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Two-fibre solid-phase microextraction combined with gas chromatography–mass spectrometry for the analysis of volatile aroma compounds in cooked pork

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Abstract

The volatile aroma compounds in cooked pork were examined using solid-phase microextraction (SPME). Two SPME fibres coated with different stationary phases were used simultaneously to collect aroma compounds from the headspace above the pork. One fibre was coated with 75 μm. Carboxen–polydimethylsiloxane and the other was coated with 50/30 μm divinylbenzene–Carboxen on polydimethylsiloxane. After extraction, the two fibres were desorbed in the injection port of a gas chromatograph sequentially, so that the aroma compounds from both of the fibres could be analysed in one gas chromatogram. This procedure resulted in a chromatogram containing a more complete aroma profile for cooked pork than the chromatograms from either of the fibres on their own. Thirty-six compounds were identified in cooked pork for the first time. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Meat; Food analysis; Extraction methods; Solid-phase microextraction; Aroma compounds; Volatile organic compounds

1. Introduction

Several workers have used solid-phase microextraction (SPME) to examine the volatile constituents of cooked meat [1–5]. Nielsen et al. [1] and Brunton et al. [2] measured aldehyde formation in cooked pork and turkey, respectively. Both sets of workers were examining SPME as a tool for the measurement of warmed-over flavour in reheated meat. Nielsen et al. used a fibre coated with a stationary phase of 100 μm polydimethylsiloxane (PDMS), whereas Brunton et al. compared three fibre coatings: 65 μm Carboxen–PDMS, 75 μm PDMS–divinylbenzene (DVB) and 65 μm Carbowax–DVB. Both sets of workers found that there was a high correlation between their SPME results and lipid oxidation measured as 2-thiobarbituric acid reactive substances (TBARS).

Ruiz et al. [3] used the 100 μm PDMS fibre to examine the volatile components of 2 g of sliced, dry-cured Iberian ham. Samples were extracted at 40°C and 60°C, for 20, 40 and 60 min at each temperature. Although most of the volatile compounds were present at highest amounts in the sample heated at 60°C for 60 min, major differences were only observed in the highest-boiling compounds. These compounds increased by up to 1000-

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fold, compared with their amounts in the ham samples heated at 40°C for 20 min.

Sen et al. [4] used an 85 μm polyacrylate fibre to measure levels of nitrosamines in cooked hams. Samples were steam-distilled and SPME was carried out above the headspace of the distillate. Elmore et al. [5] examined the volatile compounds in 30 g of chopped, cooked beef by SPME, using a 75 μm Carboxen–PDMS fibre. Samples were extracted at 60°C for 30 min. Almost 100 compounds were identified in the SPME extracts, 4 of which had never been previously reported in cooked beef. The authors noted that important Maillard reaction intermediates, such as furaneol and cyclotene, which are difficult to isolate by other aroma extraction techniques because of their relatively high polarity, were readily extracted by SPME.

A new stationary phase was introduced in 1999: 50/30 μm DVB–Carboxen on PDMS. This fibre comprises a layer of PDMS–DVB over a layer of Carboxen–PDMS [6]. The new fibre was suggested to be suitable for flavours in the molecular mass range 40–275, whereas the 75 μm Carboxen–PDMS fibre was suggested for the analysis of gases and low-molecular mass compounds (molecular mass 30–225). As part of our ongoing research into meat flavour, we evaluated the 1 cm Stable-flex SPME fibre coated with 50/30 μm DVB–Carboxen on PDMS. The Stable-flex fibre is more flexible and less breakable than previous fibres. Its coating is bonded to its fused-silica core, resulting in a more stable coating.

Preliminary analysis of cooked pork muscle by SPME, followed by gas chromatography–mass spectrometry (GC–MS) showed that the two stationary phases gave very different gas chromatographic profiles. Low-boiling aroma compounds predominated in the chromatogram of the aroma compounds desorbed from the 75 μm Carboxen–PDMS fibre, whereas the chromatogram of the aroma compounds desorbed from the fibre coated with 50/30 μm DVB–Carboxen on PDMS contained relatively high levels of higher-boiling aroma compounds.

