Undesirable Sulphur and Carbonyl Flavour Compounds in UHT Milk: A Review

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ABSTRACT

UHT processing, leads to the formation of "cooked" and "flat" flavours in milk. These undesirable notes occur due to the volatile formation of a variety of sulphur containing compounds, methyl ketones and aliphatic aldehydes derived from the constituents of the milks matrix during thermal processing and storage. The "cooked" flavour of UHT milk is associated with the presence of a variety of sulphur containing compounds while the "stale" flavour is characterised by the dissipation of these sulphur volatiles and an increase of the formation and presence of both methyl ketones and aliphatic aldehydes over time. The extent that the individual volatiles contribute to the overall flavour of UHT milk is not clear. The proposed formation of these volatiles; methods to control the intensity of "cooked" and "stale" flavours associated with UHT milk and extraction techniques for the isolation of these volatiles from milk have been reviewed.

Keywords: UHT milk, cooked flavour, stale flavour

INTRODUCTION

Ultra High Temperature (UHT) milk is a shelf-stable dairy product which is produced by holding milk at a high temperature (138-145°C) for a short time interval (1.5-9.0 seconds) (Valero et al., 2001) and packaged under aseptic conditions. This treatment provides an advantageous shelf-life of 6-9 months at room temperature when compared to approximately 14 day's refrigerated shelf-life of pasteurised milk (Perkins et al., 2005). However, according to Clare et al. (2005) UHT milk has poor consumer acceptability. This can be attributed to the chemical formation of "cooked" and "flat" flavours (Vazquez-Landaverda et al., 2005; Vazquez-Landaverde et al., 2006) which are derived from the constituents of the milks matrix during processing and storage. These chemical changes lead to an increase in the concentration of various flavour compounds such as sulphur containing compounds, methyl ketones and aliphatic aldehydes which contribute to the overall flavour of the milk (Jeon et al., 1978; Shibamoto et al., 1980; Contarini et al., 1997; Vazquez-Landaverda *et al*., 2005 and Vazquez-Landaverde et al., 2006). In addition to the "cooked" and "stale" character, heated milk may also be perceived as having a "heated" or "rich" flavour (Shipe et al., 1978). This characteristic flavour is believed to be imparted via products of the Maillard reaction during thermal processing (Rerkrai et al., 1987). These Maillard compounds include various furans, diketones, cyclic ketones, pyrazines and other sulphur and nitrogen containing compounds (Shibamoto et al., 1980). Initially UHT milk is described as having a "cooked" flavour which is attributed to the increase in the formation of sulphur containing compounds in the milk due to thermal denaturation of milk serum proteins during processing (Patrick and Swaisgood, 1976 and Vazguez-Landaverde et al., 2006). This "cooked" flavour however dissipates after several weeks and is replaced by an off flavour note characterised as being "stale" (Valero et al., 2001 and Vazquez-Landaverde et al., 2005). This is due to the formation of methyl ketones via the thermal decarboxylation of β-keto acids as well as the initiation of lipid oxidation in the milk leading to an increase in the formation of aliphatic aldehydes during storage (Shibamoto et al., 1980 and Moio et al., 1994). To consumers, flavour is one of the most significant quality attributes owing to the overall acceptability of a food product (Guichard, 2002) and due to the formation of these compounds in milk during processing and storage some consumers find the flavour of UHT milk to be undesirable (Clare et al., 2005).

Thus if the concentration of these "cooked", "stale" and "heated" off flavour producing compounds in UHT milk can be significantly reduced the overall consumer acceptance of the product may improve. The formation of off flavour volatiles in UHT milk will be reviewed. Furthermore methods to reduce the concentration of these volatiles; the interactions of these volatiles with milk packaging materials and analytical methods used to study these volatiles in milk are discussed.

FLAVOUR COMPOUNDS IN UHT MILK

According to Jaddou et al. (1978); Jeon et al. (1978); Contarini and Povolo (2002) and Vazquez-Landaverda et al. (2005) the flavour of milk is liable to change when milk is subjected to a thermal treatment and storage. Heat induced flavours in milk can be classified into four distinct groups: cooked or sulphurous; heated or rich, caramelised and scorched (Shipe et al., 1978). According to Clare et al. (2005) UHT milk has poor consumer acceptability, when compared to pasteurized milk, due to the formation of "cooked" and "flat" flavours. Several compounds which give milk a "heated" or "rich" character also appear to contribute to the overall "stale" (Scanlan et al., 1968; Jeon et al., 1978; Shibamoto et al., 1980; Rerkrai et al., 1987; Vazquez-Landaverde et al., 2005) and "cooked" (Colahan-Sederstrom and Peterson, 2005) flavour of UHT milk. Furthermore, Ferretti and Flanagan (1972) proposed that the "stale" flavour associated with dairy products is possibly due to both lipid oxidation and the Maillard reaction. The initial "cooked" flavour of UHT milk dissipates several days after UHT milk production (Patrick and Swaisgood, 1976). The milk is then rather described as "stale" (Thomas et al., 1975). These flavours occur due to the increase in the formation of various carbonyl and sulphur containing compounds in UHT milk (Jeon et al., 1978; Shibamoto et al., 1980; Contarini et al., 1997; Vazquez-Landaverda et al., 2005 and Vazquez-Landaverde et al., 2006). The concentrations of these flavour compounds are lower in raw and pasteurized milk when compared to the flavour profile of UHT milk (Table 1). The mentioned flavour compounds in UHT milk are derived via thermal denaturation of milk proteins; lipid oxidation or non enzymic reactions of the constituents of the milk's matrix i.e. milk proteins, carbohydrates, lipids and other milk constituents (Calvo and de la Hoz, 1992).

Table 1. The Concentration (μg/kg) and Odour Thresholds of Some Flavour Compounds in Raw, Pasteurized and UHT Milk (3% milk fat). Adapted from Rychlik et al. (1998), Vazquez-Landaverde et al. (2005) and Vazguez-Landaverde et al. (2006)

Compound	Raw milk	Pasteurised milk	UHT milk	Odour Threshold (μg/kg)[a]
Ketones				
Diacetyl	0.48	1.71	7.39	5
2-pentanone	0.28	0.14	9.53	n.d.
2-hexanone	0.37	0.17	1.18	n.d.
2-heptanone	0.95	0.55	34.46	5
2-octanone	3.82	2.15	4.51	n.d.
2-nonanone	0.24	0.33	52.64	5
2-decanone	n.d.	n.d.	1.33	n.d.
2-undecanone	4.58	0.7	9.7	n.d.
Aldehydes				
2-methylpropanal	0.40	0.48	2.52	0.7
3-methylbutanal	n.d.	0.17	1.14	0.04
2-methylbutanal	0.90	0.11	0.91	0.9
Hexanal	2.68	0.82	12.97	4.5
2-furaldehyde	0.20	0.13	0.38	3000
Heptanal	0.20	0.08	1.68	3
Octanal	0.52	0.09	0.95	0.7
Nonanal	1.36	0.28	3.92	1
Decanal	2.72	1.26	6.68	0.1
Sulphur compounds				
hydrogen sulphide	1.21	0.5	12.0	10
Methanethiol	4.80	0.4	23.9	5
carbon disulphide	0.0221	0.0313	0.0589	n.d.

Compound	Raw milk	Pasteurised milk	UHT milk	Odour Threshold (μg/kg)[a]
dimethyl sulphide	8.16	14.2	21.41	2
dimethyl disulphide	0.0334	0.0101	0.0328	30
dimethyl trisulpfide	0.0367	0.0157	0.0473	0.008
dimethyl sulfoxide	1260	820	1460	n.d.
dimethyl sulfone	2640	4720	1260	n.d.

[a] odour thresholds determined in water. n.d. = not determined

Sulphur containing compounds

The initial "cooked" flavour is the first of two problematic flavours occurring in UHT milk (Mehta, 1980). As raw milk is heated during UHT processing, serum proteins denature leading to the formation of free "reactive" sulphydryl groups which correlate to the liberation of volatile sulphur compounds and the "cooked" flavour (Blankenagel and Humbert, 1963; Patrick and Swaisgood, 1976). However according to Jaddou et al. (1978) the "cooked/cabbagy" flavour of UHT milk may not only be due to the collective liberation of sulphur containing compounds (hydrogen sulphide, carbonyl sulphide, methanethiol, dimethyl sulphide and carbon disulphide) but perhaps a result of these compounds interacting with unidentified compounds from the carbonyl fraction of milk.

