2005). The efficiency of adsorptive extraction is dependent on the analyte surface concentration in the extraction phase at equilibrium and the surface area of the extraction phase (Musteata and Pawliszyn 2005). During the extraction process, the volume of VOC absorbed by the fibre phase is much faster than its release from the matrix, thus the requirement for sufficient time to obtain a representative VOC profile (Zabaras and Wyllie 2001). The length of extraction time and temperature are critical for SPME extraction efficiency. Generally, longer extraction times and high temperatures benefit the equilibrium resulting in increased responses of less volatile analytes (Fang and Qian 2005). However, care must be taken not to lose, create or enhance some VOC by the application of thermal treatments. The selectivity of HS-SPME is impacted by the selectivity of different phases towards specific solutes and various degrees of polarities. For example, larger less volatile compounds are captured by the porous DVB phase, while lower molecular weight highly volatile compounds

are captured by the porous CAR layer (Garcia-Esteban et al. 2004). DVB is composed of polymerized alkyl chains with phenyl groups creating a porous phase that is used in combination with PDMS to aid in attaching the DVB and to increase selectivity (Heaven and Nash 2012). The PDMS phase tends to capture low to medium polarity compounds, with PA more suitable for highly polar compounds (Mondello et al. 2005). PA fibres are made of partially cross-linked acrylic acid monomers and swell slightly in water (Heaven and Nash 2012). CAR consists of different sized pores that capture compounds and are used in combination with PDMS as this helps attach the CAR to the fibre but also enhances selectivity (Heaven and Nash 2012). CW/DVB has ability to extract a wide range of low- to mid-molecular-weight molecules (Carpino et al. 2004).

Merkle et al. (2015) mentioned that the binding of analytes to the matrix resulted in low concentrations of the analytes in the headspace in complex food matrices. Thus, the matrix effect is worth considering when developing a HS-SPME or any HS method for the extraction of VOC in dairy products. However, high temperatures during extraction can reduce the adsorption ability of SPME fibre for the target analytes because the adsorption of fibre is an exothermic process (Ng et al. 1999). Generally, longer extraction times and high temperatures benefits the equilibrium and increases the responses of less volatile analytes, but often at the cost of sensitivity and possibly increase artifact formation (Mariaca and Bosset 1997). However, the quantification of sulphur VOCs can only be achieved under non-equilibrium conditions using short extraction time, particularly for complex matrixes due to their inherent instability (Murray 2001; Nielsen and Jonsson 2002). In certain cases, low extraction efficiencies are reported, in particular for very volatile, polar or thermally unstable analytes (Namieśnik et al. 2000). This is likely related to the relatively low capacity of the sorbent phases on the fibre in comparison to many other sorbent type extraction techniques.

Most HS-SPME studies involving milk, yoghurt, butter or dairy powders have used the three phase fibres. DVB/CAR/PDMS is particularly useful for the detection of highly volatile sulphur, alcohol, terpenes, esters and acid compounds (Abilleira et al. 2010). However, the overall recovery of more polar compounds, especially free fatty acids by the DVB/CAR/PDMS fibre is poor (Mondello et al. 2005). Tian et al. (2017) used a 50-{m DVB/CAR/PDMS fibre to extract VOCs in yoghurt. These authors found that an extraction/equilibration time of 40 min at 55°C extracted 45 VOCs (aldehydes, alcohols, ketones, organic acids and sulphur compounds), with ketones and aldehydes the most abundant chemical classes, followed by alcohols, acids and sulphur compounds. Tian et al. (2019) subsequently extracted 54 VOC also in yoghurt samples using this same HS-SPME procedure (extraction time 40 min at 55°C) and fibre. Ketones, aldehydes and alkanes were the most abundant chemical classes followed by alcohols, acids, carbonyl compounds and sulphur compounds. O'Callaghan et al. (2016) investigated VOCs in sweet cream butter derived from cows milk using a 75-{m DVB/CAR/PDMS fibre. The butter was equilibrated at 40°C for 10 min, then the fibre was exposed to the headspace for a further 20 min at 40°C. In total, 25 VOC were extracted consisting of aldehydes, ketones, alcohols, acids, esters, a terpene and toluene, p-xylene and phenol. Garvey et al. (2020) investigated VOCs in salted butter using an optimised HS-SPME method with a 50/30 {m DVB/CAR/PDMS fibre, with a pre-equilibration of 10 min at 40°C, followed by a 60 min extraction time at 40°C. They identified 30 VOCs consisting of aldehydes, ketones, acids, hydrocarbons, lactones, sulphur compounds, esters, alkenes and a terpene and alcohol compound. This study highlighted that aldehydes, ketones, acids, terpenes and lactones were the main chemical classes contributing to the volatile profile of butter. Mallia et al. (2014) identified VOCs (aldehydes, ketones, acids, lactones, hydrocarbons, sulphur compounds and an alcohol) in sour cream butters from different countries also using a 50/30 {m DVB/CAR/PDMS fibre with an extraction temperature of 45°C for 45 min. Cheng et al. (2020) also used a 50/30 {m DVB/CAR/PDMS fibre to extract VOCs from SMP with an equilibration/extraction temperature of 40°C for 20 min. These authors extracted 26 VOCs (aldehydes, ketones, alcohols, terpenes, acids, a sulphur compound and a phenyl compound). Cheng et al. (2021) found the HS-SPME with DVB/CAR/PDMS appeared to be very effective at recovering terpenes and sulphur compounds in WMP, but much less effective at recovering lactones, furans and acids.

Clarke et al. (2019) optimized the extraction of VOCs associated with lipid oxidation in WMP (2.4 g made up with 3.5 mL distilled water). The authors used the 50/30 {m DVB/CAR/PDMS fibre and found that an extraction time of 45 min at 43°C using 2.4 g of sample gave achieved the best extraction efficiency for VOC recovery. For the vast majority of the VOC selected (aldehydes and ketones), the limits of detection (LOD) varied between 0.002 and 0.006 mg/L, with limits of quantification (LOQ) of 0.05 and 0.066 mg/L. The authors noted a matrix effect, which was due to the degree of interactions of VOC with the sample, which was more apparent for longer chain aldehydes, likely due to their affinity with the fat phase in the WMP. The authors also concluded that the influence of the sample amount is less important for the recovery of polar than for non-polar VOCs. Matrix interference is a major issue with VOC analysis in foods, but especially for lipophilic compounds (Abilleira et al. 2010). One main reason is that as the solubility of VOCs increases in a hydrophobic solvent, while the vapour-liquid partition coefficient decreases (Druaux et al. 1998). Abilleira et al. (2010) also utilised the 50/30 {m DVB/CAR/PDMS fibre to quantify terpenes in ewe's milk fat, using a pre-equilibration time of 10 min at 40°C,

followed by extraction at 40°C for 30 min. The authors noted that the matrix effect was a main reason for the overall systematic error to quantify terpenes (mono- and sesquiterpenes) in milk fat by HS-SPME.

Coppa et al. (2011) extracted VOC from the cream of cows milk derived from a hay-based diet or from continuous grazing of pasture. They also used the DVB/CAR/PDMS fibre. In this study, the cream was frozen and thawed in a HS vial at 60°C for 20 min in a water bath, and then incubated with the fibre exposed at 60°C for a further 20 min. Seventy-five VOCs were identified, and the study demonstrated that the DVB/CAR/PDMS fibre recovered VOCs with both high and low polarities. However, the relatively high temperature (60°C) used in this study, may induce artifact formation, or result in the higher abundance of some VOC (Mariaca and Bosset 1997). Dursun et al. (2017) used HS-SPME with 50/30 {m DVB/CAR/PDMS fibre at 55°C for 30 min to extract VOC from UHT milk to determine the correlations between individual aroma VOC and flavour attributes in UHT milks stored at the same conditions. A total of 43 VOCs (aldehydes, alcohols, ketones, acids, aromatic hydrocarbons, nitrogenous, sulphur containing compounds and an alkane hydrocarbon) were identified. The temperature of extraction (55°C) may again have resulted in increased abundance of some VOC or even artifact formation.