By slight modification of the extraction vessel, the sample could be extracted with both types of fibre at the same time. After extraction, the volatile compounds on the SPME fibre are desorbed in the split/splitless injection port of the gas chromatograph for 3 min, onto the front of the gas chromatography column. The front of the column is cooled with solid carbon dioxide, to allow cryofocusing of the low-boiling compounds, resulting in sharp chromatographic peaks. By maintaining the injector in splitless mode for 6 min, the contents of one fibre can be desorbed, immediately followed by the contents of the other, allowing all of the extracted volatile compounds to be present in one chromatogram.

This paper shows how such a method can be used to determine a more complete range of volatile compounds in cooked pork muscle, although the method is applicable to any food. The gas chromatographic data obtained using the two fibres together is compared with the data obtained from each of the fibres used individually.

2. Materials and methods

2.1. Cooking of pork samples

Pork chops were obtained from a local supermarket. The major muscle in the chop (*M. longissimus lumborum*) was trimmed of all visible fat. The muscle was cut into two rectangular pieces with a combined mass of 30 ± 0.1 g. The pieces were placed in a 100 ml Duran borosilicate glass reagent bottle (Merck, Poole, UK), fitted with an air-tight, PTFE-lined screw top, and cooked in an autoclave at 140°C for 30 min. After cooling to ambient temperature, the cooked meat was analysed immediately.

2.2. Solid phase microextraction

Two SPME devices (Supelco UK, Poole) were used: one contained a fused-silica fibre coated with a 75 μm layer of Carboxen–PDMS, the second contained a 1 cm Stable-flex fibre coated with 50/30 μm DVB–Carboxen on PDMS. Both fibres were conditioned before use by heating them in a gas chromatograph injection port at 250°C for 30 min.

For each SPME analysis, the screw top used during the cooking of the pork sample was replaced with a similar top containing one or two drilled holes

of 3 mm radius, depending on whether one or two fibres were being used in the analysis. The PTFE lining was removed before the holes were drilled and then replaced. The lining was pierced directly beneath the drilled holes by a hypodermic needle, so that the needles of the SPME devices would not be damaged when they penetrated the lining. If the screw top had one hole it was located in the centre of the cap and if it had two holes they were both halfway between the centre and the edge of the cap, so that the holes and the centre of the cap formed a straight line.

Extractions were carried out with either the Carboxen–PDMS fibre, or the DVB–Carboxen on PDMS fibre, or both fibres simultaneously. Four pork samples were analysed using each configuration. The stainless steel needle, housing the fibre, was placed through the hole and penetrated the liner. After equilibration at 60°C for 5 min, the fibre was exposed to the headspace above the sample for 30 min. After extraction the SPME device was removed from the sample bottle and inserted into the injection port of the GC–MS system.

2.3. Gas chromatography–mass spectrometry

All analyses were performed on a Hewlett-Packard (Palo Alto, CA, USA) 5972 mass spectrometer, coupled to a 5890 Series II gas chromatograph and a G1034C Chemstation.

The volatile compounds on each SPME fibre were desorbed for 3 min in a split/splitless injection port, held at 250°C, onto a non-polar deactivated fused-silica retention gap (5 m×0.25 mm I.D.; Varian Chrompack, Middelburg, Netherlands). The retention gap contained 5 small loops in a coil, which were cooled in solid carbon dioxide, contained within a 250 ml beaker. The retention gap was attached to a CP–Sil 8 CB low bleed/MS fused-silica capillary column (5% phenyl/95% PDMS; 60 m×0.25 mm I.D., 0.25 μm film thickness; Varian Chrompack). The injection port was in splitless mode, the splitter opening after 3 min. Immediately before the desorption of the fibre, 0.1 μl of an internal standard (100 ng μl^{-1} 1,2-dichlorobenzene in methanol) were injected into the gas chromatograph. When two fibres were injected, both were desorbed for 3 min and the second fibre was desorbed immediately after the first. Therefore, the splitter was closed for 6 min in this case. Because higher levels of low-boiling volatiles were extracted using the 75 μm Carboxen–PDMS fibre, this fibre was always desorbed second. This ensured that low-boiling compounds were held on the retention gap for as short a time as possible, minimising peak distortion due to the low retention of these compounds by the GC column.

