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Application News

No. GCMS-2101

Gas Chromatography Mass Spectrometry

Optimization and Evaluation of Traditional SPME and SPME Arrow for Qualitative Analysis of Meat Aroma

■ Abstract

Plant-based meat has become more popular as public interest in sustainable protein sources increases, as it closely resembles the flavor and texture of several kinds of meat products. Because aroma is responsible for most of what we know as flavor, in comparing plant-based meat with regular meat products, looking at volatile odorants is critical. To do so, we employed headspace-solid phase microextraction fibers to absorb odorants and subsequently analyzed them by gas chromatography–mass spectrometry. Several fiber coatings and the new Arrow format were compared, and differences were found in sensitivity and compound absorption. Detected compounds were easily identified with the Wiley 12th edition/NIST 2017 library.

■ Introduction

Raw meat on its own has 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. That reaction determines which non-volatile precursors release volatile aroma compounds. Plant-based meats, products created to resemble animal meat in both look and taste, are growing in popularity. They provide valuable nutrition while reducing certain health risks, such as heart disease and type II diabetes. In addition, they provide the most environmentally sustainable source of food, since their carbon and water footprints are smaller than animal-based foods. A plant protein such as soy protein concentrate, along with colors, stabilizers, and oils, is used to successfully mimic meat flavor and texture. And, just like in animal meat, the amino acids of that protein undergo the Maillard Reaction. As a result, both meat and meat alternatives are complex matrices to analyze by gas chromatography–mass spectrometry (GC-MS) 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. Combined with a redesigned tip and outer sheath, both throughput and robustness are increased over conventional SPME analyses.

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. The optimized method is used to profile the aroma of several kinds of cooked-meat substitutes and is then compared against the regular cooked-meat aroma.

■ Samples and Analytical Conditions/Experimental

Instrument Configuration

Table 1 summarizes the instrument conditions used on the Shimadzu GCMS-QP2020 NX equipped with an AOC-6000 autosampler throughout this work. 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.

Table 1: GCMS Conditions

GCMS-QP2020 NX with AOC-6000	
SPME & SPME Arrow	**PAL: PDMS, Carboxen, and PDMS/DVB/Carboxen Coatings**
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
Detector Voltage	Relative to Tune
Scan Range	40 to 350 m/z
Event Time	0.3 seconds

■ Results and Discussion

Chromatography

We first assessed the difference between a conventional SPME fiber and the new SPME Arrow. As expected, Figure 1 demonstrates that the 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.

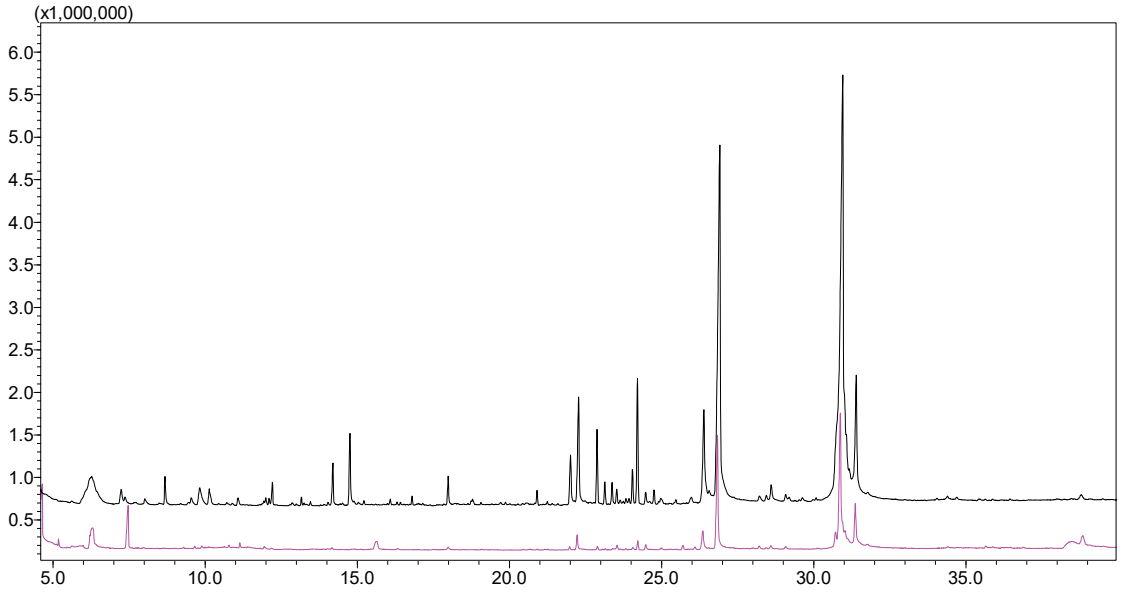

Figure 1: Overlaid representative chromatograms for organic beef, 10 min extractions, SPME Arrow (black) and SPME fiber (pink)

Multiple extraction times were compared to investigate if throughput can be improved due to increased surface area on the Arrow. We observed minimal 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).