The bipolar CAR/PDMS has also been used extensively to extract VOCs from many dairy products, and it is particularly sensitive for the extraction of low-molecular-weight polar/apolar analytes (up until C6–C8) because of its porosity and the characteristics of its micropores (Shirey 2000; Mondello et al. 2005). The DVB-coated fibres contain relatively few micropores and CAR-coated ones contain a wide range of pores (micro-, meso- and macro-) in similar volumes (Elmore et al. 2000). Studies have shown that CAR/PDMS is very effective for the analysis of lower boiling point VOCs (Elmore et al. 2000; Januszkiewicz et al. 2008). Salum et al. (2017) optimized and compared the efficiency of DVB/CAR/PDMS and CAR/PDMS fibres for the extraction of specific VOC (3-methyl-1-butanol, ethyl lactate, 2-nonanone, ethyl octanoate, 2-ethyl-1-hexanol, butanoic acid, phenethyl alcohol, phenol, δ-decalactone and decanoic acid) in white-brined cheese. These authors found that optimum conditions for the CAR/PDMS fibre were 56.2°C for 84.92 min, slightly different to that for the DVB/CAR/PDMS fibre at 54.75°C for 85.60 min.

Other studies have shown that the CAR/PDMS fiber was more suitable for the extraction of low-molecular-weight VOCs such as 3-methyl butan-1-ol, ethyl lactate and butanoic acid and increasing the extraction time resulted in an increase in the volume of extracted VOCs for CAR/PDMS (Trujillo-Rodríguez et al. 2014). Martin et al. (2011) investigated the effect of oxidoreduction potential (Eh) on the biosynthesis of aroma compounds in non-fat yoghurt by HS-SPME using a 75-{m CAR/PDMS fibre for 40 min extraction time at 50°C. These authors demonstrated that the CAR/PDMS fibre was very sensitive for the extraction of acetaldehyde, dimethyl sulphide, 2,3-butanedione and 2,3-pentanedione. Su et al. (2017) also used the CAR/PDMS fibre to evaluate the VOC profile in yoghurt for 30 min at 60°C. They identified 30 VOC mainly consisting of aldehydes, ketones and acids plus some alcohols and esters; however, the increased temperature of extraction may have inadvertently enhanced the abundance of some VOC or even created new ones. Panseri et al. (2011) developed and validated a HS-SPME GC-MS method to quantify hexanal in butter to monitor lipid oxidation. They used an 85 {m CAR/PDMS fibre at 4°C for 180 min, and the low temperature was selected to minimise matrix oxidation and hexanal production during sampling. The results showed that CAR/PDMS fibre was especially sensitive to small molecules and suitable to monitor hexanal content both in fresh and oxidised butter samples.

SPME-Arrow has been developed to overcome the capacity limitation of traditional SPME as it has 6 to 20 times more volume capacity (Kim et al. 2020), but is also much less fragile. Manousi et al. (2020) compared a range of traditional SPME fibres (PDMS 100 {m), CAR/PDMS (75 {m), DVB/PDMS (65 {m) to SPME Arrow fibres (PDMS 100 {m), CAR/PDMS (120 {m) and DVB/PDMS (120 {m). These authors found that using CAR/PDMS SPME-Arrow out preformed their equivalent traditional fibre type by 4 or 5 times in terms of recovery using optimised conditions of 50°C for 60 min without salting out for 5 mL milk, but it was VOC dependent. However, to date very little studies have been published on SPME-Arrow on dairy products. In addition, another new SPME technique, thin film solid phase microextraction (TF-SPME), has been developed that has a very different geometry (a flat planar surface), that effectively increases the surface area-to-volume ratio and thus avoiding the usual caveats of increased phase volume (Bruheim et al. 2003). The simultaneous increase of extraction phase volume and surface area for TF-SPME (CAR/PDMS and DVB/PDMS) devices increases the potential for enhanced sensitivity with as good or better extraction rates compared to traditional SPME fibre (PDMS/DVB; Emmons et al. 2019); however, to date no studies in relation to dairy products appear to have been published.

Stir bar sorptive extraction

Stir bar sorptive extraction (SBSE) is another virtually solventless sample extraction technique available with two coatings (PDMS and PDMS with polyethylene glycol-modified silicone) of varying thickness (Ochiai et al. 2013). SBSE uses a small magnetic stir bar encased in glass and coated in sorbent material to detect the organic compounds. The principle of SBSE is based on the sorption of VOCs in an aqueous solution or semi-liquid matrix. A major advantage of SBSE is its high sensitivity towards semi-volatiles (Jeleń et al. 2012). The most widely used sorptive

extraction phase is PDMS. The choice of extraction coating is a key factor that determines the extraction performance, in terms of extraction efficiency, selectivity and dynamics. PDMS is a commonly used coating for SBSE, and it has a good adsorption performance for analytes with weak polarity through hydrophobic force (Fan et al. 2020). The amount of coating (PDMS) in SBSE is usually 50–250 times larger than traditional SPME with 1 cm length × 0.5 mm or 2 cm × 1 mm length film thickness, which increases the pre-concentration efficiency (Prieto et al. 2010). The PDMS coating on the stir bar acts as an immobilized liquid into which apolar analytes in an aqueous matrix can partition. The polar matrix components (including inorganic salts, carbohydrates, ionized acids and amines) do not partition well into the PDMS because of the apolar nature of the PDMS (Baltussen et al. 1999) that significantly aids it performance in extracting VOC as sample component interferences are greatly reduced. After sampling, the extracted analytes are recovered by thermal or liquid desorption and transferred respectively to a GC-MS system for analysis. Hoffmann and Heiden (2000) identified different VOCs in milk, condensed milk, cream cheese and yoghurt samples by SBSE coated with PDMS for 60 min at 30°C. The main VOCs detected were ketones, long-chain FFAs (C:10-C:16), lactones and sulphur compounds. Schiano et al. (2019) compared SBSE (PDMS), HS-SPME (DVB/CAR/PDMS) and solvent-assisted SBSE (SA-SBSE) (PDMS) to extract vitamin degradation VOCs from fluid skim milk. The extraction conditions involved submersing the stir bar in cyclohexane for 30 min at room temperature, drying then adding to milk at 25°C for 60 min. The results showed that SA-SBSE outperformed both SBSE and HS-SPME in terms of linearity, relative standard deviation and LOD and LOQ. High et al. (2019) compared SBSE, to SAFE, HS-SPME and HS sorptive extraction (HSSE) on reconstituted spray-dried sheep milk. The authors prepared sheep's milk powder in deionized water to 20% solids (w/w). The sample preparation for SBSE (PDMS) involved immersion at 35°C for 90 min and similar conditions for HSSE analysis. For HS-SPME the reconstituted sheep milk was extracted for 60 min at 35°C SPME using the 50/30 µm DVB/CAR/PDMS fibre. For SAFE analysis 250 g reconstituted sheep milk was mixed with 100 mL of dichloromethane and distilled in the SAFE apparatus over a period of approximately 3.5 h. The organic layer was collected and dried with Na_2SO_4 (anhydrous) at room temperature under a stream of nitrogen at 100 mL/min. The authors found that SBSE was the most effective technique, with good selectivity, sensitivity and reproducibility from small sample volumes, although as anticipated some VOC selectivity exists for each technique. Typically extraction times for SBSE are longer than HS-SPME due to the enhanced phase volume, as additional time is required to enable the VOC interact with the phase.