During desorption the oven was at 40°C. After desorption, the beaker containing the solid carbon dioxide was immediately removed from the oven. The oven was maintained at 40°C for a further 2 min and then the temperature was raised at 4°C min^{-1} to 280°C. Helium at 16 p.s.i. was used as the carrier gas, resulting in a flow of 1.0 ml min^{-1} at 40°C (1 p.s.i.=6894.76 Pa). *n*-Alkanes (C_5–C_{25}) were run under the same conditions to obtain linear retention index (LRI) values for the components.

The mass spectrometer was operated in electron impact mode with an electron energy of 70 eV and an emission current of 50 μA. The ion source was maintained at 170°C. The mass spectrometer scanned from m/z 29 to m/z 400 at 1.9 scans/s. Compounds were identified by first comparing their mass spectra with those contained in the National Institute of Standards and Technology (NIST)/US Environmental Protection Agency (EPA)/National Institute of Health (NIH) Mass Spectral Database or in previously published literature, followed by comparing LRI values with either those of authentic standards or published values.

Approximate quantities of the volatiles were estimated by comparison of their peak areas with that of the 1,2-dichlorobenzene internal standard, obtained from the total ion chromatograms, using a response factor of 1.

2.4. Statistical analysis

Analysis of variance (ANOVA) was carried out on the quantitative data for each compound identified in the GC–MS analyses. For those compounds exhibiting significant difference in the ANOVA, Fisher's least significant difference test was applied to determine which sample means differed significantly ($P<0.05$).

Table 1
Volatile compounds present in the aroma extracts of boiled pork, using solid-phase microextraction with two different stationary phases, separately and combined