Initially hydrogen sulphide was considered the major sulphur containing volatile contributing to the "cooked" flavour of heated milk (Thomas et al., 1976). However more recently, Vazquez-Landaverde et al. (2006) suggested that hydrogen sulphide does not contribute as extensively to the aroma of UHT milk as previously indicated. Furthermore Vazquez-Landaverde et al. (2005) identified that dimethyl sulphide is an important aroma contributor and Vazquez-Landaverde et al. (2006) showed that methanethiol is possibly the most potent sulphur containing aroma compound in UHT milk. Both these compounds are present in UHT milk at concentrations well above their odour thresholds (Table 1) (Rychlik et al., 1998). Hydrogen sulphide is formed due to the thermal denaturation of β-lactoglobulin in particular as well as other whey proteins and from denatured protein containing material, associated with the milk fat globule membranes

(Townley and Gould, 1943; Hutton and Patton, 1952; Badings and van der Pol, 1973). These protein molecules contain the sulphur containing amino acids cysteine and methionine (Aboshama and Hansen, 1977). The now exposed sulphydryl groups of the denatured polypeptides may be oxidised leading to the release of hydrogen sulphide (Townly and Gould, 1943; Hutton and Patton, 1952). Alternatively, Strecker degradation of cysteine residues with a diketone may take place leading to the formation of hydrogen sulphide (Schutte and Teranishi 1974). Although the exact formation of dimethyl sulphide is unclear there are two proposed reaction mechanisms. Keenan and Lindsay (1968) showed that dimethyl sulphide and homoserine concentrations increased when s-methylmethionine sulphonium salts, occurring in milk from various plant food sources, decreased due to thermal desiccation during processing. However Ballance (1961) believes that dimethyl sulphide is formed from the sulphur containing amino acid methionine. During the heating of milk, methional is formed due to the Strecker degradation of methionine and is further converted to methanethiol (Schutte and Koenders 1972). Methanethiol is then oxidised to form dimethyl disulphide (Ballance, 1961) which can be further converted to dimethyl trisulphide and dimethyl sulphide (Fig. 1). According to Thomas et al. (1975) the loss of the "cooked" flavour of heated milk during storage is due to the oxidation of reactive sulphydryl groups with molecular oxygen over time. However dimethyl sulphide is known to increase in concentration during storage (Jaddou et al., 1978) possibly because dimethyl sulphide is a product of the oxidation of methanthiol (Ballance, 1961).

Ketones

The "stale" note is the second of the two problematic flavours of UHT milk (Mehta, 1980). It appears in the milk during storage as the "cooked" flavour begins to decrease (Thomas et al., 1975). A number of different ketones contribute to the overall flavour of UHT milk. Methyl ketones for instance contribute to the perceived "stale" flavour (Contarini and Povolo, 2002) while diketones and cyclic ketones add "heated" flavour notes to the milk (Shibamoto et al., 1980). Methyl ketones are present in raw and pasteurised milk however their total concentration

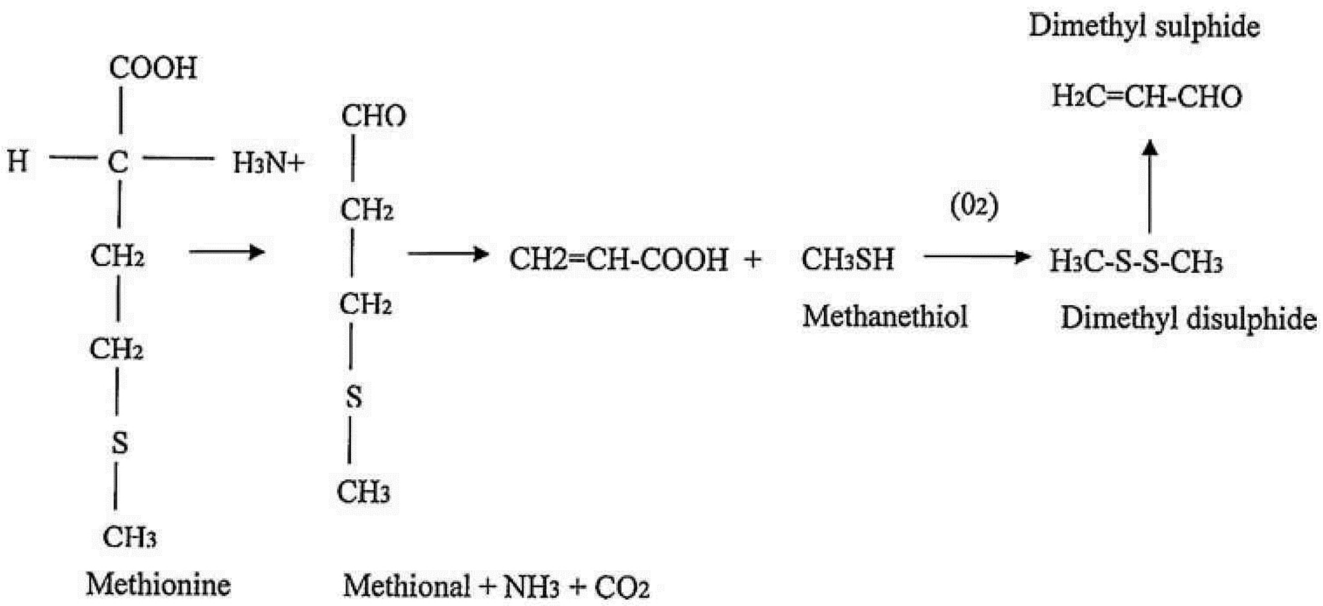

Figure 1 Formation of dimethyl sulphide via Strecker degradation of methionine and the oxidation of methanthiol. Adapted from Ballance (1961) and Schutte and Koenders (1972).

is considerably greater in UHT milk (Vazquez-Landaverde et al., 2005). The concentration of

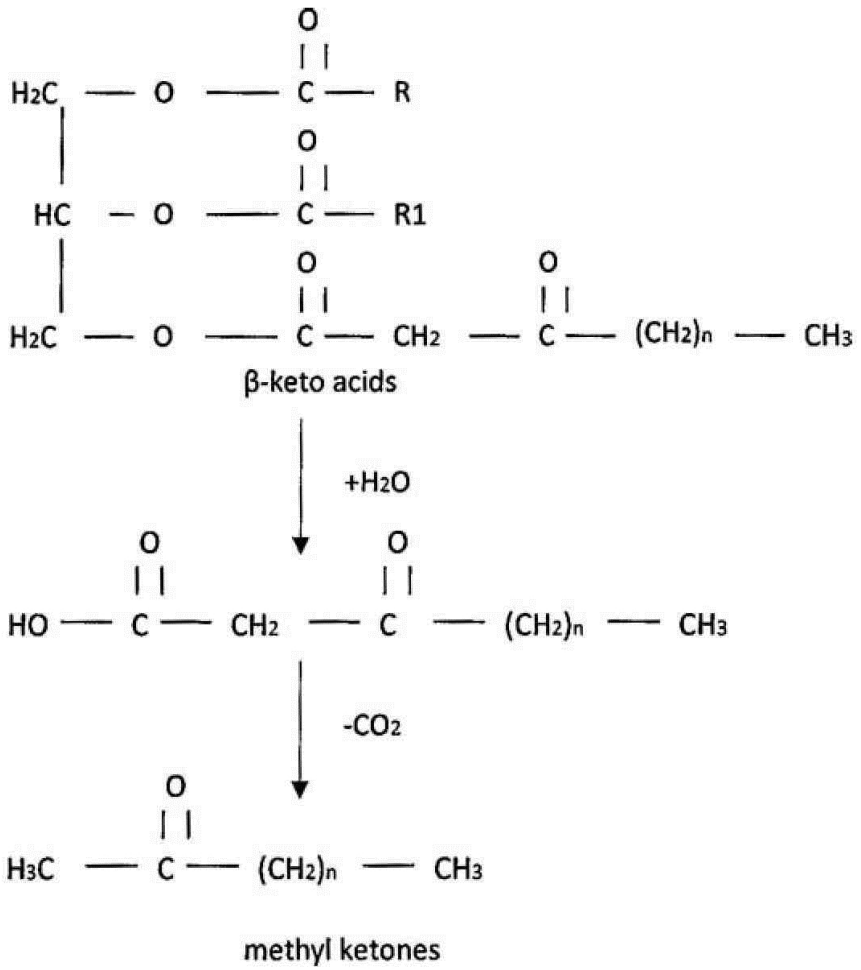

Figure 2 Thermal decarboxilation of β-keto acids to form methyl ketones. Adapted from Shibamoto et al. (1980).

methyl ketones increase during storage (Valero et al., 2001) and the rate of their formation is dependent on the milks storage temperature (Jeon et al., 1978). Contarini et al. (1997) and Vazquez-Landaverde et al. (2005) identified that the overall concentration of methyl ketones is proportional to the extent of the thermal processing treatment and the development of a "stale" flavour in UHT milk. Methyl ketones, particularly ones with odd numbered carbon atoms, are produced via the hydrolysis and decarboxylation of β-ketoalkanoic acid esters in milk fat during heating (Parks et al., 1964) (Fig. 2). Methyl ketone formation requires low activation energy (Schwartz et al., 1966) and so formation continues readily throughout the storage period (Kochhar, 1996) of the milk.