Even at a 3 min extraction, the SPME Arrow absorbs approximately the same quantity of compounds as the SPME fiber does in a 30 min extraction. Increasing the extraction time for the SPME Arrow increases not only signal intensity but also the number of detectable compounds.

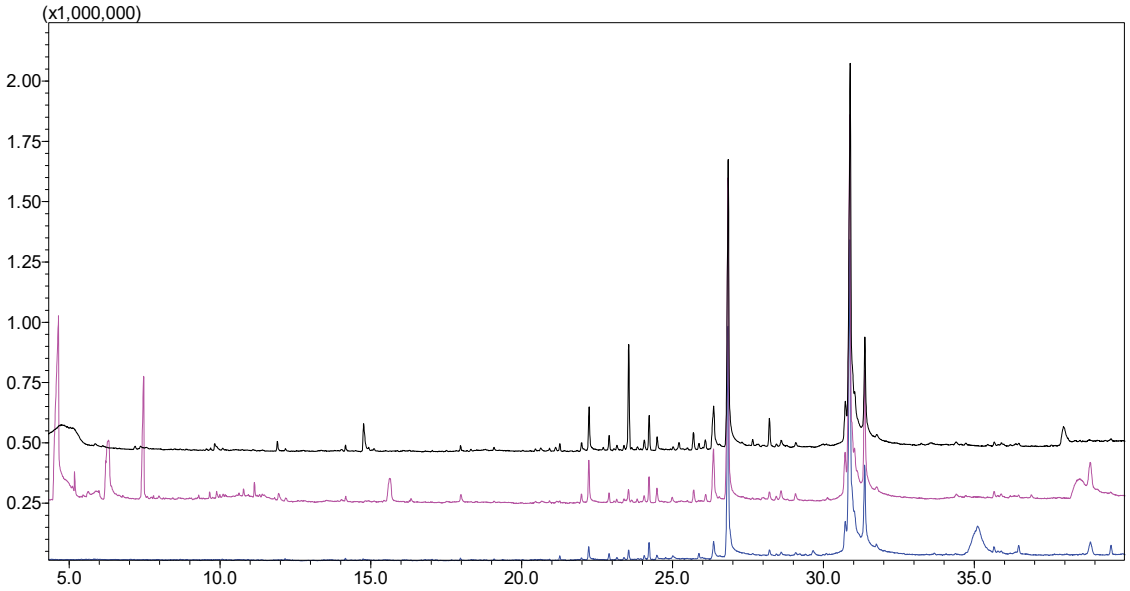

Figure 2: Overlaid representative chromatograms for organic beef, 30 min (black), 10 min (pink), and 3 min (blue) extraction times with the SPME fiber.