High capacity sorptive extraction

A new high capacity passive SE technique called HiSorb (Markes International Ltd, Bridgend, UK) has been developed that is somewhat similar to SBSE, but more automatable and can also be performed as a headspace (HS) or as a direct immersion (DI) technique (Lancas et al. 2009). Cheng et al. (2021) compared DI-HiSorb (PDMS), HS-HiSorb (PDMS), TD (Tenax/Carbograph) and HS-SPME (DVB/CAR/PDMS) for the extraction of VOCs from WMP, which was reconstituted at 10% solids in ultra-pure deionized water overnight at 4°C prior to evaluation. These authors found DI-HiSorb using a non-polar GC column identified more aldehydes, ketones, lactones, esters and terpenes than HS-SPME at 40°C for 120 min. These authors also found that DI-HiSorb was particularly effective in extracting lactones in comparison to all the other extraction techniques. Faulkner et al. (2018) compared the efficiency of HS-SPME (DVB/CAR/PDMS) and DI-HiSorb (PDMS) for the extraction of VOCs in pasteurized milk samples. These authors found that an extraction/equilibration time of 60 min at 37°C by DI-HiSorb method achieved good results for pasteurized milk samples and identified 38 VOC from a range of different chemical classes, slightly more than the 36 VOC extracted by HS-SPME. Some lactones (γ-crotonolactone, σ-valerolactone, σ-decalactone, σ-dodecalactone) and p-cresol were only identified using DI-HiSorb. Clarke et al. (2022) also used DI-HiSorb (PDMS) at 40°C for 1 h to extract volatiles in raw milk and managed to successfully identify 99 VOCs consisting of acids (20), alcohols (17), aldehydes (16), esters & ethers (9), furans (3), hydrocarbons & benzenes (7), ketones (10), lactones (5), pyrazines & pyridines (4), sulphur VOC (3) and others (5).

Solvent-assisted flavour evaporation

SAFE is an extraction technique which allows the separation and concentration of volatiles by vacuum distillation. SAFE has been shown to extract a great number of aroma compounds from different chemical classes in food (Huang et al. 2019; Zhou et al. 2019). The SAFE distillation system consists of a vacuum pump and usually two cooling traps of liquid nitrogen. The sample is mixed with a solvent (usually diethyl ether or dichloromethane), and the VOCs are collected by distillation with the solvent in the first trap, while impurities and the water condense in the second trap. Engel et al. (1999) provided an overview of the procedure, where they undertook distillation for 36–240 min under vacuum (104–106 Pa) at 40–70°C using a circulation water bath. After distillation, the sample was concentrated under a stream of nitrogen and transferred to a screw-top glass tube for phase separation. SAFE enables the extraction of VOC without extensive preparation; however, it is time consuming and expensive due to the requirements for specialist glassware. It is often frequently associated with GC-O

analysis, due to the preservation of the heat labile volatiles and lack of artifacts created through extraction at low temperature (Whetstine et al. 2006; Evans et al. 2010; Sonmezdag 2019). However, distillation-extraction techniques are becoming less favourable due to the volumes of solvents required, the time required and the variable recovery rate of highly volatile compounds (Jeleń et al. 2012).

Ning et al. (2011) compared SAFE, SDE and HS-SPME (75 µm CAR/PDMS, 65 µm PDMS/DVB and 50/30 µm DVB/CAR/PDMS) to detect VOC of fermented camel milk. A total of 26 aroma-active VOC were detected by GC-O by SAFE with dichloromethane (20 mL) at 60°C for 30 min. Compared with other pre-treatment methods, the results from SAFE proved to be effective for less volatile and more polar components (mainly alcohols and esters), but also extracted many low boiling points components such as acetaldehyde, ethanol and ethyl acetate. Smith et al. (2016) characterized the VOC profile of milk protein concentrates (MPC 70, 80, 85), milk protein isolates (MPI), acid casein, rennet casein and micellar casein concentrate (MCC) by SAFE and HS-SPME. The caseins, MPC/MPI and MCC powders were reconstituted to 10% (wt/vol) in a sodium chloride solution, and extraction was performed for 30 min at 40°C by HS-SPME with DVB/CAR/PDMS fibre. The 30 mL reconstituted powder sample was mixed with 100 mL of diethyl ether and SAFE extraction carried out for 40 min at 50°C. The extracts were concentrated under a stream of nitrogen to 20 mL. The VOCs were extracted by HS-SPME and by SAFE. SAFE detected 24 VOC not detected by HS-SPME, and HS-SPME detected 30 compounds not detected by solvent extraction (SAFE). These results highlighted that SAFE tends to favour the extraction of higher molecular weight VOCs. Evans et al. (2009) also used HS-SPME (DVB/CAR/PDMS) and SAFE to extract VOCs in milk serum protein concentrates and in whey protein concentrates (reconstituted at 10% solids, with 10% NaCl). These results demonstrated that SAFE (with 15 ml ethyl ether solvent) recovered different classes of VOCs compared with HS-SPME (DVB/CAR/PDMS) at 40°C for 25 min. Mahajan et al. (2004) investigated aroma compounds in sweet whey powder. One killogram of sweet whey powder was isolated by solvent (500 mL of 2:1 freshly distilled pentane and diethyl ether solution) extraction followed by SAFE. The most aroma-intense compounds detected by SAFE were short-chain fatty acids, aldehydes and ketones, lactones, sulphur compounds, phenols, indoles, pyrazines, furans and pyrroles. As mentioned previously, High et al. (2019) compared SAFE to HS-SPME (DVB/CAR/PDMS), HSSE (PDMS) and SBSE (PDMS) for the extraction of VOCs in spray-dried sheep milk. These authors found that SAFE was the only extraction technique capable of extracting high concentrations of both the small polar sulphur compounds (dimethyl sulfone) and also larger less volatile lactones. The diethyl ether and dichloromethane solvent were investigated in their preliminary experiment, and only dichloromethane was selected for the sheep milk SAFE extraction. This study confirmed that SAFE is suitable to extract highly polar and higher molecular weight VOCs, but is dependent upon the solvent employed. However, the authors found that SAFE was the least reproducible and the least efficient of the methods evaluated. Miyaji et al. (2021) employed SAFE (100 g yoghurt samples with 100 mL dichloromethane stirred at room temperature for 1 h) to investigate off-flavours from pasteurized drinking yoghurt made from skim milk during long-term ambient storage. Seventy eight VOC were identified. The results demonstrated that SAFE is very useful in extracting highly volatile compounds which are representative of yoghurt. Lozano et al. (2007) compared DHA (10 g butter were purging of the headspace volatiles onto a Tenax TA adsorbent tube by nitrogen at 40°C for 25 min) to SAFE (112 g butter combined with 440 mL diethyl ether at 30°C for 30 min) to analyse aroma compounds in commercial sweet cream butter by GC-O. A total of 32 and 27 aroma-active compounds were identified by SAFE and DHA, respectively. Some highly volatile compounds such as dimethyl sulphide were lost during workup and concentration using SAFE; however, less volatile compounds such as lactones were better recovered by SAFE than DHA. Sarhir et al. (2021) investigated the VOC profile of Moroccan fermented-salted 'Smen' butter and compared purge-and-trap extraction (PTE) at 36°C for 15 min with Lichrolut EN (200 mg) sorbent to SAFE (30 g butter sample with 80 mL of diethyl ether solvent) at 40°C for 30 min. A total of 27 and 30 aroma compounds were identified by the PTE and SAFE, respectively, but significant differences in the VOCs extracted existed between both methods. The results demonstrated that SAFE was more efficient in the extraction of carboxylic acids than PTE, and the aroma-active compounds detected using SAFE had higher flavour dilution (FD) factors demonstrating that greater concentrations were extracted.

