SOLID PHASE MICROEXTRACTION FOR THE ISOLATION OF VOLATILE FLAVOUR COMPOUNDS IN COOKED BEEF MEAT

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Background

In the past, several methods have been developed to isolate volatile compounds from different food matrixes. Static headspace analysis, purge and trap, distillation/extraction techniques, and more recently model mouth have been used to analyse the volatile fraction of meat (see review by Machiels et al., 2000). Solid phase microextraction (SPME) is an interesting extraction technique recently developed by Arthur and Pawliszyn (1990). It has been used to determine the volatile compounds in several foods and drinks including dry cured ham (Ruiz et al., 1998) and more recently cooked pork (Elmore et al., 2000). Its use on cooked beef steaks has not been reported yet. The technique is sensitive, selective, fast, low-cost, solvent-free, easy to handle and also compatible with low detection limits. In addition, because low extraction temperature can be used, SPME could give a better estimation of the aroma profile as perceived by the human nose. However, further research is still required to achieve good extraction conditions using SPME for the analysis of cooked meat.

Objectives

The objective of this study was to investigate the aroma profile of cooked beef meat by SPME combined with gas chromatography-mass spectrometry (GC-MS). Several extraction times, desorption times, temperature conditions and fiber types were tested to achieve a fast and reproducible extraction and a representative analysis of target compounds.

Methods

Muscle (*longissimus dorsi*) from Belgian Blue bull was trimmed of subcutaneous fat, vacuum packed and stored at -18°C. Thirty five g of sample was cut in small pieces, frozen by liquid nitrogen and ground until a powder was obtained. One g of this powder was placed in a 40 ml headspace vial, sealed with PTFE/silicone septum for analysis. The meat was cooked at 150°C in a silicone bath for 20 min, then cooled to 0°C in a water/ice bath for 10 min, warmed to 60°C for 5 min. Extraction was performed using SPME fibers for 20 min at 60°C. All analyses were performed using a Varian Saturn 2000 ion-trap mass spectrometer fitted with a Varian CP 3800 gas chromatograph. The SPME fiber was thermally desorbed at 250°C in the injector port for 2 min in the splitless mode, the split valve being opened after 2 min (split ratio 10). During desorption the oven was held at 35°C. A Rtx-5MS column (60m x 0.25 mm i.d., 0.5µm film thickness, Restek, Bellefonte, PA) was used to separate the volatile components of the cooked meat. After desorption, the oven was held at 35°C for 3 min, heated to 50°C at 10°C/min, then raised to 200°C at 10°C/min and finally by 10°C to 250°C and held for 10 min at this temperature. Helium was used as carrier gas with a constant flow of 1.5 ml/min. The volatile compounds were identified by first comparing their mass spectra with those contained in the NIST 98 Mass Spectral database and those from standards. Wherever possible, identities were confirmed by comparison of retention index (RI) values with those of published values (Kondjoyan & Berdagué, 1996).

Results and discussion

The effects of different extraction times (5, 10, 15, 20, 40 min), SPME fibers (divinylbenzene-carboxen-polydimethylsiloxane and carboxen-polydimethylsiloxane) and desorption times (30 s, 1, 2, 5, 10, 20 min) were studied, resulting in an extraction time of 20 min at 60°C, and a desorption time of 2 min at 250°C for both fibers. More than 200 volatile compounds were separated and tentatively identified on a DB-5 like column. Among those, 36 volatile flavour compounds extracted by the 2 types of fiber were compared in the aroma profile of cooked beef (Table 1). These compounds were chosen because they were previously identified as key odour active compounds in cooked beef meat. The divinylbenzene-carboxen-polydimethylsiloxane (DVB-CAR-PDMS) fiber presented lower coefficients of variance for the target compounds than the carboxen-polydimethylsiloxane (CAR-PDMS) fiber. Brunton et al. (2000) also reported lower performances of the CAR-PDMS fiber. According to the large number of parameters to be controled (cooking and extraction temperature for instance) and the "natural" heterogeneity of meat, a coefficient of variance below 20% for the DVB-CAR-PDMS fiber could be considered as acceptable.

Conclusions

The extraction of more than 200 volatile flavour compounds of cooked beef meat was achieved by SPME. Thirty six key odour active compounds of cooked beef meat were evaluated by two types of SPME fibers. The DVB-CAR-PDMS fiber presented the best performance in terms of reproducibility. The use of DVB-CAR-PDMS SPME fiber for the screening of the volatile compounds of cooked beef is an attractive alternative technique to other headspace methods.

Pertinent literature

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Table 1. Volatile flavour compounds of cooked beef meat, retention times, peak area and coefficients of variance (n=10)

Retention time[a]	Compound	Peak area (TIC)	
		DVB-CAR-PDMS[b]	CAR-PDMS[c]
6.0	Methanethiol	134352	312128
8.0	Carbon disulfide	1116590	2616898
9.2	2,3-Butanedione	83045	80268
9.5	2-Butanone	177686	1189765
11.5	3-Methylbutanal	196908	1599248
11.9	2-Methylbutanal	180988	981952
13.1	2,3-Pentanedione	348509	4611225
13.2	Pentanal	168649	1830352
13.4	2-Ethylfuran	82227	1550513
15.1	Pyrazine	17256	108467
15.3	Methylpyrrole	n.d.[d]	6952
15.5	Dimethyl disulfide	24868	275048
15.8	Pyrrole	34058	144689
17.8	Hexanal + mesityl oxide	702595	7763286
19.1	Methylpyrazine	102323	352437
19.5	Furfural	107436	138373
21.9	2-Heptanone	136134	515095
22.4	Heptanal	321285	1448022
22.8	3-Methylthiopropanal	7115	23134
23.1	2,5-Dimethylpyrazine	247509	641345
25.8	2-Octen-1-ol	564304	583026
25.9	2-Methyl-3-octanone	134011	237048
26.4	2-Octanone	54231	n.d.
26.5	2-Pentylfuran	4343465	6651387
26.9	Octanal	459575	576983
27.1	Trimethylpyrazine	176492	133962
27.9	2-Acetylthiazole	43979	56443
29.0	Benzeneacetaldehyde	248008	188616
29.3	2-Octenal	299960	192667
31.1	Nonanal	2126315	1324790
33.4	2-Nonenal	183947	49016
35.5	2,4-Octadienal	n.d.	135395
39.2	2,4-Decadienal	391866	43521
41.7	Tetradecane	544287	206255
46.5	Pentadecane	404958	109184
	CV [%]	17	25

[a] On Rtx-5MS column (60m x 0.25 mm i.d., 0.5µm)
[b] Divinyl-Carboxen-Polydimethylsiloxane (50/30µm) SPME fiber
[c] Carboxen-Polydimethylsiloxane (75µm) SPME fiber
[d] Non detected