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# **Original Paper**

# Analysis of the volatile components of Cheddar cheese by direct thermal desorption- $GC \times GC$ -TOF/MS

Volatile compounds were isolated from Cheddar cheese using direct thermal desorption (DTD) and analysed using comprehensive 2-D GC (GC × GC) coupled with TOF MS (TOF/MS). In total 12 aldehydes, 13 acids, 13 ketones, 5 alcohols, 3 hydrocarbons and 9 miscellaneous compounds were identified at desorption temperatures of 100, 150, 200 and 250°C using mature Cheddar cheese. A temperature of  $150^{\circ}$ C was found to be optimum for the DTD of volatiles from mature Cheddar cheese. The major components were acetic acid, butanoic acid, 3-hydroxy-2-butanone and 2,3-butanediol. A DTD temperature of  $150^{\circ}$ C was used to observe the effect of maturation (mild, medium or mature) on the volatiles of Cheddar cheese. The major components of the volatiles of mild, medium and mature Cheddar cheese were almost the same. However, their percentage compositions were found to change with the stage of maturity. DTD is simple, fast and requires only a small amount of sample (approximately 10 mg) and works well with comprehensive GC × GC-TOF/MS. Comprehensive GC also separated a number of components which remained overlapped on the single column, such as octane and hexanal.

**Keywords:** Cheddar cheese / Direct thermal desorption / GC × GC-TOF/MS / Volatiles Received: October 20, 2005; revised: January 18, 2006; accepted: March 7, 2006 DOI 10.1002/jssc.200500400

# **1** Introduction

The organoleptic properties of cheese are considered to be its most important characteristic in product evaluation and are also important in consumer acceptance and preference. The flavour profiles of cheeses are complex and are variety and type specific [1]. The properties of taste and aroma are the result of a very complex process involving the origin of the milk, the cheese-making process and the microflora which develop during ripening.

The analysis of volatiles is generally accomplished by an extraction step, followed by concentration, chromatographic separation and subsequent detection. Well established methods of analysis of volatiles of Cheddar cheese include steam distillation [2], low-temperature vacuum distillation [3], solvent extraction [4], dialysis [5], dynamic headspace dilution analysis [6] and aroma extract dilution analysis [7]. An overview of sample preparation

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Abbreviation: DTD, direct thermal desorption

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methods is provided by Pillonel *et al.* [8] for food volatiles and by Wilkes *et al.* [9] for food flavours and off-flavours. The chromatographic profile will vary depending upon the method of sample preparation employed, and it is not uncommon to produce artifacts during this step.

The dynamic headspace technique is a very popular method for analysing volatile compounds in plant materials [10], in airborne pollutants [11] and in cheese [12]. Direct thermal desorption (DTD) is one version of the dynamic headspace technique with cryogenic trapping postdesorption used to enrich the analytes prior to separation. DTD has important advantages over the other methods such as direct coupling to GC and MS [13], the requirement of only a small amount of sample and of course the fact that it is a rapid method. According to previous literature, the DTD method has been applied to various systems such as beef [14], plant material [11], sugar [15] and cheese [16].

Comprehensive 2-D GC (GC × GC) is a fully multidimensional technique that has an increased separation power with reduced analysis time. GC × GC has been used by Marriott *et al.* [17] and by Ozel *et al.* [18] for essential oils and by Arora *et al.* [12] for cheese. The use of a mass spectrometer is highly desirable for identification of the



numerous separated compounds found during a  $GC \times GC$  run. At present, TOF/MS is probably the most compatible MS technique, although faster scanning quadrupole instruments are also emerging. Dalluge *et al.* [19] and Shellie and Marriott [20] also reported that  $GC \times GC$ -TOF/MS provides a reliable basis for the automated analysis of complex samples.

