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Optimization and evaluation of traditional SPME vs SPME Arrow for qualitative analysis of meat aroma

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1. Introduction

Raw meat on its own has very little aroma, therefore almost all aromas associated with “meatiness” are created during the cooking process by the Maillard Reaction between amino acids and reducing sugars. This reaction determines which non-volatile precursors release volatile aroma compounds. As a result, both meat and meat alternatives are complex matrices to analyze by gas chromatography–mass spectrometry (GCMS) without additional sample preparation.

Solid phase microextraction (SPME) is a solvent-less extraction technique which makes use of a sorbent fiber to ad/absorb compounds from a headspace or liquid sample. Headspace-SPME improves selectivity and sensitivity for volatile compounds and reduces matrix effects. The new SPME Arrow contains a greater quantity of sorbent phase and larger surface area than a traditional fiber, allowing for greater analyte extraction in less time.

This work describes the development and optimization of a SPME-GCMS method suitable for qualitative analysis of cooked-meat aroma, and a comparison of several SPME fibers and the new SPME Arrow.

2. Experimental Methods

Beef samples were prepared as follows: approximately 2.5 g of organic ground beef (85 lean: 15 fat) were weighed into glass, crimp-top, 20 mL standard headspace vials and left at ambient temperature prior to analysis. Samples were run in triplicate on each type of SPME device. Identification of detected peaks was performed with the Wiley 12th edition/NIST 2017 library.

GCMS-QP2020 NX with AOC-6000	
SPME & SPME Arrow	PAL: PDMS coating
Extraction	130 °C, varied times
Desorption	10 min
Gas Chromatography	
Injection Port	270 °C, splitless (1 min); split 10:1
Column	Rtx-5MS column (30 m × 0.25 mm × 0.25 µm) He carrier gas; Constant Pressure, 90.1 kPa
Oven Temperature	60 °C - 2 min > 160 °C (7 °C/sec) > 250 °C (4 °C/sec) - 2 min
Mass Spectrometry	
Interface Temperature	250 °C
Ion Source Temperature	200 °C
Scan Range	40 to 350 m/z
Event Time	0.3 sec

3. Results

3.1 Conventional SPME fiber compared to new SPME Arrow

We first assessed the difference between a conventional SPME fiber and the new SPME Arrow. As expected, SPME Arrow absorbs more compounds over the same length extraction compared to the SPME fiber, resulting in more detectable peaks on the chromatogram. The Arrow has 20x the surface area of the fiber, and therefore has far more sorbent sites for analytes.

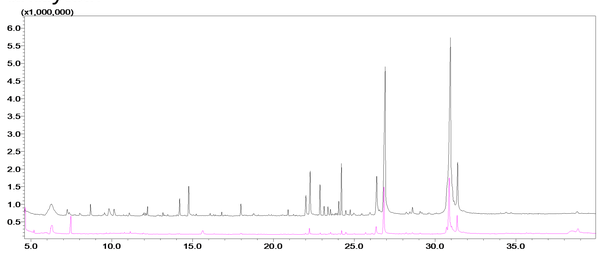

Figure 1. Overlaid representative chromatograms for organic beef, 10 min extractions, SPME Arrow (black) and SPME fiber (pink)

3.2 Extraction time optimization

Multiple extraction times were compared to investigate if throughput could improve with increased surface area on the Arrow. We observed little change between 3, 10, and 30 min extractions with the traditional SPME fiber (Figure 2); however with the SPME Arrow, a significant increase in signal was observed between each increase in extraction time (Figure 3). The SPME Arrow absorbs approximately the same quantity of compounds from a 3 min extraction as the SPME fiber does in 30 min. Increasing the extraction time for the SPME Arrow increases not only signal intensity but also the number of detectable compounds.