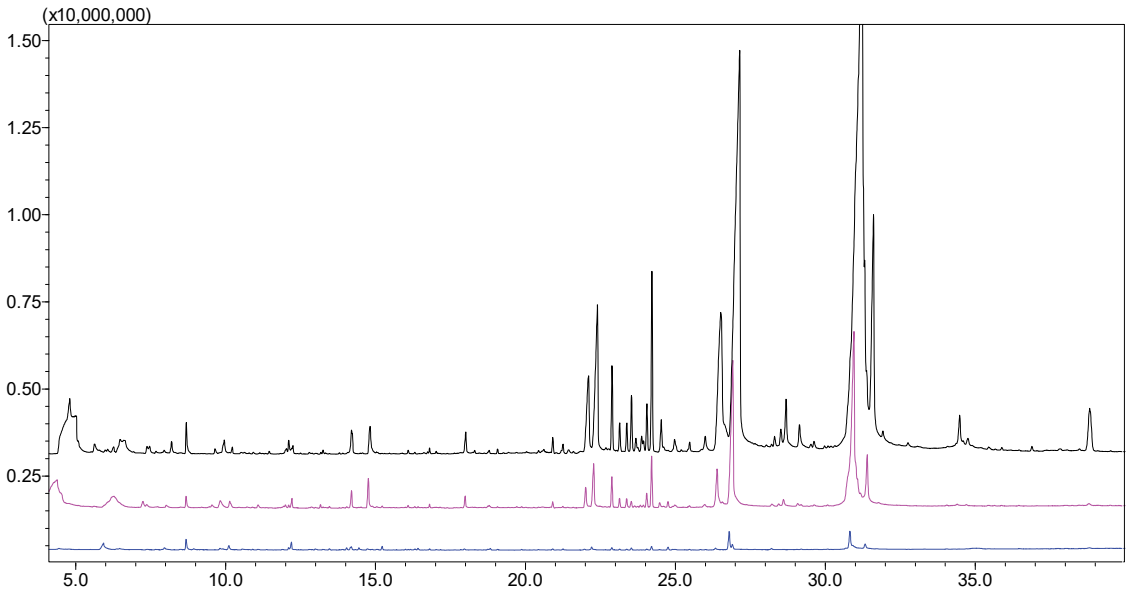

Figure 3: Overlaid representative chromatograms for organic beef, 30 min (black), 10 min (pink), and 3 min (blue) extraction times with the SPME Arrow.

Samples of imitation meat were run with the SPME Arrow (Figure 4), and the volatile profile was compared against that of the organic beef. Similar compounds were found in both types of meat (Table 2), such as fatty acids and Maillard browning reaction 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 various sources than regular meat.

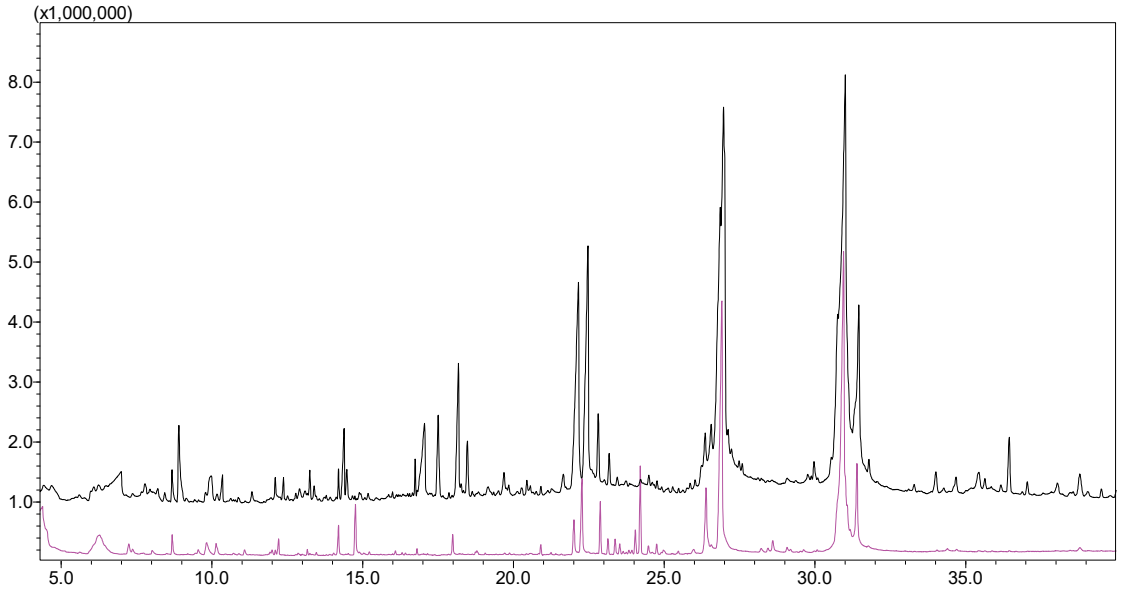

Figure 4: Overlaid representative chromatograms for imitation meat (black) and organic beef (pink) at 10 min extractions with the SPME Arrow.

Table 2: Compounds detected in imitation meat and organic beef samples with the SPME Arrow.

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

Imitation Meat	*Organic Beef*
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

Whether using the SPME Arrow or SPME fiber, the coating chemistry will be a significant indicator of what compounds can be absorbed. PDMS (Figure 5) can absorb a different range of compounds than Carboxen (Figure 6).

Furthermore, combining phases such as PDMS/DVB/Carboxen will have the greatest impact on the range of compounds that can be introduced to the GCMS (Figure 7), regardless of regular SPME vs Arrow.