A great future strength of the technique, however, is that it may allow the elimination of the traditional sample preparation stages in a number of areas. Conceptually, the DTD and the first 'GC' in 'GC  $\times$  GC' are the sample preparation stages and it is the fully online properties that may be exploited in screening studies [18]. The analytical technique of DTD coupled with GC  $\times$  GC is a very viable one. It is suitable especially for qualitative marker compound analysis. It may also have the potential as a technique for monitoring the manufacturing process of the cheese itself.

The DTD technique used here has been applied in only a limited fashion previously [20] and we believe that there is significant scope for DTD coupled with  $GC \times GC$  separation online. The particular aim of this study has been to examine the effect of maturation on the composition of Cheddar cheese volatiles by using DTD coupled with comprehensive GC-TOF/MS.

## **2 Experimental**

#### 2.1 Materials

Cheddar cheeses of different maturities (mild, medium, mature) of the same brand were purchased from a retail market (York, UK). The various grades of maturity for Cheddar cheese are defined as follows: mild Cheddar is 3 months old, medium Cheddar is 6 months old and mature Cheddar is 9-12 months old. The nutritional information as given on the labels of the cheese samples showed that they contained 25.0 g protein, 34.1 g fat and 0.1 g carbohydrate per 100 g. All cheese samples were kept in their original vacuum packages at 5°C until being used. A 1 cm slice was removed from each surface of the cheese samples to minimize the possibility of eliminating flavours that would have migrated into them. The remaining portion of each sample was grated into very small pieces of approximately 50 mg in weight. The grated pieces were placed in a glass desiccator which contained PCl<sub>5</sub> at the bottom. The glass desiccator was flushed with nitrogen gas and stored in the dark at  $5^{\circ}C$ until the cheese samples were dry. The final moisture content of the cheese was less than 1% (w/w on dry basis). Maximum drying time was 24 h.

#### 2.2 DTD method

The GC injection port liner (SGE, Ringwood, Australia) was removed and  $10 \pm 1$  mg of dried cheese sample was

placed into it using tweezers to ensure no contamination of the sample. Glass wool was employed to hold the sample in place, since it was prone to movement when the injector was pressurized and depressurized, and also to ensure that no solid material escaped into the gas lines. The GC inlet was held at 40°C and the liner containing the sample carefully inserted into it. The GC liner was purged for 2 min at ambient temperature using helium to remove water vapour and permanent gases. The head of the primary column (DB5) was cryo-cooled using liquid nitrogen. The temperature of the GC inlet was then quickly raised to the selected temperature (100, 150, 200 and 250°C) and held isothermally for a further 5 min for desorption of all volatile and semivolatile organic materials. After a 5 min desorption, the liquid nitrogen trap was removed and the oven programme initiated. The glass wool was replaced after each run.

#### 2.3 GC $\times$ GC-TOF/MS system

The GC × GC-TOF/MS system consisted of a HP 6890 (Agilent Technologies, CA, USA) gas chromatograph and a Pegasus III TOF-MS (LECO, MI, USA). The first column was a nonpolar DB5 (10 m  $\times$  0.18 mm id  $\times$  0.18  $\mu$ m film thickness) and the second column a DB17 (1.6 m  $\times$  0.18 mm  $id \times 0.18 \,\mu m$  film thickness). Both columns were from J&W Scientific (CA, USA). The columns were connected by means of a press-fit-connector. The second dimension column was installed in a separate oven, which was maintained in the main GC oven. The modulator is the key to the performance of the  $GC \times GC$  experiment. In this case, cryogenic modulation was performed using a jet-type modulator installed at the head of the second dimension column. It consisted of two cold and two hot jets, with the nozzles providing the cold jets mounted orthogonal to the hot jets. Nitrogen gas was cooled by heat exchange through copper tubing immersed in liquid nitrogen outside the GC system and delivered through vacuum-insulated tubing to the cold jets. These provided two continuous jets of approximately 10 L/min of cold nitrogen gas at approximately -140°C. The modulation time was 10 s. When the hot downstream pulse was fired the analytes were effectively injected into the second dimension column. A detailed description of the set-up is given elsewhere [21].