Although the overall concentration of methyl ketones are relatively high in UHT milk, they occur at levels far below their odour threshold values and contribute little to the overall "stale" flavour when compared with aliphatic aldehydes (Table 1) (Jeon et al., 1978). However, Contarini and Povolo (2002) showed that an increase in the perceived "stale" flavour of UHT milk correlated with an increase in the concentration of thermally derived methyl ketones in the product. It has been suggested by Langler and Day (1964) that a mixture of methyl ketones may act synergistically together to contribute to a perceivable flavour when the compounds are all present below their threshold values. Moio et al. (1994) suggested that 2-heptanone and 2-nonanone in particular are the two most potent aroma compounds in UHT milk. Recently, based on odour active values (OAV) Vazquez-Landaverde et al. (2005) identified 2-heptanone and 2-nonanone as important odourants in the overall "stale" aroma of UHT milk. Using an olfactory evaluation of captured UHT milk volatiles Naudé et al. (2009) identified that 2-heptanone had a "soapy" aroma where as 2-nonanone was perceived as more "fruity". However the synergistic effect of 2-nonanone and 2-heptanone produced a pungent cheese, sour milk-like aroma in UHT milk.

Aldehydes

According to Vazquez-Landaverde et al. (2005) aldehydes may strongly contribute to the flavour of UHT milk albeit that they occur at very low concentrations. The rate at which aldehydes form in milk are influenced by two factors, namely 1) dissolved oxygen content and 2) temperature during storage (Jeon et al., 1978). Aliphatic aldehydes are produced in UHT milk, during storage, by the oxidation of casein bound unsaturated fatty acids (Ramshaw and Dunstone, 1969) as well as via the degradation of various amino acids in the milk's matrix. For example 3-Methylbutanal or 2-methylbutanal are synthesised via the degradation of the amino acid leucine in the Maillard reaction (Morgan et al., 1957) which occurs during the heating of milk. Nonanal however, is formed as a secondary oxidation product of a C10 hydroxide derived from the autoxidation of oleic acid (Day and Lillard, 1960) (Fig. 3).

Aliphatic aldehydes are mostly present at concentrations near to or above their odour threshold values in UHT milk (Jeon et al., 1978; Rerkrai et al., 1987; Rychlik et al., 1998 and Vazquez-

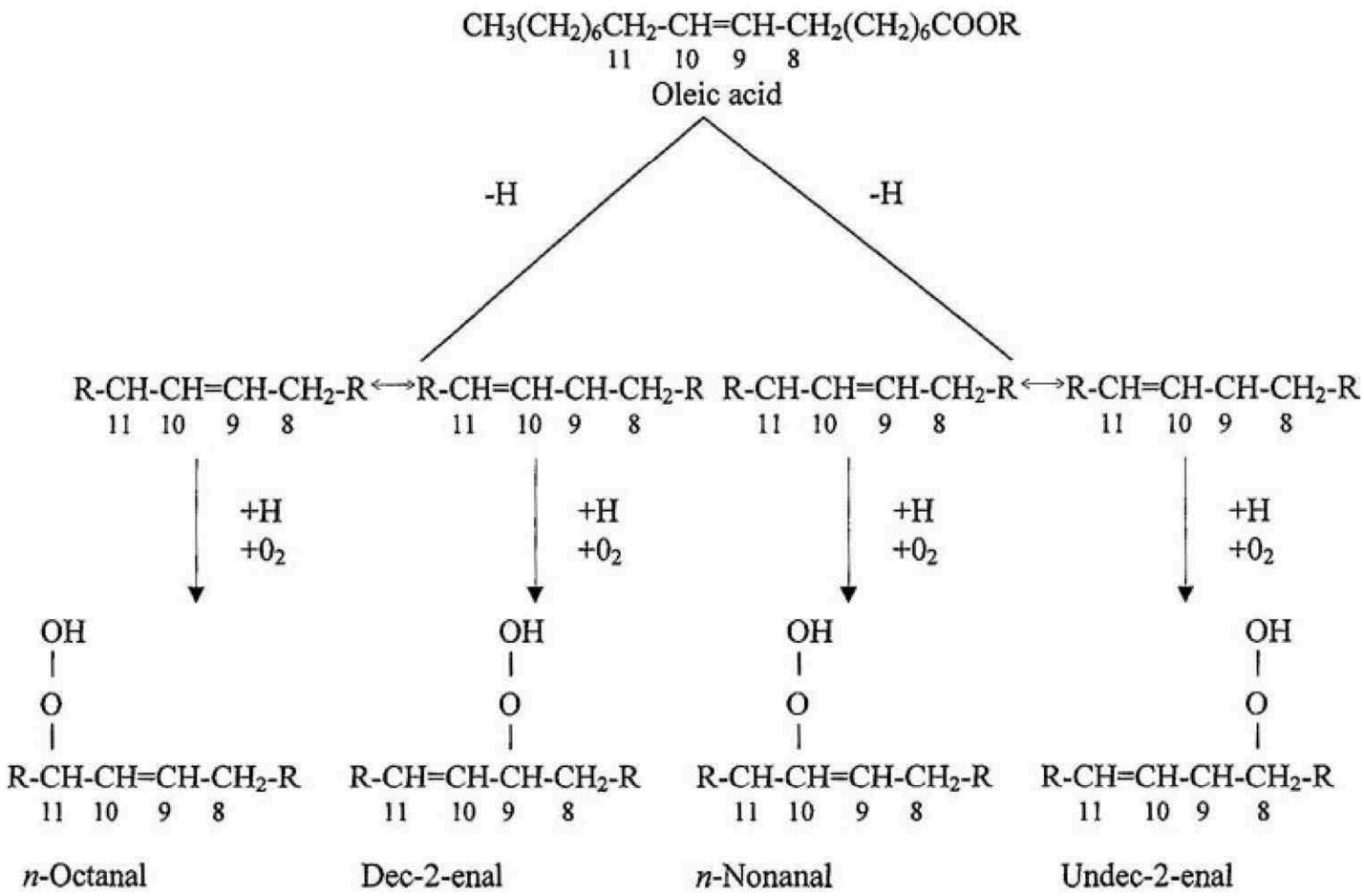

Figure 3 Formation of octanal, decanal, nonanal and undecanal via the oxidative degradation of oleic acid. Adapted from Day and Lillard (1960).

Landaverde et al., 2005). Thus, Jeon et al. (1978) concluded that even though the concentrations of methyl ketones are higher, aliphatic aldehydes in particular contribute more extensively to the overall "stale" flavour of UHT milk. Based on OAV's octanal, nonanal, decanal, 2-methylbutanal, 2-methylpropanal and methylpropanal are major contributors to the "stale" flavour of UHT milk (Vazquez-Landaverde et al., 2005). Furthermore, Day et al. (1963) suggested that a mixture of aliphatic aldehydes could act in a synergistic manner and give rise to an oxidised flavour in milk fat samples when all the aldehydes were present below their threshold values. This indicates that aldehydes may considerably contribute to the "stale" flavour of UHT milk all be it that their concentration in the milk is relatively low (Vazquez-Landaverde et al. 2005).

