HS-HCC Techniques In Combination With Multi Cumulative Trapping and GC×GC-qMS Followed by Machine Learning to Explore the Impact Of Packaging On Coffee Brew Aroma

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Introduction

Volatile and semi-Volatile Organic Compounds (VOC and sVOC) are often monitored for assessing authenticity and quality of foods. In fact, VOCs and sVOCs may contain multi-level information. Their analysis is usually performed by the sampling of the headspace by high concentration capacity sorptive (HS-HCC) techniques, as most often solid-phase microextraction (SPME), followed by gas chromatographic (GC) separation. The use of comprehensive bi-dimensional GC, which enables the separation according to two different stationary phases, has been proved to increase the information extractable from the VOCs profile, allowing treating the generated two-dimensional chromatogram as a highly informative fingerprint. On the other side, different HS-HCC tools are now available and easily automated, as well as the possibility to add a trapping step to perform multiple-cumulative extractions[1].

HS-HCCE-GC×GC-qMS

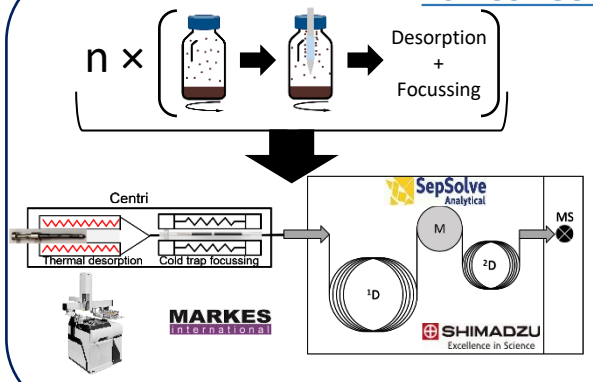

Fig 1. Sampling technique and instrument schema

HS-HCCE: HiSorb™ (67 µL PDMS), SPME DVB/CAR/PDMS 1 cm; 350 rpm stirred; 20' pre-equilibrium extraction, trap desorption 3 min at 300 °C (5.6 mL/min), injection (1:9.3) by Centri platform (Markes int.).

GC×GC: Shimadzu GCMS-TQ8050 NX; columns: 1D: BPX-5 20m × 0.18mm i.d. × 0.18 µm df 2D: BPX-50 5 m × 0.25 mm i.d. × 0.28 µm df (Restek). Flow modulator: 3.5s modulation period, INSIGHT flow modulator (SepSolve Analytical Ltd). Oven prog: 40 °C (5 min) to 180 °C at 6 °C min^{-1}.

Optimisation of extraction conditions

Comparable results were obtained using 1 and 4 mL of sample. The extraction yield increased until 30 min, not further improvement was observed further (**Fig. 2**).

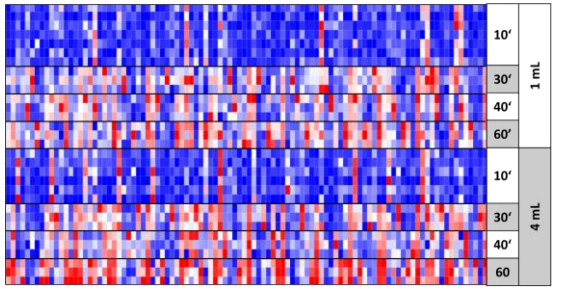

Fig 2. Time profile for 1 and 4 mL sample, extracted at 50 °C

Thus 1 mL and 30 min were selected, which also provide a better extraction of polar VOCs[2] for investigating the temperature profile (**Fig. 3**). Although 75 °C provided a higher yield, 60 °C was selected to better match the aroma perceived by the consumer[3].