Dynamic extraction (DE)

In dynamic methods, such as purge and trap (P&T) and TD, the dairy sample is typically heated and the VOCs continuously removed and subsequently concentrated in a cold trap, or adsorbed onto an inert support prior to injection onto the GC. Valero et al. (1997) described the general process used in TD, where VOC are trapped into TD tubes using an inert gas such as nitrogen or helium. In their study, tubes were subsequently desorbed to cold trap to aid peak focussing prior to desorption to the GC. A wide range of absorbent and adsorbent trapping materials are available and flows can be controlled to split extracts that gives a lot of possibilities to enrich or dilute extracts with relative ease. Samples amounts can be relatively large as the loading capacity of the tubes are large which is beneficial for trace analyte

detection (Valero et al. 1997). Cheng et al. (2021) evaluated WMP using TD and had additional equipment such as a Micro-Chamber/Thermal Extractor (μ-CTE) (Markes International Ltd) that provided greater control in the process. These authors used a Tenax/Carbograph sorbent and found that TD was more effective at extracting aldehydes, ketones, alcohols and benzene/phenols, but ineffective for lactones. However, some VOC that were not extracted by DI-HiSorb, HS-HiSorb, or HS-SPME but were detected by TD (longifolene, α-terpineol, 1-nananol, p-xylene and 2,3-pentanedione).

P&T is a good technique for the detection of highly volatile compounds with lower boiling points, such as alcoholic compounds and is solvent-free (Mallia et al. 2005), but has generally been surpassed by more automatic extraction methods. Using the P&T technique, the dairy sample is usually homogenized with water, placed in a U-shaped glass sparger and heated. Subsequently, an inert gas (nitrogen or helium) is purged through the sample to transfer the VOCs to an inert support of trapping material, which is thermally desorbed and concentrated once again in a cold trap (cryofocusing) before injection onto the GC-column. A wide range of trapping materials are available. Contarini and Povolo (2002) compared to P&T (at room temperature for 60 min with a Tenax trap) and HS-SPME (at 45°C for 30 min with DVB/CAR/PDMS fibre). Both P&T and HS-SPME were comparable in terms of repeatability. The results demonstrated that 11 VOCs were obtained from the milk samples by both PT and HS-SPME. The P&T technique was also better able to extract smaller molecular weight VOCs (such as, acetone and 2-butanone). Naudé et al. (2009) developed a novel P&T sampling method to extract VOCs from long life UHT milk (2% milk fat) by trapping it on a multi-channel open tubular traps of PDMS for at 45°C for 35 min in nitrogen at 25 mL min^{-1}. The VOCs were subsequently desorbed from the cold traps to the GC using a TD-type system. The authors found that dimethyl sulphide, 2-methylpropanal, 2,3-butanedione, 3-methylbutanal, 2-hexanone, 2-heptanone, 2-nonanone, nonanal and decanal were the predominant VOCs in these samples. Francesca et al. (2015) exploited the potential applications and setup conditions of the automated Gerstel TD (Gerstel GmbH & Co, M lheim, Germany) using microporus sinthered glass (TDU-CIS4–GC–MS) and cryogenic trapping for the identification of oxidized or non-oxidized volatile compounds of powdered milk at 30°C for 30 min. They identified 17 VOC mainly consisting of aldehydes, ketones, acids and alcohols. Ciccioli et al. (2004) developed a multiple dynamic headspace extraction TD system for the accurate determination of VOC in goat milk samples. The authors used a series of different traps in an attempt to obtain as true a volatile profile as possible (Tenax and different types of Carbograph) where helium was passed through the sample at 200 mL min^{-1} at 50°C to dynamically extract the VOC onto the tubes. The authors also used a colder empty trap before the packed traps in an attempt to reduce moisture getting onto the packed traps. These authors identified 33 VOC mainly consisting of aldehydes, ketones, terpenes, alcohols and benzene compounds in goat's milk.

VOLATILES - MILK, DAIRY POWDERS, BUTTER AND YOGHURT

Key volatiles associated with the aroma of milk and dairy powders

VOC including aldehydes, ketones, alcohols, lactones, phenols and esters in milk products originate from the degradation of the major milk constituents (lactose, citrate, milk lipids and milk proteins) (Cadwallader and Singh 2009), but many are also created through rumen metabolism can also be directly transferred from diet (Kilcawley et al. 2018; Clarke et al. 2022).

Typically, the most abundant VOC chemical class in many dairy products are short-chain carboxylic acids, known to be major components responsible for the sour taste (Coppa et al. 2011; Villeneuve et al. 2013) and in some cases rancidity (Kilcawley et al. 2018). There are derived from various sources and pathways; lipolysis, carbohydrate metabolism or amino acid metabolism depending upon the specific carboxylic acid (Kilcawley et al. 2018). Simple acids (<6 carbon) have high odour thresholds, while long chain acids (12 or more carbons) are odourless. Unsaturated acids generally have sharper and stronger odours than saturated ones (Jeleń et al. 2012). A recent study by Clarke et al. (2022) found that butanoic acid (cheesy, dairy, buttery) was a major contributor to the aroma of raw cow's milk. Karagül-Yüceer et al. (2001) determined that butanoic (cheesy), hexanoic acids (cheesy), octanoic acid (waxy, soapy) and dodecanoic acids (fat, sweet) were detected at high odour intensities in the acidic fraction of nonfat dry milk. Karagül-Yüceer et al. (2002) also found that octanoic, nonanoic, decanoic and dodecanoic acids were associated with soapy/waxy/rubbery attributes in stored nonfat dry milk. These authors also found that octanoic and decanoic acids had very high FD factors and that sour taste was correlated with pentanoic acid. Moreover, propionic acid, 2-methylpropionic acid, 2-/3-methylbutanoic and pentanoic acids with sweaty or Swiss cheese-like aroma notes were present in the acidic fractions of these nonfat dry milks.

Primary aldehydes are mainly derived from oxidation of polyunsaturated fatty acids (PUFA), but can also be transferred from plant material into milk (Kilcawley et al. 2018; Clarke et al. 2022). The impact of oxidation on VOC generation in milk and in many dairy products is significant, as the fatty acid profile of milk, especially PUFA is greatly impacted by diet (O'Callaghan et al. 2019). The chain

length of aldehydes mostly affects odour thresholds and odour properties. Aldehydes with low molecular weights (<150 Da) tend to be associated with unpleasant odours, and those with higher molecular weights tend to have sweet, fruity odours (Giri et al. 2010). Milk produced from the cows fed pasture (perennial ryegrass or perennial ryegrass and white clover) was higher in linolenic acid content, which is known to influence the degree of lipid oxidation (O'Callaghan et al. 2016). Havemose et al. (2006) found the level of other primary aldehydes such as hexanal, heptanal and pentanal increased in milk produced from cows fed grass/clover silage after exposure to fluorescent light compared to milk produced from a hay diet. Feeding pasture has also been shown to significantly elevate the levels of 2-nonenal, hexanal and octanal in milk (Glover et al. 2012). Pentanal is a product of the autoxidation of arachidonic and linoleic acid and was found at greater intensities in milk from cows fed pasture and silage than in milk from cows fed just hay (Villeneuve et al. 2013; Clarke et al. 2020a). Pentanal has also been associated with the cardboard-like or metallic-like off-flavours in milk after prolonged exposure to light (Zardin et al. 2016). Francesca et al. (2015) associated pentanal, hexanal, octanal, 2-heptenal, nonanal, 2-octenal, 2-nonenal, 2-decenal, 2,4-nonadienal, 2-undecenal, 2,4-decadienal with oxidation in powdered milk, defined as 'pungent', 'green (or herbaceous)', 'fat' and 'food-fried'. Boltar et al. (2015) noted that the primary aldehydes nonanal and octanal (products of lipid-oxidation) were significantly higher in milk produced from winter grass silage also highlighting an impact of diet on lipid oxidation. Coppa et al. (2011) found higher benzeneacetaldehyde concentrations in milk from cows on rotational grazing than in milk from a hay-based diet, or from cows on continuous grazing. Benzeneacetaldehyde is primarily derived from phenylalanine metabolism, but may also be transferred directly into the diet (Coppa et al. 2011; Kilcawley et al. 2018; Clarke et al. 2021). The Strecker aldehydes 2- and 3-methylbutanal (grassy, fatty, astringency and painty) were found to be more abundant in WMP produced from milk of cows fed hay than cows fed diets of maize silage or grass silage and results from the metabolism of isoleucine and leucine, but are also involved in the Maillard reaction (Lloyd et al. 2009).