Helium was used as a carrier gas. The initial temperature of the first dimension column was 40°C for 30 s, and the subsequent temperature programme was a heating rate of 5°C/min until 250°C was reached and held isothermally for a further 2 min. The initial temperature of the second dimension column was 60°C for 30 s, and a 5°C/ min heating rate was used until 270°C was reached and held isothermally for a further 2 min. Peak identification was made using TOF/MS with 70 eV electron impact ionisation. Mass spectra were compared against the NIST '98 (National Institute of Standards and Technology, MD, USA) mass spectral library.

## **3 Results**

A 1-D separation of the volatiles of Cheddar cheese may be compared to that performed using a 2-D GC system. Separation of the components belonging to a specific group such as hydrocarbons or aldehydes was quite clear; and hexanal, furfural, heptanal, benzaldehyde, octanal, nonanal, decanal, undecanal, dodecanal and tridecanal appeared on what was the front eluting plane of the separation. However, overlapping compounds such as octane and hexanal, and ethyl lactate and 3-octanol were only successfully separated in the second dimension. The use of fast detection TOF-MS for GC × GC of volatile components of Cheddar cheese showed that primarycolumn overlapping compounds were resolved on the second column. It should be noted that the peak identification of components was based on both library mass spectra and Kovats indices. Identification based on a mass spectral library search using similarity and reverse factors were above 750 and 800, respectively. Lower values than these were counted as unknown and the components having these low values were not compared for their Kovats indices. Dalluge et al. [19] and Ozel et al. [18] also used similarity and reverse factors above 750 and 800, respectively.

The analytical technique of dynamic headspace for the analysis of foodstuffs such as cheese is a very viable one. Table 1 lists the compounds identified in the volatiles of mature Cheddar cheese, together with their retention times and corresponding percentage compositions. The number of compounds extracted and identified at desorption temperatures of 100, 150, 200 and 250°C were 28, 43, 45 and 35, respectively (Table 1).

Most of the compounds found in this study have been previously reported in other studies on Cheddar cheese volatiles [1]. It can be seen from Table 1 that Cheddar cheese contains a high number of various volatile compounds: aldehydes, free fatty acids, hydrocarbons, ketones, lactones, alcohols, esters and sulphur compounds. Only 14 of the components identified were common to all desorption temperatures. A regular trend in the percentages of the components as a function of temperature was not found.

Even though over 90% of the material extracted at 150 and 200°C was the same, changing the temperature from 100 to 250°C made a big difference in the volatile profiles of mature Cheddar cheese samples. It was also calculated that the percentages of unidentified (unknown) components were greater at higher temperatures of 200 and 250°C. It was clear that most of the components found at 150°C were common either to the components found at

the higher or lower temperatures. The major components found common to all temperatures studied were acetic acid, butanoic acid, 3-hydroxy-2-butanone and 2,3butanediol. Some components were major at temperatures of 150, 200 and 250°C such as ethyllactate, 2-acetyl pyrrole; and some were only major at 250°C such as benzeneethanamine (7.5%), tetradecanoic acid (7.9%), penten decanoic acid (4.4%) and hexadecanoic acid (7.3%).

Table 2 shows the effect of maturation on the volatiles of Cheddar cheese. The studies to observe this effect were performed at a DTD temperature of 150°C. In earlier studies, Ozel *et al.* [18] reported that the optimum temperature of DTD was 150°C for the volatiles of *Pistacia vera L*. They reported that some components which were not the components of essential oils of *Pistacia vera L*. appeared at higher temperatures of 200 and 250°C. Although, the artifact formation was not particularly followed at the various desorption temperatures, hydrogen sulphide was not reported when the desorption temperature was below 150°C but increased dramatically at 200 and 250°C. This is a clear indication of artifact formation at high desorption temperatures.

The number of components identified at 150°C were 55, 52 and 43 for mild, medium and mature Cheddar cheese, respectively. In earlier studies Arora *et al.* [12] identified 41 components using a headspace method by purge and trap extraction; Vandeweghe and Reineccius [22] identified 21 components by distillation, 17 components by dialysis and 14 components by solvent extraction; and Suriyaphan *et al.* [7] identified 28 components by solvent extraction for various Cheddar cheese samples.