Compounds associated with the Maillard reaction

Nursten (1981) proposed that volatile compounds derived from the nonenzymic pathways of the Maillard reaction can be classified into three groups (Table 2). During milk processing, lactose and its derivatives, glucose and galactose, may either thermally degrade or interact with amino acids and other nitrogenous compounds to form a variety of flavour compounds via the Maillard reaction (Calvo and de la Hoz, 1992). Many of these compounds are believed to contribute to the "heated" or "rich" flavour of heated milks (Shipe et al., 1978 and Rerkrai et al., 1987). Maillard derived compounds such as benzaldehyde (Joen et al., 1978), acrolein (Mehta and Bassette, 1978), maltol, diacetyl (Scanlan et al., 1968), acetaldehyde, isobutanal (Rerkari et al., 1987), 1-butanol (Jaddou et al., 1978) 2-methylbutanal and 3-methylbutanal (Vazquez-Landaverde et al., 2005) have been identified in UHT milk. Diacetyl in particular, has been suggested to contribute excessively towards the "heated" flavour of UHT milk (Scanlan et al., 1968). According to Jeon et al. (1978) diacetyl occurs above its odour threshold in UHT milk and thus contributes to the flavour of the final product. The concentration of diacetyl in milk increases proportionally to the increasing severity of the applied heat treatment (Scanlan et al., 1968).

The magnitude that all Maillard reaction compounds contribute to the overall "heated" flavour of the product is still not fully understood. However, it appears that several compounds contributing to the "heated" or "rich" character may also contribute to the overall "stale" and

"cooked" flavour of UHT (Scanlan et al., 1968; Jeon et al., 1978; Shibamoto et al., 1980; Rerkrai et al., 1987; Colahan-Sederstrom and Peterson, 2005; Vazquez-Landaverde et al., 2005).

A number of different studies have been carried out in order to limits or decrease off "cooked" and "stale" flavour production in milk. According to Boyd and Gould (1957) the addition of copper sulphate, calcium chloride or disodium phosphate to milk before heating, reduces the total concentration of sulphydryl groups (-SH) and hydrogen sulphide in milk. The addition of 0.5ppm copper, prior to and after heating, led to the reduction of hydrogen sulphide where as

Table 2. Classification of Volatile Products Derived During The Maillard Reaction (Nursten, 1981)

Volatile Classification	Volatile Compounds
1- Dehydration and fragmentation products from "simple" sugars	Furans
	Pyrones
	Cyclopentenes
	Carbonyls
	Acids
2- Degradation products from "simple" amino acids	Aldehydes
	Sulphur compounds
3- Volatiles formed by further interactions	Pyrroles
	Pyridines
	Imidazoles
	Pyrazines
	Oxazoles
	Thiazoles

Inhibition of "cooked" and "flat" flavours in milk

2.0ppm led to no hydrogen sulphide being produced when compared to a control. However, Marsili (2000) indicated that when copper was added to milk in excess it led to the formation of an adverse "oxidised" flavour in the milk. Samuelsson and Borgström (1973) used sodium iodate or sodium bromate or potassium iodate or potassium bromate, which are oxidising agents, to reduce the presence of sulphydryl groups and decrease the "cooked" flavour in sterilised milk without increasing the production of an "oxidised" flavour. Ferretti (1973) and Ferretti et al. (1974) showed that the "cooked" flavour of UHT milk can be controlled by the addition of organic thiolsulphonates and organic thiosulphates to the milk before heating. However, apart from two of these compounds adding other undesirable flavours to the milk (Ferretti, 1973) neither are permitted to be used as food additives (Ferretti et al., 1974) and may not be added to milk. The "cooked" flavour of UHT milk may be removed by treating the milk with a native milk enzyme, sulphydryl oxidase. The enzyme initiates the conversion of –SH groups in heated milk to form disulphides but, it cannot be added to milk prior to heating because high temperatures lead to the inactivation of the enzymic activity of sulphydryl oxidase (Swaisgood, 1977). Renner and Berlage-Weining (1983) added L-cysteine to heated milk in order to reduce the concentration of hydrogen sulphide and the overall intensity of the "cooked" flavour in heated milk where cysteine is formed due to the reaction of L-cysteine with hydrogen sulphide.
According to Colahan-Sederstrom and Peterson (2005) sulphur-containing methional showed a significant reduction along with other Maillard reaction-derived compounds when the polyphenol, epicatchin (0.2%), was added to milk prior to UHT processing. Although the addition of epicatchin lead to a decrease in the "cooked" flavour, at the effective level of 0.2% it also imparts a bitter note to milk (Colahan-Sederstrom and Peterson, 2005). Al-Attabi et al. (2009) have recently and extensively similarly summarised a number of the attempted methods used to reduce the "cooked" flavour of heated milk.

Although copper has been used to decrease the "cooked" flavour of UHT milk (Boyd and Gould, 1957; Marsili, 2000) the presence of copper increased the formation of acetal, propanal, n-pentanal and n-hexanal in pasteurised milk because copper ions may initiate lipid oxidation

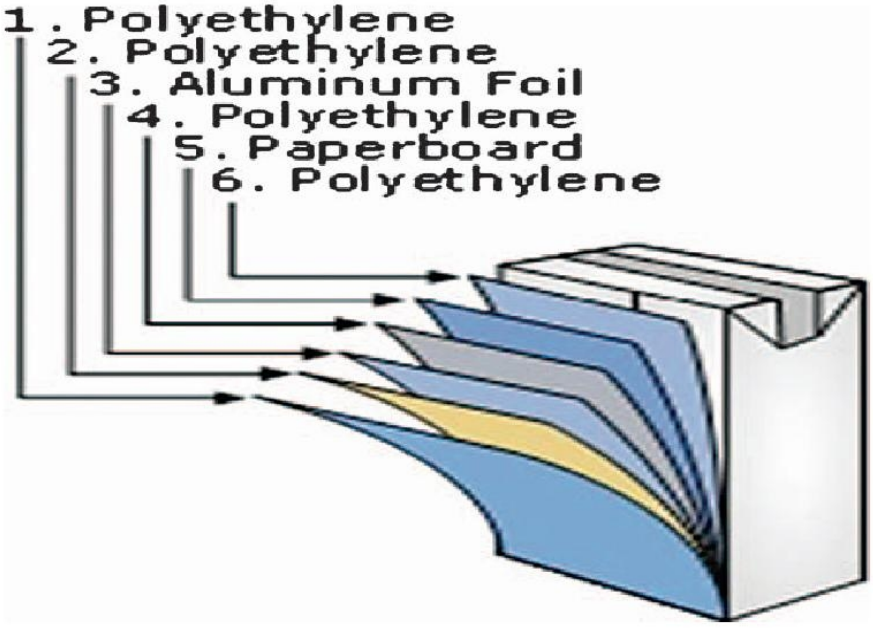

Figure 4 Schematic diagram of the individual layers making up the multilayered Tetra Brick® (Tetra Pak, 2008). (color figure available online.)

leading to the production of "stale" flavours (Jenq et al., 1988). To control the "flat" flavour of UHT milk research has focused on active packaging to selectively adsorb aldehydes and ketones. Brodie III and Visioli (1994) decreased the concentration of hexanal from a "hexanal environment" by exposing the environment to a polyethylene imine and polyolefin polymer which can absorb volatile aldehydes. Suloff et al. (2003) lowered the concentration of acetaldehyde, propionaldehyde, butyraldehyde, valeraldehyde and caproaldehyde from an acidified aqueous model solution by active packaging incorporated with aldehyde-scavenging agent's nylon MXD6, D-sorbitol and α-cyclodextrin. This film showed selective binding for lower molecular weight aldehydes (Suloff et al., 2003). There is no indication that this film can bind methyl ketones, which are key component of the "stale" flavour of UHT milk (Shibamoto et al., 1980 and Contarini and Povolo, 2002) and the analysis was also not carried out in a more complex matrix such as milk. Colahan-Sederstrom and Peterson (2005) added epicatchin (0.2%) to milk prior to UHT processing. Even though epicatchin is a powerful oxidising agent it had a limited affect on the total production of nonanal a compound formed during lipid oxidation (Colahan-Sederstrom and Peterson, 2005). Recently Perkins et al. (2006) incorporated an oxygen scavenging film into a UHT milk package which lowered the amount of dissolved

oxygen in UHT milk, leading to the reduction of 2-hexanone, hexanal, 2-heptanone, 2-nonanone and nonanal by 23-41% after 14 weeks of storage when compared to a control. However based on odour rather than flavour a sensory panel found no significant difference between the treated and untreated samples and the oxygen scavenger used in the trials was not an approved food additive (Perkins et al., 2006).