Ketones are also mainly derived from oxidation of FA in dairy products, but some are also the result of carbohydrate metabolism, it has been suggested that many may not have a significant impact on milk flavour due to their relatively higher odour thresholds and relatively low concentration (Kilcawley et al. 2018). In heat-treated milk, ketones are mainly products of the heat-initiated decarboxylation of β-oxidized saturated fatty acids or decarboxylation of β-ketoacids (Jansson et al. 2014). Contarini et al. (1997) noted that ketones having a higher carbon number are responsible for heated milk flavour. These authors demonstrated that the abundance of 2-heptanone and 2-pentanone increased in milks stored at room temperature and were responsible for heated milk flavour. Moreover, acetone and 2-butanone were also lower in UHT milk and are thought to derive mainly directly from cow's diet (Contarini et al. 1997). Coppa et al. (2011) found that 2,3-octanedione was more abundant in milk derived from diverse pastures and suggested this was due to oxidation of linoleic acid and linolenic acid. Clarke et al. (2020b) found that 3-octen-2-one was correlated with 'caramelised flavour' and 'sweet taste' in WMP. Vazquez-Landaverde et al. (2005) noted that 2-pentanone, 2-hexanone, 2-heptanone, 2-nonanone and 2-undecanone have been identified as thermally derived off-flavours linked to the level of fat in the milk. Clarke et al. (2020b) found that 3,5-(E,E)-octadien-2-one (grassy, fruity and green), a product of linolenic acid oxidation, was significantly higher in pasteurised milk derived from concentrate and correlated with hay-like flavour. Clarke et al. (2022) found that 2,3-butanedione, a product of pyruvate metabolism (fresh, sweet, caramel, butterscotch, biscuit and baked), was a key odourant of milk from cows outdoors on pasture (perennial ryegrass).

Sulphur volatiles are potentially very important aroma compounds due to their high odour activities (Falchero et al. 2010). Kobayashi et al. (2008) found methyl 2-methyl-3-furyl disulphide, furfuryl methyl disulphide and bis(2-methyl-3-furyl) disulphide were present in high heat-treated SMP and in UHT milk, which presented a 'canned corn-like', 'rice bran-like' and 'vitamin-like' odour profile. Vazquez-Landaverde et al. (2005) found that dimethyl sulphide was almost three times higher in UHT than in raw milk and was formed from the sulfhydryl group of milk proteins subjected to thermal denaturation. Clarke et al. (2022) found that methanethiol (cabbage) was an important odorant in raw cow's milk from pasture (perennial ryegrass).

Terpenes are naturally occurring plant secondary metabolites derived from isoprene units (C5) and also derived from larger terpenoids; monoterpenes (C10) and sesquiterpenes (C15). Terpenes are odour active but have a high odour threshold and therefore need to be at high concentrations to have a sensory impact (Kalač 2011). Ciccioli et al. (2004) noted the maximum monoterpene (α- and β-pinenes) content in milk was associated when the largest variety of herbs was present in the pasture. Faulkner et al. (2018) also found that β-pinene is most likely derived directly from forage, but concentrations are dependent upon the diversity of the pasture. These authors also found that β-pinene was absent in cow's milk derived from a concentrate diet. Coppa et al. (2011) found the concentrations of β-pinene and cymene-(p) and all sesquiterpenes (β-caryophyllene, alloaromadendrene, germacrene-D and γ-cadinene) were higher in milk derived from animals on continuous grazing than on less diversified pasture under rotational grazing. Coppa et al. (2011) also found that sesquiterpenes were more

influenced by different grazing systems than monoterpenes. Limonene (sweet citrus-like) is also a product of bioconversion of sesquiterpenes and was the most common terpenes in milk from a range of highland and lowland pastures (ryegrass, clover) or from concentrates (maize silage, hay and cereals) over different seasons (Fernandez et al. 2003).

Phenolic compounds can be important volatile compounds in milk related to forage intake. Alkylphenols in ruminant milks are derived from phenolic compounds ingested through feed and were responsible for the 'cowy flavour' of milk (Feo et al. 2006). p-Cresol is a major alkylphenol and has a characteristic 'barn-like flavour' that blends with the more medicinal notes of m-cresol in milk (Ha and Lindsay 1991). Faulkner et al. (2018) found a direct link between p-cresol levels in raw milk from cows fed clover with 'barnyard aroma', which was also subsequently linked to isoflavone metabolism by Clarke et al. (2019). Karagül-Yüceer et al. (2002) noted that p-cresol and skatole may be the contributors to undesirable flavours in milk. Phenols (clove-like, medicinal and smoky) are described as heat-generated compounds in UHT milk (Dursun et al. 2017). Most phenolic compounds are excreted, but some end up in milk and depending upon their abundance may influence sensory perception.

Hydrocarbons compounds with high odour thresholds can also play an essential role in food aroma when present at high concentrations (Czerny et al. 2011). Toluene, a product of β-carotene light-induced oxidation, has been implicated as responsible for rancid notes and was more abundant in pasture-derived milk than milk from cows fed indoors (Coppa et al. 2011). Faulkner et al. (2018) found toluene was significantly higher in CLV milk than concentrate milk and linked as a potential biomarker for pasture, derived from metabolism in the rumen. Xylene (sweet) may be the result of carotenoid degradation, namely β-carotene degradation in the rumen or possibly directly transferred from feed (Buchin et al. 1998).

Lactones are cyclic compounds formed by the intramolecular esterification of hydroxyacids through the loss of water, described as having a buttery-type, creamy, fruity or otherwise pleasant odour. Few differences in lactone content were linked to diet, but they appear to be more important in pasteurized milk than in raw milk because heat is a factor in their production (Urbach 1997; Li et al. 2020). Villeneuve et al. (2013) found the detection intensity of δ-octalactone and δ-tetradecalactone were affected by forage types. In their study, the content of γ-decalactone, γ-dodecalactone and γ-dodecaenolactone in milk was higher in hay-fed cows, lower in silage-fed cows and intermediate for cows on pasture. Karagül-Yüceer et al. (2002) showed that δ-decalactone and γ-dodecalactone gave sweet odour properties to milk powder. Sweet and milky odour properties were characterized by lactones including γ-undecalactone, γ-dodecalactone, δ-decalactone and δ-undecalactone. Clarke et al. (2022) found that γ-butyrolactone was an important odour active volatile in raw cow's milk, and that γ-hexalactone influenced the aroma of cow's milk produced from a concentrate diet.