The major components of the volatiles of Cheddar cheese samples of various maturity were qualitatively almost the same as each other. However, it was observed that the percentage composition of these major components decreased or increased with varying maturity in some cases. For example, acetic acid, 3-hydroxy-2-butanone, nonanal, 2-acetylpyrrole and decanal increased, and ethanol, formic acid, 3-methylbutanal, glycerol, ethyl lactate and tetradecanoic acid decreased with increasing maturity. There were also some components with almost the same percentage in all samples. They were 2,3-butanediol, octanal, 3-octanol, octanoic acid and nonanoic acid. It has been described that having a sweet or a bitter flavour is a defect in normal Cheddar cheese [23]. The main components of these flavours, found in some earlier studies on Cheddar cheese, were 4-hydroxy-2,5dimethyl-3(2H)-furanone, ethyl butanoate and glutamic acid and serine [24, 25]. None of these components were detected in this study. Acetic acid has been found to be one of the highest percentage components in mild Cheddar cheese of both normal and reduced fat content [26]. It has also been reported that the acetic acid increased

Compound <sup>a)</sup>	Group name	$RT(s)^{b)}$	% <sup>c</sup> )							
			100°C	RSD <sup>d)</sup>	150°C	RSD	200°C	RSD	250°C	RSD
Acetaldehyde	А	320			1.1	2.9	1.3	3.2		
Hydrogen sulfide	Μ	320					0.5	6.2	2.6	6.0
Acetone	K	340	0.8	3.1	2.5	1.8	0.9	3.2		
Ethanol	OH	400	2.6	2.4	1.0	5.9	1.7	5.1	1.6	1.6
Butanone	K	410			0.5	5.8	0.3	4.7	0.3	3.3
Formic acid	С	430	0.7	2.7	0.8	5.6	1.0	2.3	0.5	5.4
Acetic acid	С	460	47.6	2.5	17.5	3.8	15.5	4.1	12.6	2.4
3-Methylbutanal	А	470			0.3	3.0	0.7	5.3	1.7	4.6
Propanoic acid	С	510			0.5	2.2				
3-Hydroxy-2-butanone	K	540	7.3	1.1	8.8	3.1	6.4	3.3	3.0	5.2
Hexanal	А	620			1.5	2.6	0.3	6.5		
Octane	HC	620			0.4	5.1	0.2	2.8		
2,3-Butanediol	OH	630	21.6	5.1	13.5	2.1	10.5	3.7	2.2	3.2
1,3-Butanediol	OH	650	0.6		1.5	3.4	0.2	2.8		
Butanoic acid	С	690	2.6	1.8	2.9	3.9	7.3	1.9	4.4	6.1
Furfural	А	710					0.1	5.8	1.0	3.8
Heptenone	К	720	0.2	2.9						
Nonane	HC	790	1.1	5.4	2.3	4.5	2.5	3.9		
2-Furanmethanol	OH	790					0.1	6.8	0.3	6.1
2-Heptanone	K	800					0.8	3.8	0.9	4.8
Heptanal	А	810	1.0	1.1	2.1	5.7	3.2	2.7		
N.N-Diethylformamide	М	890			0.5	3.1	0.7	2.1	0.7	4.6
Benzaldehyde	А	910	0.1	5.0	0.3	1.9	0.7	4.9		
2(5H)-Furanone	K	910							0.2	6.5
Glycerol	OH	960			0.1	1.2	0.2	6.1	0.4	7.3
Decane	HC	970	0.2	6.4	0.3	2.7	0.2	011	0.11	/10