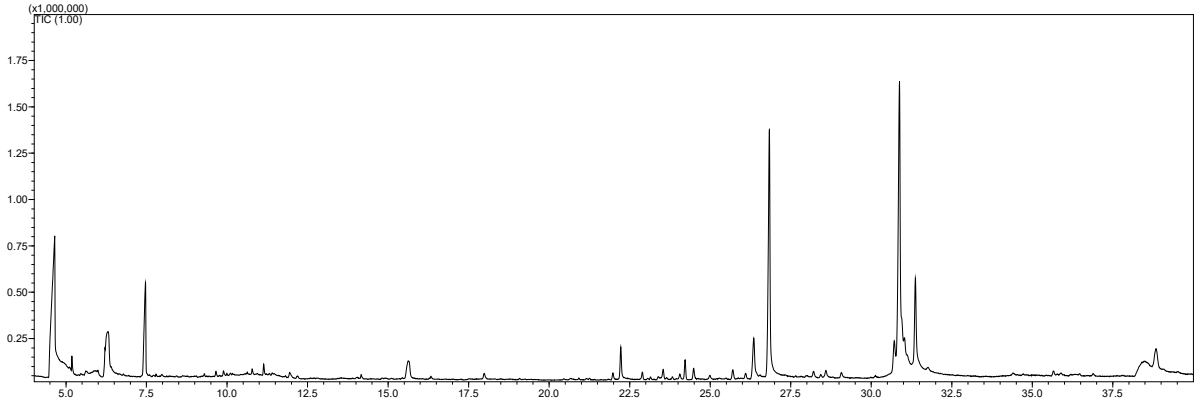

Figure 5: Representative chromatogram for organic beef with PDMS fiber

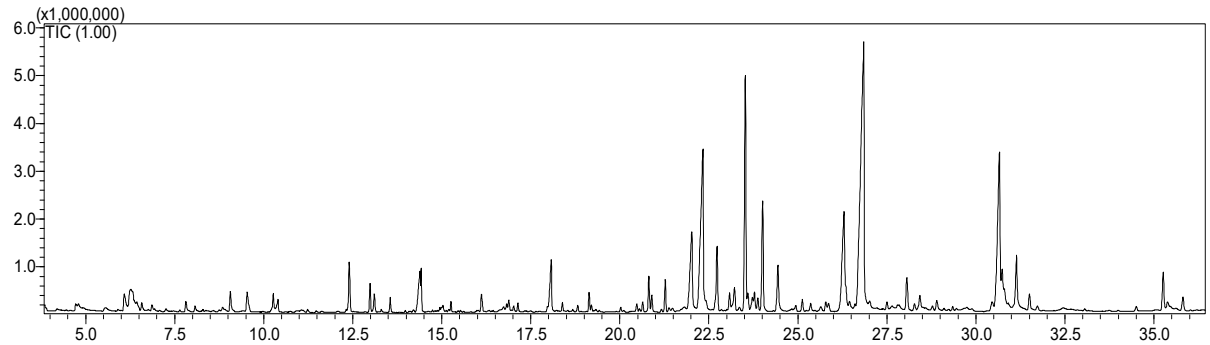

Figure 6: Representative chromatogram for organic beef with Carboxen fiber

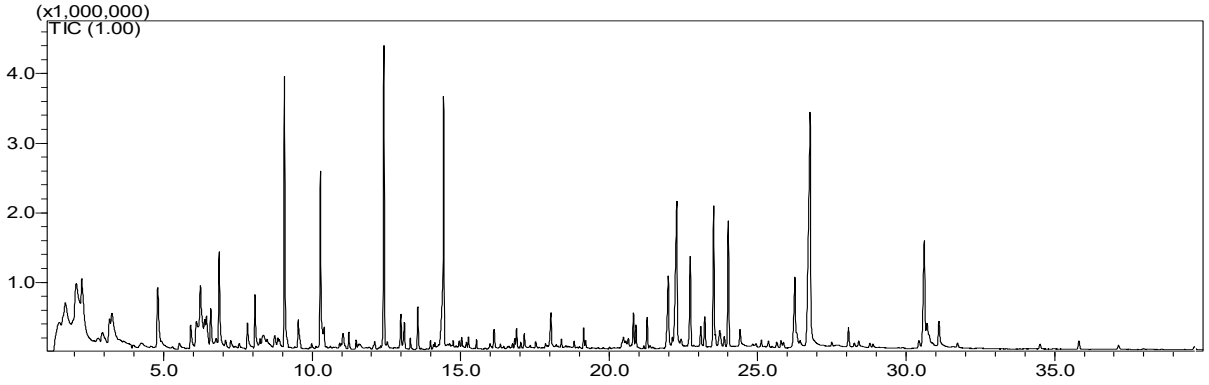

Figure 7: Representative chromatogram for organic beef with PDMS/DVB/Carboxen fiber

■ 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.

By comparing different SPME fiber coatings, we demonstrate that the compounds detected by either SPME technique is largely dependent on the fiber coating material. 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.

■ Consumables

Part number	Description
221-75855-30	Capillary column
REST-23279	Splitless inlet liner
220-97331-08	Vials
220-94906-31	Caps
REST-27485	PDMS SPME Arrow
REST-27480	PDMS SPME fiber
REST-27491	SPME Arrow injection port conversion kit

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