Key volatiles associated with the aroma of butter

Garvey et al. (2020) found pentanal (paint-like) and decanal (green, fatty), derived from oleic acid and linoleic acid (also arachidonic acid for pentanal), were more abundant in butter produced from cows outdoors fed perennial ryegrass and white clover than perennial ryegrass alone or from cows indoors fed concentrate. In their study, heptanal was significantly more abundant in butters produced from milk derived from a pasture (perennial ryegrass or perennial ryegrass and white clover) in comparison to a concentrate diet and has a 'green sweet' aroma. Glover et al. (2012) noted butanoic acid levels were higher in butter produced from milk from cows fed the concentrate compared with pasture. Butanoic acid is likely a very important aroma compound in butter and was a main contributor to 'fresh butter' aroma in sweet cream butter (Lozano et al. 2007). O'Callaghan et al. (2016) found that acetone (earthy, strong fruity and hay) was significantly correlated with butter produced from milk derived from cows outdoors on perennial ryegrass and white clover diets, than in butter produced from milk from cows outdoors on perennial ryegrass, or indoors on concentrate. These same authors also found 2-butanone (buttery, sour milk and etheric) was significantly more abundant in butter produced from concentrate diets. Mallia et al. (2008) found that the concentrations of 1-octen-3-one (mushroom) increased when butter oil was stored at room temperature. Li et al. (2020) found 3-penten-2-one is a product of lipid oxidation, with low levels indicating freshness in butter. 2,3-Butanedione is a very odour-active compound with a characteristic buttery aroma, derived from pyruvate (Liu et al. 2020b). Garvey et al. (2020) found 2,3-butanedione was significantly more abundant in butter produced from milk derived from cows outdoors fed perennial ryegrass and white clover compared to butter produced from cows indoors on a concentrate diet. Li et al. (2020) found that δ-decalactone was the most important odour active aroma in butter, and that overall lactones in general were important odour compounds in butter. Lozano et al. (2007) compared the VOC profile of fresh sweet cream butter and butters stored at refrigeration (4°C), frozen (−20°C) and at room temperature. These authors identified butanoic acid, δ-octalactone, δ-decalactone, 1-octen-3-one, 2-acetyl-1-pyrroline, dimethyl trisulphide and 2,3-butanedione as the most intense aroma compounds associated with fresh butter samples and that dimethyl sulphide is possibly a contributor to cooked/nutty flavour in butter, which is in agreement with Peterson and Reineccius (2003. Lozano et al. (2007) also noted that the main changes in aroma active VOC over storage were related to an increase in the intensity of lactones (δ-octalactone, δ-decalactone and

δ-dodecalactone), lipid oxidation VOC ((E)-2-nonenal, 2-heptanone, (Z)-4-heptenal, (E,Z)-2,6-nonadienal and hexanal) and acidic odourants such as acetic and butanoic acids. These authors also noted that styrene levels increased over storage due to migration from packaging material and may adversely impact on fresh butter flavour.

Lozano et al. (2007) also suggested that toluene (nutty, bitter, almond and plastic) may be associated with stale butter flavour it is a product of β-carotene degradation and has been previously shown to be significantly higher in butter derived from milk of cows fed outdoors on pasture (perennial ryegrass or perennial ryegrass and white clover) than cows indoors fed concentrate (O'Callaghan et al. 2016).

Key volatiles associated with the aroma of yoghurt

Acetaldehyde, predominantly derived from pyruvate decarboxylation or generated by the metabolism of threonine, is a major aroma compound in yoghurt and exhibits a green apple or nutty flavour (Cheng 2010; Eram and Ma 2013; Settachaimongkon et al. 2014). Tian et al. (2017) demonstrated that the concentration of acetaldehyde increased after the end of fermentation, reached a maximum at the beginning of storage and then declined sharply with increasing storage time. This study highlighted that yoghurt samples fermented with a *Lactobacillus acidophilus* culture produced the highest concentrations of acetaldehyde in comparison to other strains evaluated. Tian et al. (2019) also demonstrated that acetaldehyde contributes a 'green apple' or 'nutty' attribute at lower concentrations, but negatively influences aroma at high concentrations. 2,3-Butanedione and acetoin are produced by pyruvate or citrate metabolism by various lactic acid bacteria and are typical carbonyl compounds and contribute greatly to the 'butter and cream' aroma of yoghurt (Hugenholtz 1993; Neves et al. 2005). Acetoin derives from the enzymatic degradation of 2,3-butanedione and although has a much weaker aroma than 2,3-butanedione helps to contribute to a 'mild creamy' aroma in yoghurt (Cheng et al. 2010). Innocente et al. (2016) found that a 1:1 acetaldehyde to 2,3-butanedione ratio gave the most preferential yoghurt aroma, while too much acetaldehyde resulted in a 'green off-flavour'. A study by Tian et al. (2017) demonstrated that 2,3-butanedione and acetoin reached maximum concentrations after 14 days refrigerated storage post production. Acetoin, 2,3-butanedione and 3-heptanone are also all known to contribute to yoghurt odour by providing 'fruity, sweet' aromas (McSweeney and Sousa 2000; Gallardo-Escamilla et al. 2005). Tian et al. (2017) reported that 2-butanone, 2-pentanone and 2-heptanone (originating from oxidation, carbohydrate metabolism and/or direct transfer) were all identified as significant aromatic volatiles in yoghurt samples fermented with *Lactobacillus casei*. Short-chain fatty acids are also produced during yoghurt fermentation by both lipolytic processes and by lactic acid starter fermentation (Tamine and Richard 2007). Acetic acid, one of the important acidic compounds produced by hetero fermentative LAB, contributes an undesirable vinegar taste at high concentrations, which can unbalance the overall flavour (Buffa et al. 2004). However, as acetic acid is not that odour active, excessive levels are generally not a major issue in yoghurt production. Innocente et al. (2016) demonstrated that both hexanoic and butanoic acids were significantly higher in yoghurt samples fermented with *Lactobacillus casei* than yoghurt fermented with *Lactobacillus rhamnosus*. Tian et al. (2019) documented that butanoic and octanoic acids contribute to the characteristic cheese flavour of yoghurt, and that decanoic acid provides a 'light cream' flavour. Some alcohol compounds also contribute to the aroma of yoghurt. Lower alcohols (from C1 to C10) affect the flavour of yoghurt and can be important as they positively influence sensory perception (Cheng 2010). Ethanol, the final product of glucose metabolism or amino acid degradation in milk, is thought to influence sweetness (Urbach 1995), but unlikely to have a major contribution due to its very high odour threshold. Tian et al. (2019) demonstrated that 3-pentanol and 1-hexanol contribute to a 'grass' flavour in yoghurt. 1-Pentanol and 2,3-butanediol were demonstrated to provide a 'fruit' flavour and improve the overall flavour quality (Tian et al. 2019).

GAS CHROMATOGRAPHY OLFACTOMETRY – MILK, DAIRY POWDERS, BUTTER AND YOGHURT

In GC-O, the human nose is used as a detector to evaluate the character and odour intensity of VOCs (Zellner et al. 2008). Thus, it is possible to discern key aromatic compounds in dairy products by GC-O, but impossible to completely understand the whole aroma profile using GC-O, partly because other factors influence aroma perception, but also because aromas often consist of a combination of two or more VOC. As previously stated, VOCs are challenging to extract, separate, identify and quantify as they can interact synergistically or additively to produce an overall odour (Brattoli et al. 2013). Even though GC-O has existed for decades, it remains a relatively obscure research technique especially for milk, dairy powders, butter or yoghurt (~10 publications to date). Overall, the limited use of olfactory analysis for these products is difficult to fathom as even though it is not a complete solution in relation to fully understanding the relationship between VOC and aroma perception, it does provide a very good insight into the aroma characteristics of a product. A likely aspect for its limited use is that it requires highly trained assessors and is quite time consuming (Zellner et al. 2008). However, the potential benefits easily outweigh any disadvantages. GC-O and chemical sensor technologies such as electronic nose and tongue (e-nose and e-tongue), combined with multivariate

data processing methods, are promising relatively novel approaches for rapid analysis of food (Wardencki et al. 2013). Merging both GC-O and GC–MS as an integrated instrument is particularly useful for the identification of aroma-active VOC.

To date, HS-SPME, SAFE and dynamic headspace sampling (DHS) are the most commonly used as pre-treatment methods for GCO analysis (Song and Liu 2018). VOC are typically present from trace amounts to even a few mg kg^{-1} (such as fatty acids in cheese) with odour thresholds varying from ppt to many ppm. In strong smelling dairy products, it may not be necessary to concentrate the VOC profile for GC-O, but for products such as fresh milk it is necessary, thus the choice of extraction method can be dependent upon the sample type. However, care must be taken in GC-O to avoid losing thermal labile VOC or specifically enhancing or creating VOC during the extraction processes, otherwise spurious information may be generated.