Octanal	A	980	0.2	6.1	2.9	4.7	0.7	3.9		
Valerolactone	K	1090	0.2	011	0.4	1.9	0.6	3.4		
Ethyllactate	M	1120	0.5	4.3	4.5	2.8	6.9	1.5	6.5	5.1
3-Octanol	OH	1120	0.1	1.8	0.3	4.3	0.5	1.0	0.0	0.1
Hexanoic acid	C	1120	1.2	3.1	1.2	4.4	1.6	2.1	3.1	3.9
2-Nonanone	ĸ	1140	1.2	5.1	1.2		1.3	6.1	2.8	3.3
2-Acetylpyrrole	M	1140			2.5	1.1	4.0	6.1	4.6	5.0
Nonanal	A	1160	1.2	4.8	4.0	5.5	2.3	4.5	1.0	0.0
2-Pyrrolidinone	M	1190	1.2	4.0	4.0	0.0	2.5	1.5	1.0	6.5
Benzoic acid	C	1270			0.2	6.3	0.2	6.8	0.6	6.3
Octanoic acid	C	1270	0.2	5.7	1.9	3.9	1.5	1.6	0.0	0.5
Decanal	A	1340	4.5	2.1	11.6	6.0	3.1	2.8	0.8	6.1
Pulegone	K	1420	1.0	4.1	0.1	0.0 4.9	0.1	4.5	0.0	0.1
Nonanoic acid	С	1420			0.1	5.7	0.1	4.5 1.9		
Benzeneethanamine	M	1430			<b>F.</b> 0	5.7	2.2	2.4	7.5	3.5
2-Undecanone	K	1430			0.1	5.7	4.4	2.4	7.5 0.8	3.5 7.2
Undecanal	к А	1490	1.1	3.1	0.1 1.9	2.8	0.9	3.1	0.0	1.4
Decanoic acid	C	1590	0.2	3.1 6.1	2.1	2.8 4.1	0.9 1.5	3.1 5.5	0.4	2.7
			0.2	0.1		4.1 1.9				
Dodecanal Corapyl acotopo	A K	1670 1730			0.5 0.6	1.9 4.1	1.6	5.2	3.3	4.6
Geranyl acetone	к К	1730	0.1	6.6	0.6 0.1	4.1 6.1	1.4	4.3	3.3	3.6
γ-Decalactone Tridocanal			0.1	0.0						
Tridecanal δ-Dodecalactone	A	2010 2090	0.0		0.8	2.6	1.0	2.7	2.2	3.9 E 1
DL-Isoleucine	K		0.0		0.0		0.2	7.1	0.1	5.1
	M	2100			0.2	2 5	2.0	ΕC	0.9	6.3
Fetradecanoic acid	С	2140			0.2	3.5	2.0	5.6	7.9	2.5
sopropyl myristate	M	2220					0.0	4.0	0.7	1.9
Pentadecanoic acid	C	2280					0.8	4.2	4.4	3.1
Hexadecanoic acid	C	2380	0.1	5.0		2.0	3.5	3.9	7.3	5.4
Dodecanoic acid	С	2400	0.1	5.6	1.1	2.8	1.1	4.1	1.2	4.9
Stearyl alcohol	М	2410	0.4	3.1	0.9	4.1				<i>.</i> .
Unknown			3.9	2.2	3.6	3.1	6.4	3.3	8.5	6.1
			100.0		100.0		100.0		100.0	

 Table 1. Compounds, retention times and percentage compositions of the volatiles of mature Cheddar cheese at various temperatures of DTD

RT: retention time, RSD: relative standard deviation, A: aldehyde, C: acid, HC: hydrocarbon, K: ketone, M: miscellaneous, OH: alcohol.

a) As identified by  $GC \times GC$ -TOF/MS software; names according to NIST mass spectral library, and by comparing their Kovats retention indices. b) Retention time in seconds (column: BP5).

b) Retention time in seconds (column: BP5).

c) Percentage of each component is calculated as peak area of analyte divided by peak area of total ion chromatogram times 100 (In the case of multiple identification, the areas of the peaks that belong to one analyte were combined to find the total area for this particular analyte).

d) The RSDs for four (n = 4) experiments.