Compound [m/z (rel. intensity)]	Mean concentration in headspace (ng/100 g)[a]			P[c]	LRI[b]	Method of identification[d]
	Carboxen PDMS	DVB Carboxen on PDMS	Both fibres			
Alkanes						
Heptane	9 (8)	–	6 (3)		699	MS+LRI
Toluene	25 (8)c	1 (0)a	11 (7)b	**	771	MS+LRI
Octane[e]	50 (33)	tr	27 (17)		800	MS+LRI
1,3-Octadiene[e]	11 (6)	–	11 (10)		827	ms
Styrene[e]	21 (10)	3 (1)	14 (9)	NS	898	MS+LRI
Decane	9 (3)	tr	6 (2)		1000	MS+LRI
Undecane	10 (4)	tr	6 (2)		1100	MS+LRI
Pentadecane	7 (6)	9 (4)	11 (4)	NS	1499	MS+LRI
Aldehydes						
3-Methylbutanal	65 (57)	2 (4)	15 (9)	NS	656	MS+LRI
2-Methylbutanal[e]	50 (52)	1 (2)	13 (6)	NS	665	MS+LRI
Pentanal	27 (9)b	1 (2)a	19 (9)b	*	709	MS+LRI
Hexanal	368 (274)	18 (10)	227 (144)	NS	804	MS+LRI
Heptanal	194 (130)	12 (5)	121 (45)	NS	905	MS+LRI
Benzaldehyde	369 (135)b	131 (43)a	295 (69)b	**	973	MS+LRI
Octanal	409 (294)	41 (16)	255 (84)	NS	1006	MS+LRI
5-Ethyl-1-formylcyclopentene	12 (12)	tr	8 (6)		1040	ms [9]
Benzeneacetaldehyde[e]	11 (8)	4 (2)	11 (12)	NS	1054	MS+LRI
(*E*)-2-Octenal	38 (48)	6 (1)	18 (11)	NS	1063	MS+LRI
Nonanal	724 (572)	184 (59)	544 (165)	NS	1108	MS+LRI
(*E*)-2-Nonenal	23 (22)	9 (2)	20 (13)	NS	1165	MS+LRI
Decanal	53 (45)	32 (8)	59 (37)	NS	1209	MS+LRI
(*E*)-2-Decenal	39 (44)	44 (21)	40 (20)	NS	1268	MS+LRI
Undecanal	19 (22)	23 (5)	23 (19)	NS	1311	MS+LRI
(*E*,*E*)-2,4-Decadienal	11 (10)	21 (10)	12 (4)	NS	1327	MS+LRI
(*E*)-2-Undecenal	34 (31)	62 (37)	43 (18)	NS	1371	MS+LRI
Dodecanal	6 (8)a	22 (6)b	16 (8)b	*	1414	MS+LRI
Tridecanal	–	8 (5)	7 (5)		1516	MS+LRI
Tetradecanal	–	12 (13)	14 (13)		1619	MS+LRI
Pentadecanal[e]	–	23 (14)	18 (19)		1721	ms+lri
Hexadecanal	31 (42)	361 (243)	245 (297)	NS	1825	ms+lri
Alcohols						
1-Pentanol	16 (4)c	1 (0)a	9 (5)b	**	768	MS+LRI
1-Heptanol	27 (19)	3 (1)	13 (7)	NS	971	MS+LRI
1-Octen-3-ol	57 (48)	6 (2)	32 (19)	NS	983	MS+LRI
2-(2-Ethoxyethoxy)ethanol[e]	tr	22 (18)	12 (11)		1001	MS+LRI
1-Octanol	62 (46)	14 (6)	36 (4)	NS	1072	MS+LRI
Ketones and Hydroxyketones						
Acetone	17 (10)	7 (2)	20 (12)	NS	500	MS+LRI
2,3-Butanedione	9 (4)	1 (1)	6 (3)	NS	595	MS+LRI
2-Butanone[e]	41 (28)b	3 (1)a	12 (3)a	*	602	MS+LRI
1-Hydroxy-2-propanone[e]	71 (17)b	11 (5)a	24 (11)a	**	674	MS+LRI