Table 3 highlights the odour active VOCs associated with milk, milk powder, butter and yoghurt products. A recent study employed HS-SPME-GC-O (8 mL milk sample with 2 g of sodium chloride at 50°C for 40 min extraction with 75 µm DVB/CAR/PDMS), to compare the volatile profiles of raw and pasteurized milk and pulsed electric field (PEF) treated milk (Zhang et al. 2011). PEF is a nonthermal processing technology that can be applied to liquid milk to inactivate both spoilage and pathogenic microorganisms but also maintains the original nutrients of milk (Amiali and Smith 2007). In the study by Zhang et al. (2011), a total of 19 active VOC were detected with aldehydes making major contributions to a 'fruity, green, cream' note in both pasteurized and PEF-treated milk. 2 (5H)-Furanone was only detected in PEF-treated milk and described as 'caramel' odour. Although concentrations of aldehydes and methyl ketones differed between pasteurized and PEF-treated milk, it appeared not to impact their aroma activities. Colahan-Sederstrom and Peterson (2005) determined if epicatechin addition to raw milk would inhibit the thermal generation of Maillard-type aroma compounds in UHT-processed fluid milk. A total of 32 aroma-active VOC were identified in UHT milk using SAFE-GC-O and GC-MS (1 kg milk sample was extracted with 875 mL diethyl ether for 1 h at 40°C). Methional, furfural, 2-isopropyl-3-methoxypyrazine, 2-acetyl-1-pyrroline and 2-acetyl-2-thiazoline (Maillard-type aroma compounds) showed the largest changes in FD post heat treatment and contributed to the 'cooked' and 'bitterness' flavour of UHT milk. This study demonstrated that epicatechin had the greatest inhibitory effect on the Maillard-derived compounds. In another study, a direct solvent extraction and high-vacuum distillation extraction method was developed for detection of chemical and sensory profiles of stored nonfat dry milk by GC-O (Karagül-Yüceer et al. 2002). Fifty six aroma active VOCs were detected, and a variety of aldehydes, ketones, alcohols and free fatty acids were found to be responsible for the development of undesirable flavours. These authors stated that p-cresol, 3-methylindole (skatole) and some unknown compounds with 'cowy', 'fecal' or 'animal-like' odours appear to contribute to undesirable flavour in milk. Methional and o-aminoacetophenone had high odour intensities in these nonfat dry milks and had characteristics 'boiled potato' and 'animal' odours, respectively. Free fatty acids, including butanoic and hexanoic (cheesy notes) and octanoic, nonanoic, decanoic and dodecanoic acids (waxy note), were also found to contribute to the aroma of milk. Sun et al. (2021) investigated key aroma-active compounds in butter by SAFE-GC-O and GC-MS (40 g butter distilled with 200 mL dichloromethane at room temperature for 30 min). Fifty-three odorants were identified. 2-Furfurylthiol, 2-acetylthiazole, anethole, (E)-2-decenal and 1,8-cineole were the key odorants for the overall aroma of butter and contributed the 'beef', 'boiled beef', 'anise', 'tallow', 'mint, herb' aromas, respectively. As previously mentioned, Lozano et al. (2007) investigated the major aroma components of sweet cream butter. These authors identified 32 and 27 aroma-active VOC were identified by SAFE-GC-O and DHS-GC-O, respectively. VOCs such as lactones were easily recovered by SAFE but poorly by DHS. Butanoic acid, 1-octen-3-one, 2,3-butandione, 2-acetyl-1-pyrroline, dimethyl trisulphide, δ-octalactone and δ-decalatone were the main contributors to fresh butter aroma. Peterson and Reineccius (2003) determined key odourants in heated sweet cream butter aroma by using static headspace analysis (5.6 g butter solution placed in P&T vessel for 45 min extraction at 38°C by Tenax TA trap) by GC-O. These authors identified 19 odour-active VOC in the HS of heated butter. Methanethiol, methional, 3-methylbutanoic acid, 2-heptanone and furaneol were the key odour-active VOC in heated butter in comparison to fresh butter and contributed to the 'pungent', 'cooked potato', 'cheesy', 'blue cheese' and 'sweet caramel' aroma, respectively. Liu et al. (2022) evaluated the odour-active VOC of yoghurt using DHS, SPME, SAFE and SBSE/GC-O and by GC-MS. A total of 31 odour-active VOC were perceived by four extraction methods with DHS providing the most VOCs. 2,3-Butanedione, hexanoic acid, acetophenone, 2,3-pentanedione, acetic acid, octanoic acid, 3-methyl-2-buten-1-ol, butyl acrylate, 2-heptanone, ethyl 2-methylbutyrate and ethyl butyrate were identified as the key odour-active components of yoghurt by DHDA (dynamic headspace dilution analysis). Aroma extraction dilution analysis and OAV identified 'green apple-like', 'sweat-like', 'sweet-like', 'fruit-like', 'butter-like', 'vinegar-like', 'red bean-like', 'green-like' and 'cream-like' flavour properties in these yoghurts. 2,3-Butanedione was found to be the most important odour-active VOC with the highest FD value in yoghurt, contributing 'buttery' odour.

Table 3 Odour active volatile compounds identified in milk, milk powder, yogurt and butter by GCO-MS

Compounds	Odour description	References			
		Milk	Milk Powder	Yogurt	Butter
Alcohols					
Methanethiol	Pungent, sulphury				f
Ethanol	Floral, medicine				g
3-Methyl-1-butanol	Floral-fresh, cheesy, rubber, painty	c			g
2-Heptanol	Fatty-oily				g
2,3-Butanediol	Creamy			e	g
1,3-Butanediol	Musty-wet				g
1-Octen-3-ol	Mushroom, earthy	a, b			h
1-Heptanol	Mushroom				h
Furfuryl alcohol	Caramel				h
α-Terpineol	Green				h
2-Butanol	Sweet almond-like			e	
1-Butanol	Balsam-like, burnt, sweet	c		e	
1-Pentanol	Sweet			e	
1-Hexanol	Greasy			e	
2-Ethyl hexanol	Citrus			e	
2-Phenyl ethanol	Rose		j		
Aldehydes					
2-Methyl propanal	Dark chocolate				i
2-Methyl butanal	Dark chocolate				i
3-Methyl butanal	Dark chocolate, sweet, fruity, fatty		j	e	i
Hexanal	Green, grass, tallow, fruity, floral	b, c, d	j	e	h, i
(Z)-4-Heptenal	Rancid, crabby, biscuit-like	b	j		i
Nonanal	Mushroom, waxy, fatty, floral, green, rosy, sweet, floral	d		e	h, i
Decenal	Green, fatty, floral			e	h, i
(E)-2-Nonenal	Hay, green, fatty, cucumber, oxidized	a, b	j		f, i
(E,Z)-2,6-Nonadienal	Cucumber, cardboard	a, b	j		i
Acetaldehyde	Green, pungent, apple like			e	f
Benzaldehyde	Almond-nutty			e	g
Heptanal	Fat, citrus, cheesy, caramel, fruity	c, d			h
(E)-2-Hexenal	Apple, green				h
Octanal	Fat, soap, orange, fragrant, citrus	c, d		e	h
(E)-2-Heptenal	Fat, fruity	c			h
(E,E)-2,4-Hexadienal	Fat, green	c			h
(E)-2-Octenal	Green, fatty	b, c	j		h
(E,E)-2,4-Heptadienal	Fat, green	c			h
(E)-2-Decenal	Tallow				h
(E)-2-Undecenal	Fat, metallic	b			h
(E,E)-2,4-Decadienal	Fat, soapy, hay, fried	b	j		h
(E,E)-2,4-Nonadienal	Cardboard	a	j		
Butanal	Cocoa-like			e	
Pentanal	Fermented like, fruity, floral	d		e	
Methional	Cooked potato	a, b			f
Phenylacetaldehyde	Rose		j		
Carbonyl compounds					
Ethenylbenzene (styrene)	Styrene, plastic, overripe fruit, clean		e	g	i
p-Cresol	Cowy, barny	b	j		
Toluene	Painty	c			
Benzothiazole	Rubber		j		

(continued)

Table 3 (Continued).