PACKAGING OF UHT MILK

In the food packaging industry, low density polyethylene (LDPE) is the most used plastic film (Robertson, 2006) because it is a good moisture barrier; it is stable at high processing temperature; it is inert to most food products; it is resistant to chemical degradation (Sajilata et al., 2007) and it is the most cost effective plastic film available on a per unit mass basis (Selke, 2003). UHT milk is aseptically packaged into a polyethylene (LDPE)-lined paper-board pack containing a layer of aluminium foil (Robertson, 2006). This package is known as a Tetra Brick ® (Tetra Pak, 2008). The three materials used in the multilayered brick are allocated into six individual layers, where each layer fulfils a desired function (Fig. 4 and Table 3).

Table 3 The Function of LDPE, Paper-Board and Aluminium Foil Layers in The Multilayered Tetra Brick®. Adapted from Tetra Pak (2008)

Layer	Material	Function
1	LDPE	Interior moisture barrier, preventing the gain of moisture to the milk
2	LDPE	Binding surface for layer 1 and 3
3	Aluminium Foil	Prevents the penetration of UV light and O_2 into the milk (prevents oxidation[1])
4	LDPE	Binding surface for layer 3 and 5
5	Paper-Board	Provides mechanical rigidity to the package
6	LDPE	Exterior moisture barrier, preventing the loss of moisture from the milk

LDPE = low density polyethylene. [1] Lipid and nutrient oxidation

Interactions between flavour compounds and UHT packaging material

Interactions between flavour compounds inherent in milk and the UHT packaging material itself have been studied (van Willige et al., 2000b, Simon et al., 2001 and Czerny and Schieberle, 2007). According to Hansen et al. (1974) after 16 days of storage, flavour sorption of volatiles to the UHT packaging material takes place in milk packaged inside polyethylene and cardboard boxes. Flavour volatiles in UHT milk, including 2-nonanal, may move into and through polyethylene packaging material during storage (Czerny and Schieberle, 2007). Furthermore the scalping of these compounds by LDPE is influenced by the volatiles molecular size as well as the polarity and solubility characteristics of the volatile and the LDPE itself (Nielsen et al., 1992). Arora et al. (1991) showed that the more hydrophobic flavour compounds with longer carbon chains have a greater affinity for non polar LDPE films while Linssen et al. (1991) stated that volatiles in drinking yoghurt with shorter carbon chains have a lower affinity to polyethylene bottles.

According to Arora et al. (1991) LDPE has an affinity for a range of hydrophobic compounds adsorbing aldehydes to a greater degree than methyl ketones in aqueous flavour solutions. LDPE also has an affinity for branched sulphur containing compound (Arora et al., 1991). Most studies on the interaction of flavours with packaging materials are completed in simple aqueous flavour solutions and not with actual food products with complex matrixes.

In model food systems the concentration of flavour compounds adsorbed by linear low density polyethylene (LLDPE) was influenced by the presence of oil or fat, polysaccharides and proteins where these constituents decreased the amount of flavour adsorbed by the packaging material (van Willige et al., 2000a and van Willige et al., 2000b).

No information could be found regarding the interaction of flavour compounds with the paper-board of the brick type aseptic packages. However, packaging material consisting of polyethylene (PE)/paper-board/PE/aluminium/PE retains sulphur containing hydrogen sulphide and methanethiol for longer than packages without the aluminium barrier because the barrier reduces the overall transfer of volatiles in the system (Simon et al., 2001).

EXTRACTION TECHNIQUES FOR THE ISOLATION OF VOLATILES FROM FOOD MATRICES

In general gas chromatography (GC) involves the headspace extraction and analysis of volatile compounds in the gaseous phase above a non-volatile liquid or solid phase (Snow and Slack, 2002 and Wampler, 2002). Due to the fact that flavour or aroma compounds are volatile in nature, techniques used to sample or extract the volatiles from the headspace above milk include static headspace extraction, dynamic headspace (purge-and-trap) extraction, headspace solid-phase microextraction (HS-SPME) and stir bar sorptive extraction (SBSE) (Christensen and Reineccius, 1992; Marsili, 1999; Naudé et al., 2009; Valero et al., 2001 and van Aardt et al., 2009). These extraction techniques are then usually coupled to analytical methods such as GC-MS and GC- flame ionisation detection (FID) to identify and quantify the extracted volatiles (Vazquez-Landaverde et al., 2005 and Naudé et al., 2009). Headspace sampling/extraction techniques allow for solvent free extraction with the extracted volatiles being more representative of the food flavour profile being analysed when compared to solvent extraction techniques (Naudé et al., 2009). This review will further cover the basic principles pertaining to static headspace extraction, dynamic headspace (purge-and-trap) extraction, HS-SPME and SBSE.

Static headspace extraction

Static headspace extraction involves the sampling of the headspace above a food matix sample in a sealed vial under predetermined equilibrium time/temperature conditions to reach equilibrium (Snow and Slack, 2002 and Wampler, 2002). Once at equilibrium the volatiles in the headspace are sampled using a gas-tight syringe and injected directly into a GC-MS or incorporated into the GC-MS via a pressurised heated transfer line (Friedrich and Acree, 1998 and Snow and Slack, 2002). According to Wampler (2002) static headspace extraction can easily be set up for automatic sampling. Furthermore is has low analysis costs and uncomplicated sample preparation. However, a drawback of this technique is its poor sensitivity for less volatile

compounds, when compared to dynamic headspace (purge-and-trap) extraction or SPME, and is mainly used to sample volatiles at higher concentrations (Snow and Slack, 2002).

Dynamic headspace (purge-and-tap) extraction

This sampling technique involves the bubbling or purging of an inert carrier gas into an aqueous sample in a closed purging vessel (Snow and Slack, 2002). The gas carrying the purged volatiles passes through a trap made up of a sorbent material, with a large surface area, which adsorbs and concentrates the volatiles (Wampler, 2002 and Friedrich and Acree, 1998). The trapped volatiles are then thermally desorbed from the trap and analysed using GC-MS or GC-FID (Contarini et al., 1997; Contarini and Povolo, 2002 and Naudé et al., 2009). According to Imhof and Bosset (1994) artefact formation during dynamic headspace extraction is subsequently reduced due to the single extraction and concentration step at relatively low extraction temperatures. Furthermore the variety of different sorbent traps available, such as Tenax, Carbosieve, Carboxen and Ambersorb allow for efficient concentration and selective extraction of specific volatiles during headspace analysis (Wampler, 2002). However, due to the fact that these traps are so tightly packed with sorbent material some have been noted to cause problematic desorption because of high inlet pressure and gas flow shutdown (Naudé et al., 2009). Polydimethylsiloxane (PDMS) multi channel traps (MCT) traps have been used to sample the headspace of different food matrices including UHT milk (Sivakumar et al., 2008 and Naudé et al., 2009). It has been noted by Naudé et al. (2009) that gas flow and inlet pressure problems were not experienced during the thermal desorption of PDMS MCT's due to the open tubular structure of the MCT. A major advantage of dynamic headspace extraction is the techniques sensitivity allowing analysis of volatiles at ppb concentration ranges (Snow and Slack, 2002 and Wampler, 2002). However, at high analyte concentrations volatile carry-over can occur between consecutive GC sequences leading to incorrect volatile quantification (Marsili, 1999). Furthermore analyte breakthrough and collection of moisture on the traps can lead to volatile losses and decreased retention time repeatability of early eluting compounds (Naudé et al., 2009).