Table 1. Continued

Compound [m/z (rel. intensity)]	Mean concentration in headspace (ng/100 g)[a]			P[c]	LRI[b]	Method of identification[d]
	Carboxen PDMS	DVB Carboxen on PDMS	Both fibres			
2-Pentanone[e]	17 (16)	–	4 (3)		693	MS+LRI
2,3-Pentanedione[e]	57 (23)b	3 (1)a	28 (20)a	*	703	MS+LRI
3-Pentanone[e]	15 (16)	–	3 (2)		703	MS+LRI
3-Hydroxy-2-butanone	7 (5)	tr	2 (0)		717	MS+LRI
Acetoxyacetone[e]	17 (5)c	1 (0)a	6 (2)b	***	867	ms
2-Heptanone	41 (15)b	2 (0)a	23 (16)b	*	891	MS+LRI
2,3-Octanedione	15 (18)	tr	8 (5)		986	ms+lri
2-Octanone	11 (6)b	1 (0)a	6 (2)b	*	992	MS+LRI
2-Nonanone	15 (10)	2 (1)	10 (3)	NS	1092	MS+LRI
2-Decanone[e]	15 (13)	6 (2)	12 (2)	NS	1193	MS+LRI
2-Pentadecanone	3 (3)	11 (6)	14 (10)	NS	1702	MS+LRI
Furans						
2-Methylfuran[e]	20 (11)	2 (1)	13 (13)	NS	602	MS+LRI
Tetrahydrofuran[e]	2 (2)a	12 (7)b	12 (4)b	*	629	MS+LRI
2-Ethylfuran[e]	37 (12)	2 (1)	27 (24)	NS	702	MS+LRI
Dihydro-2-methyl-3(2*H*)-furanone	22 (21)	tr	–		812	MS+LRI
2-Furfural	91 (42)	23 (13)	83 (54)	NS	837	MS+LRI
2-Furanmethanol	57 (32)	10 (1)	23 (12)	NS	856	MS+LRI
2-Butylfuran[e]	19 (11)	tr	15 (12)		894	MS+LRI
5-Methylfurfural[e]	11 (3)	3 (2)	8 (4)	NS	967	MS+LRI
2-Pentylfuran	959 (564)	100 (26)	766 (338)	NS	994	MS+LRI
(*E*)- *or* (*Z*)-2-(2-Pentenyl)furan[e]	17 (12)	2 (1)	11 (7)	NS	1000	ms [10]
(*E*)- *or* (*Z*)-2-(2-Pentenyl)furan[e]	66 (46)	14 (6.7)	52 (31)	NS	1002	ms [10]
A dimethylfurfural[e]	17 (7)	6 (4)	13 (5)	NS	1050	ms [11]
(*E*)- *or* (*Z*)-2-(1-Pentenyl)furan[e]	18 (10)	–	15 (4)		1058	ms
A methylpentylfuran[e]	13 (9)	2 (0)	10 (7)	NS	1083	ms [12]
2-Hexylfuran[e]	17 (12)	3 (1)	13 (6)	NS	1094	MS+LRI
An ethylmethylfurfural[e]	18 (9)	10 (5)	14 (4)	NS	1145	ms [11]
2-Heptylfuran[e]	31 (27)	10 (4)	25 (11)	NS	1195	MS+LRI
3-Phenylfuran[e]	3 (1)	7 (2)	5 (3)	NS	1238	ms
2-Octylfuran[e]	83 (86)	67 (49)	102 (36)	NS	1297	MS+LRI
Nitrogen-containing						
Pyrazine[e]	23 (11)b	2 (1)a	10 (6)a	*	739	MS+LRI
Methylpyrazine	129 (29)b	15 (9)a	74 (50)b	*	829	MS+LRI
2,5 *and* 2,6-Dimethylpyrazine	106 (31)c	11 (6)a	58 (39)b	*	917	MS+LRI
Ethylpyrazine[e]	30 (6)b	2 (2)a	18 (13)b	*	921	MS+LRI
2-Ethyl-6-methylpyrazine	40 (13)c	5 (3)a	22 (11)b	*	1002	MS+LRI
Trimethylpyrazine	13 (5)c	2 (1)a	7 (3)b	**	1007	MS+LRI
2-Ethyl-5-methylpyrazine	13 (6)b	3 (1)a	8 (4)b	**	1008	MS+LRI
2-Acetylpyrrole[e]	8 (4)	3 (1)	6 (3)	NS	1068	MS+LRI
3-Ethyl-2,5-dimethylpyrazine	13 (6)c	3 (1)a	8 (4)b	*	1081	MS+LRI
Sulfur-containing						
Sulfur dioxide[e]	17 (18)	25 (40)	136 (105)	NS	<500	ms
Methanethiol	11 (4)	15 (14)	39 (20)	NS	<500	ms
Carbon disulfide[e]	7 (2)	53 (54)	51 (32)	NS	538	MS+LRI
Thiophene	8 (4)	tr	4 (3)		671	MS+LRI
Dimethyl disulfide	27 (25)	5 (3)	14 (5)	NS	748	MS+LRI