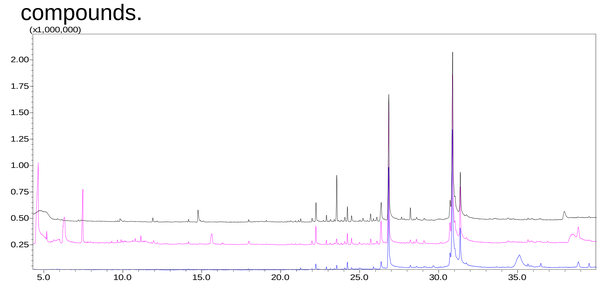

Figure 2. Overlaid chromatograms for organic beef, 30 min (black), 10 min (pink), and 3 min (blue) extractions, SPME fiber

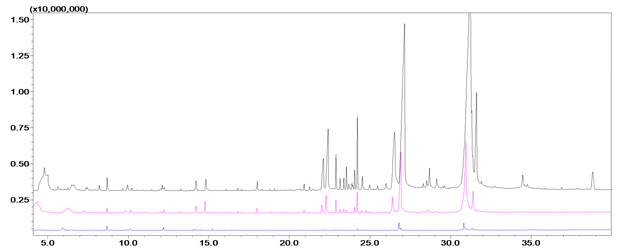

Figure 3. Overlaid chromatograms for organic beef, 30 min (black), 10 min (pink), and 3 min (blue) extractions, SPME Arrow

3.3 Organic beef aroma compared to plant-based meat aroma

Similar compounds were found in both types of meat (see table below), such as fatty acids and Maillard browning products. This is not surprising, since almost all meat aroma comes from the cooking process, and the samples were heated under identical conditions. The differences can be explained by the different and wide variety of precursors present in imitation meat, since it contains amino acids and sugars from different sources than regular meat.

Imitation Meat	Organic Beef
1,3-Propanediol	Propanoic acid, 2-hydroxy-, methyl ester, (.+/-.)-
Pentaethylene glycol	Dimethyl sulfone
2(5H)-Furanone	
Glycerin	Glycerin
Furaneol	3-Pentanone, 2,4-dimethyl-
3,5-Octadien-2-one, (E,E)-	Hexyl n-valerate
Nonanal	Nonanal
Maltol	
4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-	4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-
2(3H)-Furanone, dihydro-4-hydroxy-	2(3H)-Furanone, dihydro-4-hydroxy-
Octanoic acid	Octanoic acid
Caprolactam	Thiophene, 2,3-dihydro-
2-Decenal, (E)-	Piperidine, 1-nitroso-
Nonanoic acid	Nonanoic acid
2-n-Octylfuran	1,2-Benzenediol, 3,5-bis(1,1-dimethylethyl)-
2,4-Decadienal, (E,E)-	
cis-4-Decenal	
n-Decanoic acid	n-Decanoic acid
2-Tridecanone	Niacinamide
Tetradecane	6,10-Dodecadien-1-ol, 3,7,11-trimethyl-
Thiazole, 4,5-dimethyl-	2-Tridecanone
n-Nonylcyclohexane	
Dodecanoic acid	Dodecanoic acid
1-Pentadecyne	Phosphonofluoridic acid, (1-methylethyl)-, cyclohexyl ester
8-Heptadecene	Eicosane
2-Dodecanone	1-Hexadecanol
	Methanone, (1-hydroxycyclohexyl)phenyl-
	Hexadecenoic acid, Z-11-
Tetradecanoic acid	Tetradecanoic acid
Tetradecanoic acid, ethyl ester	1-Dodecanol, 3,7,11-trimethyl-
	Octadecane
	Hexadecane, 2,6,10,14-tetramethyl-
	Tetradecanal
	Pentadecanal-
	Pentadecanoic acid
	2-Heptadecanone
	.delta.-Dodecalactone
Erucic acid	Erucic acid
n-Hexadecanoic acid	n-Hexadecanoic acid
	Heptadecanoic acid
	2(3H)-Furanone, 5-dodecyldihydro-
Oleic Acid	Oleic Acid
Octadecanoic acid	Octadecanoic acid
Hexadecanamide	8,11,14-Eicosatrienoic acid, (Z,Z,Z)-
	Squalene

4. Conclusion

The GCMS-QP2020 NX equipped with an AOC-6000 autosampler was employed for SPME and SPME Arrow analyses of organic beef and imitation meat. Qualitative results from the Wiley library search showed that SPME Arrow can absorb greater quantities of a wider range of compounds than conventional SPME, improving both sensitivity and throughput. Future work could focus on method development to target specific compound classes or increase sensitivity for quality marker odorants to further improve imitation meat quality.