Headspace solid-phase microextraction (HS-SPME)

HS-SPME involves the direct extraction and concentration of volatiles in the headspace above a sample onto a sorbent phase coated onto the outside of a fused silica fibre (Snow and Slack, 2002). The trapped analytes are then thermally desorbed from the fibre and analysed using a GC-MS or in the case of solvent desorption, injected into a high performance liquid chromatograph (HPLC) (Kataoka et al., 2000 and Povolo and Contarini, 2003). In-tube-SPME or solid-phase dynamic extraction (SPDE) involves the coating of a sorbent phase onto the inside surface of a fused silica fibre or the inside walls of the needle of a gas tight syringe (Zhang et al., 1994 and Lord and Pawliszyn, 2000). This coating arrangement allows for larger extraction volumes (up to six times) when compared with a 100 µl SPME fibre (Musshoff et al., 2002). According to Jochmann et al. (2006) SPDE has comparable or improved sensitivity for a number of volatile compounds, from water samples, when compared with other enrichment techniques including SPME. SPME techniques can be executed manually or by using an autosampler (Zhang et al., 1994).

A number of articles have been published comparing dynamic headspace (purge-and-trap) techniques to HS-SPME to analyse the flavour profile of different food stuffs (Contarini and Povolo, 2002; Elmore et al., 1997; Marsili, 1999 and Povolo and Contarini, 2003).

In the volatile analysis of two different cola drinks Elmore et al. (1997) indicated that there was no difference in the reproducibility between the two techniques and that for trace analysis dynamic headspace extraction is most suitable where for analysis of major volatile components HS-SPME is a better option. Similarly, comparable reproducibility for the two different methods was reported by Contarini and Povolo (2002) and Povolo and Contarini (2003) for the volatile analysis of milk and butter respectively. Furthermore greater amounts of volatiles were extracted by dynamic headspace extraction however; HS-SPME had superior extraction efficiency for higher molecular weight volatiles (Povolo and Contarini, 2003). In a study for the analysis of light induced lipid oxidation products in milk Marsili (1999) indicated that HS-SPME, when compared to dynamic headspace (purge-and-trap) techniques, is less expensive; did not lead to carryover or artefact formation and showed superior precision and accuracy. Furthermore the

author indicated that the sensitivity of HS-SPME is equal to that of dynamic headspace techniques. In general HS-SPME is a rapid, sensitive, uncomplicated, inexpensive and versatile technique (Kataoka et al., 2000; Roberts et al., 2000, Snow and Slack, 2002 and Vazquez-Landaverde et al., 2005). However limitations such as competition of volatiles for the sorbent phase of the fibre which may lead to erroneous results (Grote and Pawliszyn, 1997 and Roberts et al., 2000). It has been noted that the risk of artefact formation can increase at higher extraction temperatures (Perkins et al., 2005). Furthermore the sorbent phase coated onto the SPME fibre has a relatively small sorbent volume of approximately 0.9 mm^3 (Schulz et al., 2007) when compared to a volume of approximately 635 mm^3 of the PDMS MCT, used in dynamic headspace analysis, providing a much larger sample enrichment (Naudé et al., 2009).

Stir Bar Sorptive Extraction (SBSE)

SBSE, a SPME variant and relatively new extraction technique, involves the coating of glass covered magnetic stir bars with a thick layer of PDMS (Baltussen et al., 1999 and Snow and Slack, 2002). By introducing the phase coated stir bar directly into an aqueous sample, extraction can take place during stirring (Baltussen et al., 1999). After efficient stirring the stir bar can be inserted into a glass desorption tube and thermally desorbed into a GC inlet for analysis (Snow and Slack, 2002). Blasco et al. (2004) reported that SBSE has a greater volatile concentration capacity, accuracy and sensitivity for several different pesticides in honey when compared to SPME at similar experimental conditions. Similarly van Aardt et al. (2009) indicated that SBSE proved to be more sensitive than SPME for the quantitative analysis of volatile compounds in UHT milk. These advantages are achieved because the coated stir bars have a larger sorbent volume of 25-200 µl PDMS (Bicchi et al., 2004) compared to the relatively lower sorbent volume of 0.6-0.9 µl associated with the SPME fibre coating (Bicchi et al., 2000; Bicchi et al., 2004 and Schulz et al., 2007). However, SBSE cannot be fully automated which is a clear disadvantage of the procedure (Popp et al., 2003).

CONCLUSION

UHT processing leads to the formation of a variety of volatile compounds derived from the milks chemical constituents. Some of these volatiles have been identified to contribute to specific undesirable flavour notes in UHT milk. The "cooked" flavour of UHT milk is associated with the presence of a variety of sulphur containing compounds while the "stale" flavour is characterised by the dissipation of these sulphur volatiles and an increase of the formation and presence of both methyl ketones and aliphatic aldehydes over time. However, the extent that the individual volatiles contribute to the overall flavour of UHT milk is still unclear. A number of different methods, including the use of drop in additives as well as active packaging, have been identified as potential means to remove or decrease the occurrence of these undesirable flavours associated with UHT milk and ultimately improve the consumer acceptability of the product. Continuous improvements in gas chromatography and the development of new analytical techniques, may offer a better understanding as to how the volatile components of UHT milk interact with the milks constituents, packaging material and storage environment. Furthermore, this may improve the understanding of which odorants contribute most to the undesirable flavour of UHT milk, allowing for further research to focus on selective reduction of the these flavour volatiles from the milk.

REFERENCES

Aboshama, K., and Hansen, A.P. (1977). Effect of Ultra-High-Temperature Steam Injection Processing on Sulfur-Containing Amino Acids in Milk. *J. Dairy Sci.* 60: 1374-1378.

Al-Attabi, Z., D'Arcy, B.R., and Deeth, H.C. (2009). Volatile Sulphur Compounds in UHT Milk. *CRC. Cr. Rev. Food Sci.* 49: 28-47.

Arora, D.K., Hansen, A.P., and Armagost, M.S. (1991). Sorption of Flavour Compounds by Low Density Polyethylene Film. *J. Food Sci.* 56: 1421-1423.

Badings, H.T., and Van der Pol, J.J.G. (1973). Effect of cooling milk on the heat-release of hydrogen sulphide from the fat-globule membrane. *Neth. Milk Dairy J.* 27: 45-53.

Ballance, P.E. (1961). Production of Volatile Compounds Related to The Flavour of Foods From The Strecker Degradation of DL-Methionine. J. Sci. Food Agric. 12, 532-536.

Baltussen, E., Sandra, P., David, F., and Cramers, C. (1999). Stir Bar Sorptive Extraction (SBSE), a Novel Extraction Technique for Aqueous Samples: Theory and Principles. *J. Microcolumn Separations*. 11:737-747.

Bicchi, C., Cordero, C., Lori, C., Rubiolo, P., and Sandra, P. (2000). Headspace Sorptive Extraction (HSSE) in the Headspace Analysis of Aromatic and Medicinal Plants. J. *High Resolut. Chromatogr*. 23: 539-546.

Bicchie, C., Cordero, C., Liberto, E., Rubiolo, P., and Sgorbini, B. (2004). Automated headspace solid-phase dynamic extraction to analyse the volatile fraction of food matrices. *J. Chromatogr. A* 1024: 217-226.

Blasco, C., Fernández, M., Picó, Y., and Font, G. (2004). Comparison of solid-phase microextraction and stir bar sorptive extraction for determining six organophosphorus insecticides in honey by liquid chromatography-mass spectrometry. *J. Chromatogr. A* 1030: 77-85.

Blankenagel, G., and Humbert, E.S. (1963). Sulfhydryl groups and cooked flavour in ultra-high-temperature processed skim milk. *J. Dairy Sci.* 46: 614.

Boyd, E.N., and Gould, I.A. (1957). Volatile and Nonvolatile Sulphydryl Content of Heated Milk and Milk Products. *J. Dairy Sci.* 40: 1294-1307.

Brodie, III, V., and Visioli, D.L. (1994). Aldehyde scavenging compositions and methods relating thereto. United States Patent and Trademark Office 5284892.

Calvo, M.M., and De la Hoz, L. (1992). Flavour of Heated Milks. A Review. *Int. Dairy J.* 2: 69-81.

Christensen, K.R., and Reineccius, G.A. (1992). Gas Chromatographic Analysis of Volatile Sulphur Compounds from Heated Milk Using Static Headspace Sampling. *J. Dairy Sci.* 75: 2098-2104.

Clare, D.A., Bang, W.S., Cartwright, G., Drake, M.A., Coronel, P., and Slmunovic, J. (2005). Comparison of Sensory, Microbiological, and Biochemical Parameters of Microwave Verses Indirect UHT Fluid Skim Milk During Storage. *J. Dairy Sci.* 88: 4172-4182.

