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Comparison of solid-phase micro-extraction methods for beef volatile analysis (#634)

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Introduction

Volatile analyses have been conducted previously for cooked beef samples using a manual solid-phase micro-extraction (SPME) collection method [Farmer et al., unpublished data, 1]. The variation between replicate analyses has often been high. Automatic SPME methods are available and have been evaluated for raw beef [2] but their applicability to cooked beef is more difficult, due to the need for a consistent and representative cooking method. This study compares automatic SPME collection methods for grilled beef volatile analysis.

Methods

Three methods (method *A, B, C*) were selected to collect headspace volatile compounds from beef samples (Figure 1). Beef samples were grilled to 65°C internal temperature using a clam grill (S-143, SILEXIA UK. Ltd, York, United Kingdom).The cooked beef samples were cored (1.27cm diameter) for method *A* while samples for methods *B* and *C* were immersed in liquid nitrogen for 5 mins and homogenised. Beef sample (2±0.1g) was transferred into a glass vial and placed on gas chromatography-mass spectrometry (Agilent 5977B MSD/7890B GC). Following 5 mins of equilibration in 65°C agitator, the headspace volatiles were collected using an automated SPME injection system (Gerstel Multi Purpose Sampler Robotic Pro with SPME injection tool holder) equipped with SPME fibre (Supelco, Bellefonte, PA, USA). 75µm CAR/PDMS SPME fibre was used in method *A* and *B* while 50/30µm DVB/CAR/ PDMS SPME fibre was used in method *C*.

The quantities of individual volatile compounds was quantified using a MassHunter integration method and known injection quantities of the authentic compounds, based on one quantification ion and three target ions. Total volatile quantities were calculated by adding all identified volatile compounds. Volatile compounds were categorised into groups for the purpose of this paper. Means, standard deviations and coefficients of variation (CV) for six replicate analyses for each of the volatile groups were calculated, for each method and each ageing period. Average CVs for the five ageing periods were calculated.

Results

Methods *A, B* and *C* identified 66, 67 and 68 volatiles respectively. Volatiles were categorised into 21 groups, such as n-aldehydes, Strecker aldehydes, sulphur containing compounds, pyrazines. Method *B* had lower reproducibility compared to method *C* and method *A* was best (Table 2).

Table 2 Reproducibility of 21 volatile groups based on average CV of each method.

Mean CV	Method A	Method B	Method C
High (CV<35%)	8	2	4
Good (35≤CV<50%)	8	4	6
Middle (50≤CV<80%)	3	9	10
Low (CV>80%)	1	5	1
Not detected	1	1	0

Figure 2 shows the ratio of volatile groups detected by each method relative to method *C*. Method *A* usually extracted the highest quantities of volatiles, followed by method *B* and method *C*. Exceptions are n-aldehydes, pyrazines and low molecular weight (MW) ketones. Interestingly, method *A* failed to pick up most of the volatiles from the alcohol group. Method *B* failed to pick up long chain acids and alkanes.

Five criteria were considered to select the most suitable method for beef volatile analysis (Table 3), and each criterion was given a score from a scale of 1 (bad) to 5 (very good). Methods *A* and *C* scored the highest. Although method *A* was easy to use, most reproducible and detected the highest quantities of volatile compounds, it failed to detect some volatile compounds such as unsaturated alcohols.

Table 3 Method selection and score.

Criteria for method selection	Method A	Method B	Method C
Lower amount of beef sample required	3	5	5
Ease of use	5	4	4
High reproducibility	5	1	3
Detect wide range of volatile compounds	2	3	5
Detect high quantities of volatile compounds	5	4	3
Total score	20	17	20

Conclusion

The comparison of three SPME methods showed that the three methods all had strengths for different compound groups. However, Method *C* was the most suitable for analysis of a wide range of volatile compounds in cooked

Notes

beef.

References

1. Legako, J. F., Dinh, T. T., Miller, M. F., Adhikari, K. and Brooks, J. C. 2015. Consumer palatability scores, sensory descriptive attributes, and volatile compounds of grilled beef steaks from three USDA Quality Grades. Meat Sci, 112, 77-85.

2. Bueno, M., Resconi, V.C., Campo, M.M., Ferreira, V. and Escudero, A. 2019. Development of a robust HS-SPME-GC-MS method for the analysis of solid food samples. Analysis of volatile compounds in fresh raw beef of differing lipid oxidation degrees. Food Chemistry, 281, 49-56.

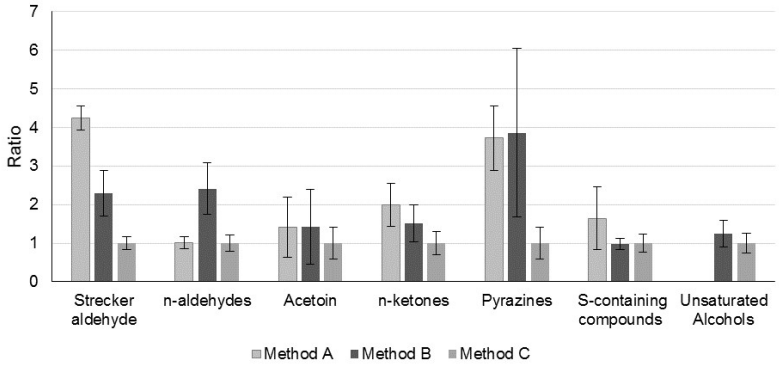

Figure 2 Ratio of volatiles collected relative to method C for main volatile groups in beef samples

Strecker aldehydes: 3-methylbutanal, 2-methylbutanal, methional, benzaldehyde, benzeneacetaldehyde; n-aldehydes: pentanal, hexanal, heptanal, octanal, nonanal, decanal, undecanal, dodecanal and tridecanal; n-ketones: 2-heptanone, 2-octanone, 2-nonanone, 2- decanone; pyrazines: methyl pyrazine, pyrazine, 2,3/5-dimethyl pyrazine, trimethyl pyrazine, 2,3-diethyl-5-methyl pyrazine, 2-ethyl-3,5-dimethyl pyrazine; S-containing compounds: dimethyl disulfide, dimethyl trisulfide, 2-acetylthiazole, benzothiazole, 2-methyl thiophene; unsaturated alcohols: 1-penten-3-ol, 1- octen-3-ol

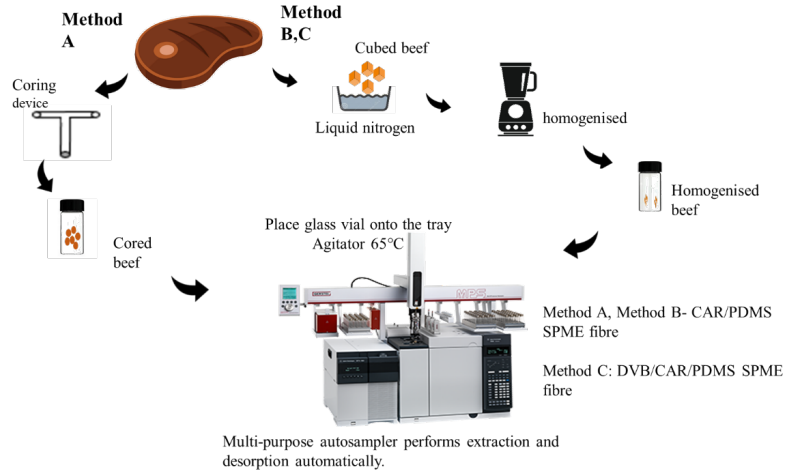

Figure 1 Description of methods employed for solid-phase micro-extraction (SPME) analysis.

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