Compounds	Odour description	References			
		Milk	Milk Powder	Yogurt	Butter
Ketones					
2,3-Butanedione (diacetyl)	Buttery, cream, cheese	b, d	j	e	f, i
1-Hexen-3-one	Plastic, veggie, rubbery	b	j		f, i
2-Heptanone	fatty, blue cheese, cheesy-nutty, sweet, fruity, milky, plastic	d		e	f, g, i
Acetoin	Buttery-creamy, mild creamy			e	g
2-Nonanone	Milky, sweet, herb-like			e	g
Acetone	Fruity			e	
2-Butanone	Fruity, buttery, cheese	d		e	
2-Pentanone	Wine-like, malty, fruity	d		e	
3-Hexanone	Rum-like			e	
2,3-Pentanedione	Sweet			e	
3-Heptanone	Green			e	
2-Undecanone	Fruity			e	
Acetophenone	Sweet-almond			e	
(Z)-1,5-Octadien-3-one	Metallic		j		
Lactones					
δ-Octalactone	Herbaceous, peach	a			f, i
γ-Nonalactone	Peachy				i
δ-Decalactone	Waxy, sweet, coconut, hot milk	a, b, d	j		h
δ-Undecalactone	Coconut, butter, green, cilantro	b	j		i
δ-Dodecalactone	Coconut, cheesy, sweet, fruity	d		e	i
γ-Dodecalactone	Sweet, green	b	j		
δ -Decanolactone	Peach				f
δ -Hexanolactone	Creamy, chocolate, sweet aromatic				f
γ-Decalactone	Sweet, perfume	a			
γ-Butyrolactone	Creamy			e	
Sulphur compounds					
Dimethyl sulphide	Sulphur, sweet			e	i
Dimethyl trisulphide	Cabbage, garlic, sulphury	b	j		f, i
Ethyl disulphide	Gasoline		j		
Hydrogen sulphide	Boiled egg, eggy				f
Dimethyl disulphide	Vegetable-like			e	
Esters					
Ethyl acetate	Fruity, mild, sweet, solvent	d		e	i
Ethyl butanoate	Fruity, berry, fruity-rose, herb-like, sweet			e	g, i
Ethyl lactate	Creamy-whey				g
Ethyl octanoate	Floral				g
Ethyl decanoate	Fruity-pear				g
Ethyl propionate	grape-like			e	
Methyl butanoate	pineapple-like			e	
Butyl propionate	Rosy, sweet			e	
Ethyl hexanoate	pineapple-like			e	
Furans					
Furaneol	Sweet caramel-like	a			f, h
Furfural	Almond, roasted, nutty	a, c			h
2-Furanmethanol	toast bread-like, vitamin, rubber, caramel	b	j	e	h
2-Acetylfuran	Plastic, nutty	c			
Acids					
Acetic acid	Vinegar, sour	c		e	h, i
Butanoic acid	Fecal, cheesy, rancid, ripened cheese, buttery, sour, creamy	a,d		e	f, g, i

(continued)

Table 3 (Continued).

Compounds	Odour description	References			
		Milk	Milk Powder	Yogurt	Butter
3-Methylbutanoic acid	Sweaty, cheesy, whey-flowery, sour	a,b			f, g, i
Hexanoic acid	Doughy, sweaty,cheesy acrid,rancid, buttery-soapy,sour	a, b, d		e	f, g, i
Propanoic acid	Fatty, cheesy			e	g
2-Methylpropanoic acid	Rancid buttery			e	g
Pentanoic acid	Cheesy-musty, swiss cheese	b			g
Octanoic acid	Cheesy, goat, foul	a		e	g
Nonanoic acid	Green, fat, sour	a		e	g
Decanoic acid	Soapy, rot-like	a		e	
Heptanoic acid	Sour	a		e	
Dodecanoic acid	Waxy	b			
Tetradecanoic acid	Coconut-like			e	
Terpenes					
α-Pinene	Mint, pine oil, dry, woody	c			i
D-Limonene	Citrusy, Lemon, orange			e	g, h
β-Myrcene	Balsamic, rosin				h
3-Methylthiophene	Plastic		j		
β-ionone	Hay		j		
Other					
2-Acetyl-1-pyrroline	Popcorn, roasted	a, b	j		i
2-Acetyl-2-thiazoline	Cooked, popcorn, roasted	a, b	j		i
Skatole	Skatole, fecal, mothball		j		i
Acetylpyrazine	Roast				h

Abbreviation: DE, Dynamic extraction; SAFE, Solvent-assisted flavour evaporation; SBSE, Stir bar sorptive extraction; SPME, Solid-phase microextraction.

The data adapted from (a) Colahan-Sederstrom and Peterson (2005); (b) Karagül-Yüceer et al. (2002); (c) Yeh et al. (2017); (d) Zhang et al. (2011); (e) Liu et al. (2022); (f) Peterson and Reineccius (2003); (g) Sarhir et al. (2021); (h) Sun et al. (2021); (i) Lozano et al. (2007); (j) Karagül-Yüceer et al. (2002).

CONCLUSIONS

Significant advances in our understanding of the key aroma active VOC that impact the sensory perception of milk, dairy powders, butter and yoghurt have been outlined. In terms of sensory approaches, both traditional and novel sensory techniques have been discussed, but also cultural factors influencing choice. This review has reiterated the importance of product familiarity and how critical this is in relation to cross-cultural sensory acceptance, especially in countries where dairy products have little tradition. Much more sensory research of dairy products is required to better understand cultural factors influencing choice/acceptability and to ensure that all participants in such studies unambiguously comprehend what is required of them, most notably avoiding words that could be misinterpreted or have dual meanings from a cultural or language perspective.

The importance of VOCs impacting the aroma of milk, dairy powders, butter and yoghurt are discussed. More than 300 different VOC, belonging to 10 or more chemical classes, have been identified in milk, dairy powder, butter and yoghurt to date. This review has focussed on GS-MS, which is by far the most widely used approach to identify these compounds, but with particular emphasis on the different VOC extraction techniques used, highlighting their advantages and/or shortcomings. Aldehydes, alcohols, lactones, ketones, acids, terpenes, carbonyl compounds and furans are by far the most prominent and potent VOC that appear to influence the sensory appeal of these products. A single or multiple source can be responsible for the generation of VOC. Some are directly or indirectly dietary related, in that they can be transferred from the diet by ingestion or inhalation, or indirectly created during rumen metabolism and end up in the milk. Others are created during processing for example by heat treatments or by the inclusion of ingredients/processing aids or in final product formulation. Thus diet and milk quality plus product processing and formulation have a major role in VOC formation in the final product, which subsequently impacts on aroma generation and thus sensory perception.

As product variation within these dairy products is relatively large, and as a wide range of odour active VOC are

typically present, it is difficult to absolutely identify individual VOC responsible for the overall aromatic characteristics of these dairy products. However, some informed conclusions can be made based on research to date. This review has highlighted the benefits of GC-O, especially in combination with complementary techniques such as GC-MS and also highlights that much more research is required combining sensory and analytical techniques in order to better understand flavour development in these products in order to improve quality but also adjust in-farm and process inputs to create products more suited to particular markets.

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AUTHOR CONTRIBUTIONS

Zeng Cheng: Conceptualization; visualization; writing – original draft; writing – review and editing. **maurice O'Sullivan:** Supervision; writing – review and editing. **Song Miao:** Funding acquisition; writing – review and editing. **Joseph P. Kerry:** Supervision; writing – review and editing. **Kieran Noel Kilcawley:** Conceptualization; funding acquisition; resources; supervision; writing – review and editing.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

Data sharing not applicable – no new data generated.

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