Colahan-Sederstrom, P.M., and Peterson, D.G. (2005). Inhibition of Key Aroma Compound Generated during Ultrahigh-Temperature Processing of Bovine Milk via Epicatechin Addition. *J. Agric. Food Chem*. 53: 398-402.

Contarini, G., and Povolo, M. (2002). Volatile Fraction of Milk: Comparison between Purge and Trap and Solid Phase Microextraction Techniques. *J. Agric. Food Chem*. 50: 7350-7355.

Contarini, G., Povolo, M., Leardi, R., and Toppino, P.M. (1997). Influence of heat treatment on the volatile compounds of milk. *J. Agric. Food Chem*. 45: 3171–3177.

Czerny, M., and Schieberle, P. (2007). Influence of the polyethylene packaging on the adsorption of odour-active compounds from UHT-milk. *Eur. Food Res. Technol.* 225: 215-223.

Day, E.A., and Lillard, D.A. (1960). Autoxidation of Milk Lipids. I. Identification of Volatile Monocarbonyl Compounds From Autoxidised Milk Fat. *J. Dairy Sci.* 43: 585-597.

Day, E.A., Lillard, D.A., and Montgomery, M.W. (1963). Autoxidation of Milk Lipids. III. Effect on Flavour of The Additive Interactions of Carbonyl Compounds at Subthreshold Concentrations. *J. Dairy Sci.* 46: 291-294.

Elmore, J.S., Erbahadir, M.A., and Mottram, D.S. (1997). Comparison of Dynamic Headspace Concentration on Tenax with Solid Phase Microextraction for the Analysis of Aroma Volatiles. *J. Agric. Food Chem*. 45: 2638-2641.

Ferretti, A. (1973). Inhibition of Cooked Flavour in Heated Milk by Use of Additives. *J. Agric. Food Chem*. 21: 939-942.

Ferretti, A., Edmondson, L.F., and Douglas Jr, F.W. (1974). Control of Cooked Flavour in High-Temperature Short-Time Milk Concentrates with a Sulphydryl-Blocking Agent. *J. Agric. Food Chem*. 22: 1130-1132.

Friedrich, J.E., and Acree, T.E. (1998). Gas Chromatography Olfactometry (GC/O) of Dairy Products. *Int. Dairy J.* 8: 235-241.

Grote, C., and Pawliszyn, J. (1997). Solid-Phase Microextraction for the Analysis of Human Breath. *Anal. Chem*. 69: 587-596.

Guichard, E. (2002). Interaction between flavour compounds and food ingredients and their influence on flavour perception. *Food Rev. Int.* 18, 49-70.

Hansen, A.P., Turner, L.G., and Jones, V.A. (1974). Effect of Ultra-high Steam Injection on Flavour Acceptability of Whole and Fortified Skim Milks. *J. Dairy Sci.* 57: 280-284.

Hutton, J.T., and Patton, S. (1952). The Origin of Sulphydryl Groups in Milk Proteins and Their Contributions To "Cooked" Flavour. *J. Dairy Sci.* 35: 699-705.

Imhof, R., and Bosset, J.O. (1994). Quantitative GC-MS Analysis of Volatile Flavour Compounds in Pasteurised Milk and Fermented Milk Products Applying a Standard Addition Method. *Lebensm. Wiss. Technol.* 27: 265-269.

Jaddou, H.A., Pavey, J.A., and Manning, D.J. (1978). Chemical analysis of flavour volatiles in heat-treated milks. *J. Dairy Res.* 45: 391-403.

Jenq, W., Bassette, R., and Crang, R.E. (1988). Effects of Light and Copper Ions on Volatile Aldehydes of Milk and Milk Fractions. *J. Dairy Sci.* 71: 2366-2372.

Jeon, I.J., Thomas, E.L., and Reineccius, G.A. (1978). Production of Volatile Compounds in Ultrahigh-Temperature Processed Milk During Aseptic Storage. *J. Agric. Food Chem*. 26: 1183-1188.

Jochman, M.A., Kmiecik, M.P., and Schmidt, T.C. (2006). Solid-phase dynamic extraction for the enrichment of polar volatile organic compounds from water. *J. Chromatogr. A* 1115: 208-216.

Kataoka, H., Lord, H.L., and Pawliszyn, J. (2000). Applications of solid-phase microextraction in food analysis. *J. Chromatogr. A* 880: 35-62.

Keenan, T.W., and Lindsay, R.C. (1968). Evidence for a Dimethyl Sulphide Precursor in Milk. *J. Dairy Sci.* 51: 112-114.

Kochhar, S.P. (1996). Oxidative Pathways to The Formation of Off-Flavours. In: Food Taints and Off-Flavours, pp. 168-225. Saxby, M.J. Eds., Chapman and Hall, Glasgow.

Langler, J.E., and Day, E.A. (1964). Development and Flavour Properties of Methyl Ketones in Milk Fat. *J. Dairy Sci.* 47: 1291-1296.

Linssen, J.P.H., Verheul, A., and Roozen, J.P. (1991). Adsorption of Flavour Compounds by Packaging Material: Drink Yoghurts in Polyethylene Bottles. *Int. Dairy J.* 1: 22-40.

Lord, H., and Pawliszyn, J. (2000). Evolution of solid-phase microextraction technology. *J. Chromatogr. A* 885: 153-193.

Marsili, R.T. (1999). Comparison of Solid-Phase Microextraction and Dynamic Headspace Methods for the Gas Chromatographic-Mass Spectrometric Analysis of Light-Induced Lipid Oxidation Products in Milk. *J. Chromatogr. Sci.* 37: 17-23.

Marsili, R.T. (2000). Shelf-Life Prediction of Processed Milk by Solid-Phase Microextraction, Mass Spectrometry, and Multivariate Analysis. *J. Agric. Food Chem*. 48: 3470-3475.

Mehta, R. S. (1980). Milk Processed at Ultra-High-Temperatures- A Review. *J. Food Protect.* 43: 212-223.

Mehta, R.S., and Bassette, R. (1978). Organoleptic, Chemical and Microbiological Changes In Ultra-High-Temperature Sterilised Milk Stored at Room Temperature. *J. Food. Protect*. 41, 806-810.

Moio, L., Etievant, P., Langlois, D., Dekimpe, J., and Addeo, F. (1994). Detection of powerful odoants in heated milk by use of extract dilution sniffing analysis. *J. Dairy Res.* 61: 385-394.

Morgan, M.E., Fross, D.A., and Patton, S. (1957). Volatile Carbonyl Compounds Produced In Skimmilk By High-Heat Treatment. *J. Dairy Sci.* 40: 571-578.

Musshoff, F., Lachenmeier, D.W., Kroener, L., and Madea, B. (2002). Automated headspace solid-phase dynamic extraction for the determination of amphetamines and synthetic designer drugs in hair samples. *J. Chromatogr. A* 958: 231-238.

Naudé, Y., Van Aardt, M., and Rohwer, E.R. (2009). Multi-channel open tubular traps for headspace sampling, gas chromatographic fraction collection and olfactory assessment of milk volatiles. *J. Chromatogr. A* 1216: 2798-2804.

Nursten, H.E. (1981). Recent Developments In Studies of The Maillard Reaction. *Food Chem*. 6: 263-277.

Nielsen, T.J., Jägerstad, I.M., Öste, R.E., and Wesslén, B.O. (1992). Comparative Adsorption of Low Molecular Aroma Compounds into Commonly Used Food Packaging Polymer Films. *J. Food Sci.* 57: 490-492.

Parks, O.W., Keeney, M., Katz, I., and Schwartz, D.P. (1964). Isolation and characterization of the methyl ketone precursor in butter fat. *J. Lipid Res.* 5: 232-235.

Patrick, P.S., and Swaisgood, H.E. (1976). Sulfhydryl and Disulfide Groups in Skim Milk Affected by Direct UltraiHigh-Temperature Heating and Subsrquant Storage. *J. Dairy Sci.* 59: 594-600.

Perkins, M.L., D'Arcy, B.R., Lisle, A.T., and Deeth, H.C. (2005). Solid phase microextraction of stale flavour volatiles from the headspace of UHT milk. *J. Sci. Food Agr.* 85: 2421-2428.