Table 1. Continued

Compound [*m*/*z* (rel. intensity)]	Mean concentration in headspace (ng/100 g)[a]			P[c]	LRI[b]	Method of identification[d]
	Carboxen PDMS	DVB Carboxen on PDMS	Both fibres			
2-Methylthiophene	80 (62)b	2 (1)a	29 (26)a	*	776	MS+LRI
Dimethyl trisulfide	88 (63)	35 (15)	82 (41)	NS	982	MS+LRI
5-Methyl-3(2*H*)-dihydrothiophenone	8 (7)	tr	3 (1)		996	ms [13]
2-Thiophenealdehyde	26 (8)c	8 (5)a	17 (10)b	**	1012	MS+LRI
2-Acetylthiazole	10 (2)	3 (2)	9 (9)	NS	1027	MS+LRI
2-Methyl-3-formylthiophene[e]	19 (8)c	6 (3)a	13 (6)b	*	1097	MS+LRI [7]
2-Pentylthiophene	15 (14)	4 (1)	13 (7)	NS	1170	MS+LRI
2,3-Dihydro-6-methylthieno-2,3*c*-furan	21 (6)	10 (3)	19 (13)	NS	1204	MS+LRI
Dimethyl tetrasulfide	3 (3)	7 (4)	9 (6)	NS	1241	ms+lri
Dihydromethylthienothiophene[e]	6 (4)	7 (3)	9 (4)	NS	1392	ms [12]
Miscellaneous and unknown						
Acetic acid	26 (32)	171 (177)	88 (62)	NS	606	MS+LRI
A tridecatetraene[e]	7 (8)	15 (3)	11 (5)	NS	1300	se
79(100), 91(69), 108(59), 81(49), 120(46), 77(31)		22 (27)	6 (5)		1422	
134(100), 69(53), 58(13), 136(12), 70(12), 45(12)	–					

[a] Means for the same breed with different letters (a, b, c) are significantly different ($P<0.05$); means are from four replicate samples; –, less than 1 ng in the headspace of 100 g of cooked lamb.

[b] Linear retention index on a CP–Sil 8 CB low bleed/MS column.

[c] Probability that there is a difference between samples; NS, no significant difference between means ($P>0.05$); * significant at the 5% level; ** significant at the 1% level; *** significant at the 0.1% level.

[d] MS+LRI, mass spectrum and LRI agree with those of authentic compound; ms+lri, mass spectrum identified using NIST/EPA/NIH Mass Spectral Database and LRI agrees with literature value [13]; ms, mass spectrum agrees with spectrum in NIST/EPA/NIH Mass Spectral Database or with other literature mass spectrum [7,9–12]; se, tentative identification from structure elucidation of mass spectrum.

[e] Reported for the first time in pork.

3. Results and discussion

Ninety-six compounds were present in the headspace at levels above 5 ng per 100 g of sample in at least one of the extracts. These compounds are listed in Table 1. Compounds present below 1 ng per 100 g of sample are labelled as trace. The detection limit for each compound was 0.2 ng per 100 g. Thirty-six of the identified compounds have not been previously reported as constituents of cooked pork aroma. However, many of these compounds have been previously reported as components of boiled beef and lamb [7,8].

The two fibres gave results that differed qualitatively (Fig. 1). Five compounds were absent and ten were at trace levels from the DVB–Carboxen on PDMS fibre. Four compounds were absent and one was at trace levels from the Carboxen–PDMS fibre. Of the compounds quantified on both types of fibre, 23 were significantly different. Of these 23 compounds, only tetrahydrofuran and dodecanal were found at higher levels on the DVB–Carboxen on PDMS fibre than the Carboxen–PDMS fibre.

Using the two fibres together resulted in a chromatogram with only one of the 95 peaks being absent and all of the peaks found at trace levels on the single fibre were found at levels above trace detection. This demonstrates that the use of the two fibres together will give more information on the aroma of boiled pork than using either separately.