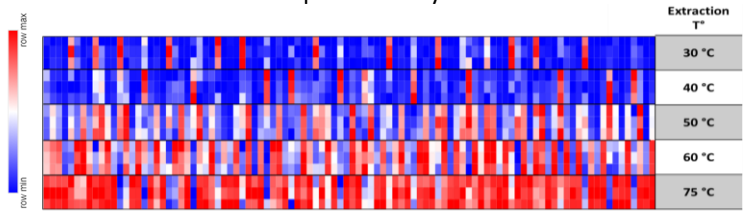

Fig 3. Temperature profile for 1 mL sample, extraction: 30 min

Packaging impact on the brewed aroma: single- and multiple cumulative trapping extraction by SPME and HiSorb

Samples: 6 aluminum and 6 biodegradable coffee capsules, and 11 grinded coffees stored in aluminum bag ("Pack") prepared using a stainless-steel reusable capsule. **Coffee preparation:** Nespresso Inissia coffee machine

- → All extraction conditions allow perfect clustering of packages according to their aroma (**Fig. 5**) .
- → MCT extractions applied with HiSorb™ provide better separation of samples according to their package groups for the same extraction time.

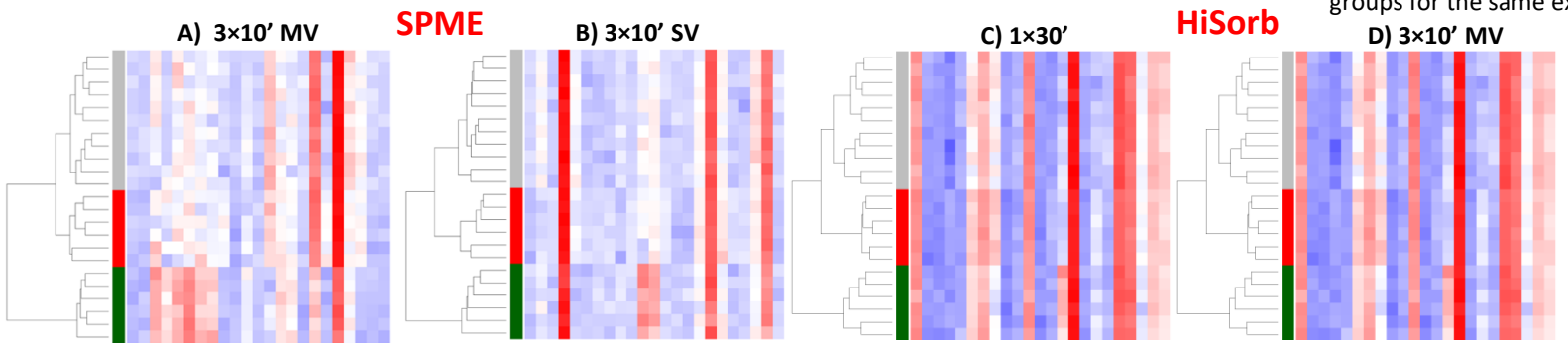

Fig 5. Heatmaps of model selected features for 1 mL coffee extracted at 60 °C 3×10 min MV and 3×10 min SV by SPME DVB/CAR/PDMS (A and B) and 1x30 min and 3x10 min mV by HiSorb™ (C and D)

- → HiSorb™ extractions offer better separation than SPME for the same extraction conditions.
- → MV approach slightly improves sample separation compared to SV for SPME.
- → SPME SV-MCT extractions have nearly the same performance as HiSorb™ mono extraction for the same extraction time (**Tab. 1**).

	HiSorb™		SPME DVB/CAR/PDMS	
	1×30'	3×10' MV	3×10' SV	3x10' MV
caps_alu vs caps_bio	1.1	2.9	1.1	1.8
pack vs caps_alu	0.8	0.9	0.7	0.8
pack vs caps_bio	1.0	3.0	1.0	1.7

Tab 1. Euclidian distance between packaging groups

References: [1]Stilo *et al.*, J. Sep. Sci., 44 (2021) 1592– 1611, [2]Bicchi *et al.*, J. Chromatogr. A., 1071 (2005) 111–118; [3]J. Abraham, K. Diller, J. Food Sci., 84 (2019) 2011-2014.

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