Compound <sup>a)</sup>	% <sup>c)</sup>									
	$RT^{b)}(s)$	KRI	Mild	RSD <sup>d)</sup>	Medium	RSD	Mature	RSD		
Acetone	340	477	0.8	5.1	2.4	1.6	2.5	1.8		
Ethanol	400	600	6.0	3.2	3.1	2.2	1.0	5.9		
Butanone	410	603	0.2	3.8	0.4	6.0	0.5	5.8		
Formic acid	430	620	2.4	6.1	1.9	5.0	0.8	5.6		
Acetic acid	460	640	11.7	2.8	13.8	2.5	17.5	3.8		
3-Methylbutanal	470	650	5.5	1.9	2.6	3.5	0.3	3.0		
Propanoic acid	510	668	0.6	6.5	0.5	2.8	0.5	2.2		
2-Methylbutanal	530	677	0.6	3.8	0.8	3.2				
3-Hydroxy-2-butanone	540	718	3.8	3.4	5.0	2.9	8.8	3.1		
Pentanal	560	732	0.5	5.4	0.5	6.2	0.0	011		
3-Methyl-2-butenal	600	783	0.1	6.2	0.7	6.5				
Octane	620	800	0.5	2.9	0.5	4.8	0.4	5.1		
Hexanal	620	801	0.6	3.6	1.7	4.3	1.5	2.6		
2,3-Butanediol	630	806	13.3	1.9	13.3	3.1	13.5	2.0		
-										
1,3-Butanediol	650	810	0.4	3.3	0.6	5.2	1.5	3.4		
Butanoic acid	690 720	820	4.4	2.9	3.5	3.2	2.9	3.9		
Heptenone	720	873	0.7	3.6	0.4	6.5	2.2	4 -		
Nonane	790	900	1.9	6.0	2.5	6.6	2.3	4.5		
Heptanal	810	903	0.5	65	0.7	3.5	2.1	5.7		
Pentanoic acid	830	911	0.3	6.5	0.8	5.4		. ·		
N,N-Diethylformamide	890	950			1.1	1.9	0.5	3.1		
Benzaldehyde	910	960	0.3	2.9	0.6	3.2	0.3	1.9		
Glycerol	960	990	1.5	3.9	0.7	6.3	0.1	1.2		
Decane	970	1000	0.2	6.1	0.3	2.8	0.3	2.7		
Octanal	980	1006	2.1	3.2	2.6	5.1	2.9	4.7		
/alerolactone	1090	1010	0.2	5.6	0.1	6.1	0.4	1.9		
Ethyl lactate	1120	1011	9.9	5.0	7.2	6.6	4.5	2.8		
3-Octanol	1120	1012	0.2	4.8	0.3	5.9	0.3	4.3		
Hexanoic acid	1130	1019	1.4	3.5	2.0	5.3	1.2	4.4		
2-Acetylpyyrole	1140	1045	0.5	3.3	0.9	2.6	2.5	1.1		
E)-2-Octen-1-ol	1150	1064	0.5	6.2	015	210	210			
Methyl benzoate	1160	1081	0.1	6.8	0.1	7.3				
2-Nonanone	1160	1093	0.3	3.6	0.1	7.0				
Nonanal	1160	1104	2.8	2.8	3.3	3.9	4.0	5.5		
Heptanoic acid	1180	1112	0.8	6.1	0.8	2.8	4.0	5.5		
Benzoic acid	1270	1176	0.8	5.4	0.8	2.8 5.4	0.2	6.3		
							0.2			
Octanoic acid	1280	1179	1.5	5.2	0.9	3.5	1.9	3.9		
Dodecane	1330	1200	0.7	3.9	0.4		11.5	6.0		
Decanal	1340	1209	6.1	5.4	9.4	2.7	11.6	6.0		
Pulegone	1420	1223					0.1	4.9		
Nonanoic acid	1430	1275	0.3	6.4	0.3	7.2	0.4	5.7		
2-Butyl-1-octanol	1460	1277	0.2	3.9	0.4	3.5				
2-Undecanone	1490	1296			0.1	6.6	0.1	5.7		
Undecanal	1500	1298	1.1	4.4	1.8	5.9	1.9	2.8		
Decanoic acid	1590	1373	1.6	5.1	1.7	4.2	2.1	4.1		
Dodecanal	1670	1408	0.1	4.6	0.5	4.3	0.5	1.9		
Geranyl acetone	1730	1448	0.9	2.8	0.9	6.3	0.6	4.1		
<i>r</i> -Decalactone	1810	1469	0.2	6.2	0.1	5.1	0.1	6.1		
4-Methyl-8-hexadecen-1-ol	1820	1476	0.1	4.9						
Jndecanoic acid	1830	1490	0.3	6.5						
Tridecanal	2010	1505	0.5	5.2	0.9	5.7	0.8	2.6		
-Dodecalactone	2050	1685	0.3	2.9	0.1	3.1	0.0	2.0		
-Nonadecanone	2030	1703	0.2	3.5	~	5.1				
-Dodecalactone	2070	1703	0.2	3.3	0.2	5.9	0.0			
etradecanoic acid						5.9 5.8	0.0	3.5		
	2140	1771	1.2	6.1	0.6		0.2	5.5		
sopropyl myristate	2220	1942	0.7	2.5	0.2	2.1				
Hexadecanoic acid	2380	1972	0.4	3.2				a -		
Dodecanoic acid	2400	2156	1.3	2.4	0.7	6.1	1.1	2.8		
Stearyl alcohol	2410	2232	0.4	6.1	0.7	2.7	0.9	4.1		
Unknown			6.3	7.1	4.3	6.6	3.6	3.1		
			100.0		100.0		100.0			