Perkins, M.L., Zerdin, K., Rooney, M.L., D'Arcy, B.R., and Deeth, H.C. (2006). Active Packaging of UHT Milk to Prevent the Development of Stale Flavour during Storage. *Packag. Technol. Sci.* 20: 137-146.

Popp, P., Bauer, C., Hauser, B., Keil, P., and Wennrich, L. (2003). Extraction of polycyclic aromatic hydrocarbons and organochlorine compounds from water: A comparison between solid-phase microextraction and stir bar sorptive extraction. *J. Sep. Sci*. 26: 961-967.

Povolo, M., and Contarini, G. (2003). Comparison of solid-phase microextraction and purge-and-trap methods for the analysis of the volatile fraction of butter. *J. Chromatogr. A* 985: 117-125.

Ramshaw, E.H., and Dunstone, E.A. (1969). Volatile compounds associated with the off-flavour in stored casein. *J. Dairy Res.* 36: 215-223.

Renner, E.H. and Berlage-Weining, L. (1983). Influence of added L-cystine on organoleptic quality of UHT milk. *Milchwissenschaft* 38, 324-327.

Rerkrai, S., Jeon, I.J., and Bassette, R. (1987). Effects of Various Direct Ultra-High Temperature Heat Treatments on Flavour of Commercially Prepared Milks. *J. Dairy Sci.* 70: 2046-2054.

Roberts, D.D., Pollien, P., and Milo, C. (2000). Solid-Phase Microextraction Method Development for Headspace Analysis of Volatile Flavour Compounds. *J. Agric. Food Chem*. 48: 2430-2437.

Robertson, G.L. (2006). Food Packaging: Principles and Practice Second Edition, pp. 20-21, 395-396. Eds., Boca Raton: Taylor & Francis Group.

Rychlik, M., Schieberle, P., and Grosch, W. (1998). Compilation of odour thresholds, odour qualities and retention indices of key food odourants. Garching: Deutsche Forschungsanstat Für Lebensmittelchemie and Instit Für Lebensmittelchemie der Technischen Universität München. pp. 1-53.

Sajilata, M.G., Savitha, K., Singhal, R.S., and Kanetkar, V.R. (2007). Scalping of Flavours in Packaged Foods. *Compr. Rev. Food Sci. F.* 6: 17-35.

Samuelsson, E.G., and Borgström, S. (1973). Method for protecting milk against flavour deterioration when heat treated. *Milchwissenschaft* 28: 25-26.

Schulz, K., Dreβler, J., Sohnius, E., and Lachenmeir, D.W. (2007). Determination of volatile constituents in spirits using headspace trap technology. *J. Chromatogr. A* 1145: 204-209.

Schutte, L., and Koenders, E.B. (1972). Components Contributing to Beef Flavour. Natural Precursors of 1-Methylthio-ethanethiol. *J. Agric. Food Chem*. 20: 181-184.

Schutte, L. and Teranishi, R. (1974). Precursors of Sulphur-Containing Flavour Compounds. *CRC. Cr. Rev. Food Sci.* 4, 457-505.

Schwartz, D.P., Parks, O.W., and Yoncoskie, R.A. (1966). Quantitative Studies on Methyl Ketone Formation in Butteroil: Effect of Temperature. *J. Am. Oil Chem. Soc.* 43: 128-129.

Selke, S.E. (2003). Applications of Plastic Films in Packaging. In: Handbook of Plastic Films, pp. 242-243. Abdel-Bary, E.M. . Eds., Rapra Technology Limited, Shawbury UK.

Shibamoto, T., Mihara, S., Nishimura, O., Kamiya, Y., Aitoku, A., and Hayashi, J. (1980). Flavour Volatiles Formed By Heated Milk. *The Analysis and Control of Less Desirable Flavours in Food and Beverages*., pp. 241-26. Eds., Academic Press, New York.

Shipe, W.F., Bassette, R., Deane, D.D., Dunkley, W.L., Hammond, E.G., Harper, W.J., Kleyn, D.H., Morgan, M.E., Nelson, J.H., and Scanlan, R.A. (1978). Off Flavours of Milk: Nomenclature, Standards, and Bibliography. *J. Dairy Sci.* 61: 855-869.

Simon, M., Hansen, A.P., and Young, C.T. (2001). Effect of Various Packaging Materials on the Headspace Analysis of Ultrapasterized Milk. *J. Dairy Sci.* 84: 774-783.

Scanlan, R.A., Lindsay, R.C., Libbey, L.M., and Day, E.A. (1968). Heat-Induced Volatile Compounds in Milk. *J. Dairy Sci*. 51: 1001-1007.

Sivakumar, D., Naudé, Y., Rohwer, E.R., and Korsten, L. (2008). Volatile compounds, quality attributes, mineral composition and pericarp structure of South African litchi export cultivars Mauritius and McLean's Red. *J. Sci. Food Agr. 88*: 1074-1081.

Snow, N.H., and Slack, G.C. (2002). Head-space analysis in modern gas chromatography. *Trac-Trend. Anal. Chem*. 21: 608-617.

Suloff, E.C., Marcy, J.E., Blakistone, B.A., Duncan, S.E., Long, T.E., and O'Keefe, S.F. (2003). Sorption Behavior of Selected Aldehyde-scavenging Agents in Poly (ethylene terephthalate) Blends. *J. Food Sci.* 68: 2028-2033.

Swaisgood, H.E. (1977). Process of Removing the Cooked Flavour From Milk. United States Patent and Trademark Office 4053644.

Thomas, E.L., Burton, H., Ford, J.E., and Perkin, A.G. (1975). The effect of oxygen content on flavour and chemical changes during asceptic storage of whole milk after ultra-high-temperature processing. *J. Dairy Res.* 42: 285-295.

Thomas, E.L., Reineccius, G.A., Dewaard, G.J., and Slinkard, M.S. (1976). Determination of Hydrogen Sulphide in Heated milks by Gas-Liquid Chromatographic Headspace Analysis. *J. Dairy Sci.* 59: 1865-1869.

Townley, R.C., and Gould, I.A. (1943). A Quantitative Study of Heat Labile Sulfides of Milk. 1. Method of Determination And The Influence of Temperature and Time. *J. Dairy Sci.* 26: 689-703.

Valero, E., Villamiel, M., Miralles, B., Sanz, J., and Martinez-Castro, I. (2001). Changes in flavour and volatile components during storage of whole and skimmed UHT milk. *Food Chem.* 72: 51-58.

Van Aardt, M. (2009). Quantification of volatile flavours associated with cooked milk- stir bar sorptive extraction (SBSE) coupled with gas chromatography-mass spectrometry, pp. 1-14. Post Doctorial Fellowship Final Report, University of Pretoria, Pretoria, South Africa.

Van Willige, R.W.G., Linssen, J.P.H., and Voragen, A.G.J. (2000a). Influence of food matrix on absorption of flavour compounds by linear low-density polyethylene: protein and carbohydrates. *J. Sci. Food Agr.* 80: 1779-1789.

Van Willige, R.W.G, Linssen, J.P.H., and Voragen, A.G.J. (2000b). Influence of food matrix on absorption of flavour compounds by linear low-density polyethylene: oil and real food products. *J. Sci. Food Agr* 80: 1790-1797.

Vazquez-Landaverde, P.A., Velazquez, G., Torres, J.A., and Qian, M.C. (2005). Quantitative Determination of Thermally Derived Off-Flavour Compounds in Milk Using Solid-Phase Microextraction and Gas Chromatography. *J. Dairy Sci.* 88: 3764-3772.

Vazquez-Landaverde, P.A., Torres, J.A., and Qian, M.C. (2006). Quantification of Trace Volatile Sulfur Compounds in Milk by Solid-Phase Microextraction and Gas Chromatography-Pulsed Flame Photometric Detection. *J. Dairy Sci.* 89: 2919-2927.

Wampler, T.P. (2002). Analysis of Food Volatiles Using Headspace-Gas Chromatographic Techniques. In: Flavour Fragrance and Odour Analysis, pp. 25-54. Marsili, R., Eds., Marcel Dekker, Inc, New York.

Zhang, Z., Yang, M.J., and Pawliszyn, J. (1994). Solid-Phase Microextraction: A Solvent-Free Alternative for Sample Preparation. *Anal. Chem.* 66: 844A-853A.