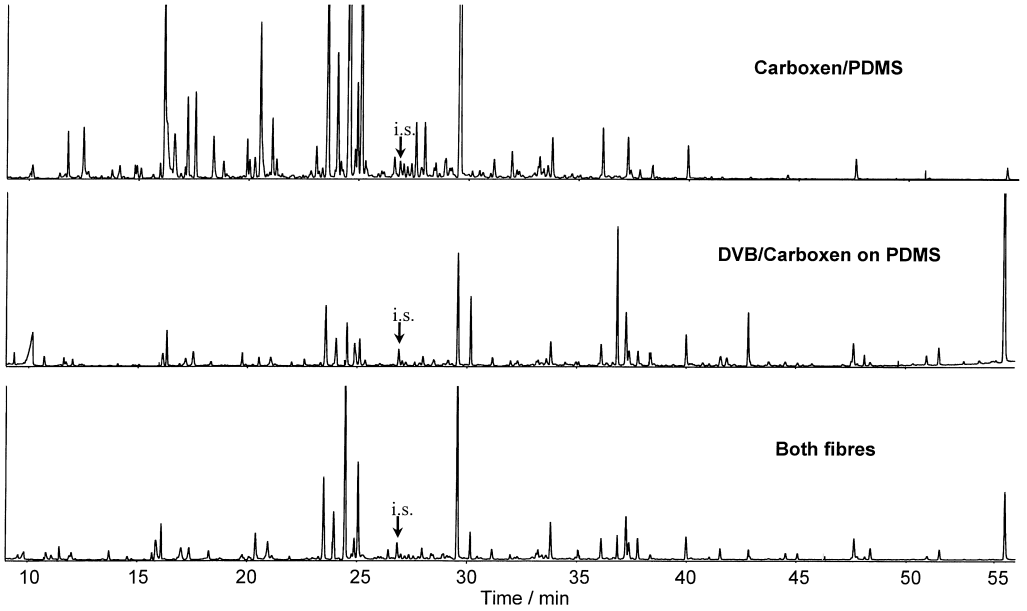

Fig. 1. Gas chromatographic traces of the aroma volatiles of cooked pork, using solid-phase microextraction with two different stationary phases, separately and combined. i.s., internal standard.

However, for most compounds, the peak area obtained when using the two fibres together was not as great as the peak area obtained with one or other of the fibres used individually. Instead, for nearly all of the compounds, the two fibres together gave a peak area that was between the values for the individual fibres. This effect was observed for 80 of the 96 peaks. The total concentration of the 96 peaks present in the extract from the Carboxen–PDMS fibre was 5.4 μg per 100 g for sample, for the DVB–Carboxen on PDMS it was 1.8 μg per 100 g of sample and when both fibres were used together the value was 4.4 μg per 100 g of sample. These results suggested that using the two fibres together altered the attainment of equilibrium between the sample, the headspace and the fibre.

Shirey [6] compared six different SPME fibres for the analysis of ten low-molecular mass aroma compounds. The fibre types included DVB–Carboxen on PDMS and Carboxen–PDMS. He showed that GC peak area responses using the Carboxen–PDMS fibre were greatest for every compound except one, isopropylamine, for which DVB–Carboxen on PDMS gave the greatest response. Shirey stated that fibres containing adsorbents, such as Carboxen and DVB, extracted more than fibres composed of a liquid stationary phase, such as PDMS or Carbowax. DVB had been shown to have a high affinity for amines but in general Carboxen was a more effective coating than DVB for low-molecular mass compounds. DVB contains relatively few micropores (2–20 Å diameter) whereas Carboxen contains relatively similar volumes of micro-, meso- (20–500 Å) and macropores (>500 Å), allowing adsorption of a wider molecular mass range.

The average RSD for all quantified peaks was measured. In all cases variability was high: 50% for the DVB–Carboxen on PDMS, 67% for Carboxen–PDMS and 57% for the two fibres together. It is likely that reproducibility would have increased if the samples had been minced to reduce surface area effects. However, this work has shown that SPME using two fibres together is an extremely simple method for yielding a large amount of data on the complete aroma profile of a relatively small amount of pork meat. The technique has been shown to be

suitable for the analysis of volatile compounds up to a molecular mass of at least 240 (hexadecanal). In fact, trace levels of octadecanal, which has a molecular mass of 268, were detected using the DVB–Carboxen on PDMS fibre.

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