RT, retention time; KRI, Kovats retention indices; RSD, relative standard deviation.

a) As identified by  $GC \times GC$ -TOF/MS software; names according to NIST mass spectral library, and by comparing their Kovats retention indices. b) Retention time in seconds (column: BP5).

c) Percentage of each component is calculated as peak area of analyte divided by peak area of total ion chromatogram times 100 (In the case of multiple identification, the areas of the peaks that belong to one analyte were combined to find the total area for this particular analyte).

d) The RSDs for four (n = 4) experiments.

with decreasing fat content. Acetic acid was found to be the component with the highest percentage in this study also. However, even if it is high in concentration, its role on the final flavour score of the cheese is not as dominant as the lactones and alcohols [7].

Avsar *et al.* [27] found that specific volatile chemical compounds, the Strecker aldehydes: 2-methypropanol, 2methylbutanal and 3-methylbutanal were detected in higher amounts in Cheddar cheeses with nutty flavours when compared with Cheddar cheeses without nutty flavours. They also reported that most nutty flavoured Cheddar cheeses are older. Strecker aldehydes 3-methylbutanal and 2-methylbutanal were detected in this study also. However, the percentage of 3-methylbutanal decreased with increasing maturity.

### 4 Discussion

It was found that the numbers of components extracted at desorption temperatures of 100, 150, 200 and 250°C were 28, 43, 45 and 35, respectively. It has been shown that DTD as a sample preparation technique is advantageous in extracting all the components and in preventing contamination. High thermal desorption temperatures, such as 200 and 250°C, are not recommended because of the possible production of degradation products.

The major components of the volatiles common to Cheddar cheeses at varying stages of maturity were acetic acid, 3-hydroxy-2-butanone, decanal, ethyl lactate, 2,3butanediol and butanoic acid. It was also found that the percentages of some components increased, decreased or remained the same with changing maturity.

 $GC \times GC$ -TOF/MS has an increased separation and identification power for the analysis of volatile components of foods. The number and certainty of the identified components increased using the optimized conditions of  $GC \times GC$ .

The DTD method shows great promise as a technique for the evaluation of foodstuffs such as cheese. It can be performed quickly and easily without any time-consuming and conventional sample preparation techniques. A useful application would be that of reliable quality control during the manufacturing processes of foodstuffs.

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