

Introduction

HiSorb™ is a relatively new high concentration capacity tool introduced to fill the gap between the more common solid phase microextraction (SPME) and the stir bar sorptive extraction. SPME is widely used thanks to the easiness of use and the possibility to automate the entire process, oppositely from SBSE, which, despite a higher sensitivity requires additional manipulation since not easily automated. HiSorb™ provides an increased sensitivity due to the significantly higher sorbent volume along with easy automation. The increased trapping capacity was explored in comparison to traditional SPME and coupled to comprehensive gas chromatography-quadrupole mass spectrometry (GC×GC-qMS) to maximise the level of information extractable from the chromatographic fingerprint obtained from the volatile profile of brewed coffee.

HiSorb™-GC×GC-qMS

HS-HiSorb™-HCCE: 67 µL PDMS; 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.

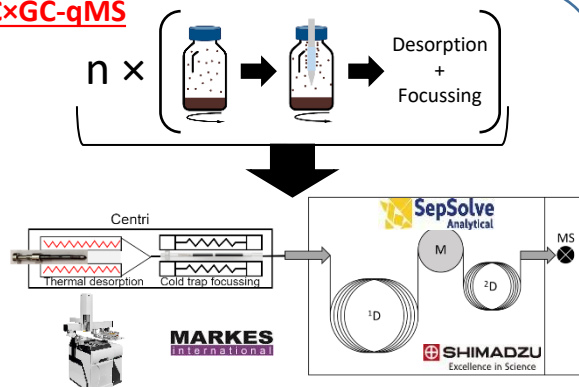


Fig 1. Sampling technique and instrument schema

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 above (Fig. 2).

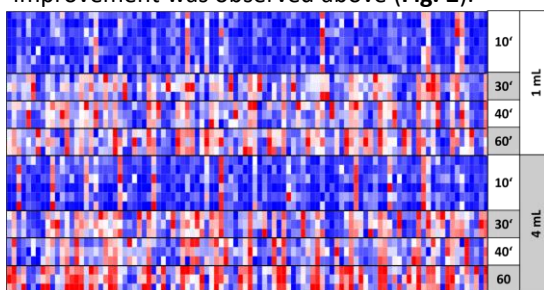


Fig 2. Time profile for 1 and 4 mL sample, extracted at 50 °C

Thus 1 mL and 30 min were selected, which also provide a better extraction of polar VOCs [1] 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 [2].

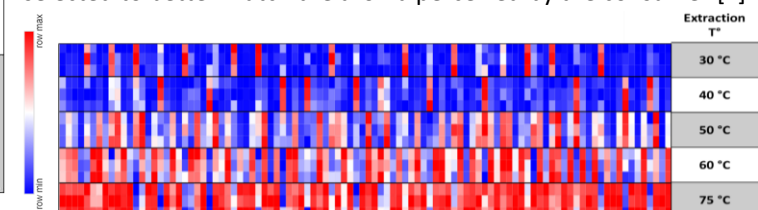


Fig 3. Temperature profile for 1 mL sample, extraction: 30 min

HiSorb™ and SPME comparison

Optimised conditions: 1 mL coffee extracted during 30 min at 60 °C

HS-SPME-HCCE: 100 µm PDMS and 1 cm long DVB/CAR/PDMS 50/30 µm df (Supelco®)

The extraction of coffee volatiles by HiSorb has been compared with the extraction performed with SPME using two different coatings under the same conditions (Fig. 4). HiSorb™ gives significantly better extraction than both SPME fibres.

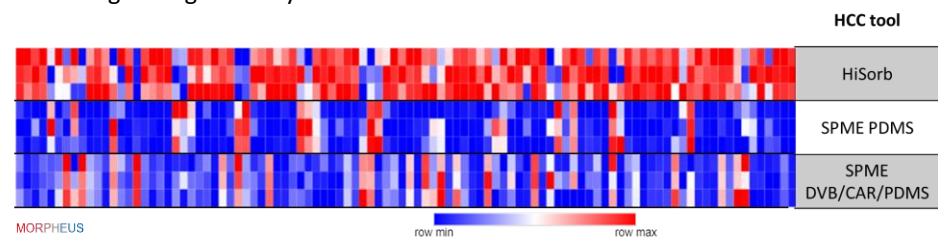


Fig 4. Tool comparison for 1 mL coffee extracted 30 min at 60 °C by HiSorb, SPME PDMS and SPME DVB/CAR/PDMS

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

Coffee preparation: Nespresso Inissia coffee machine and stainless-steel reusable capsule

Samples: 6 aluminum and 6 biodegradable coffee capsules, and 11 grinded coffees stored in aluminum bag ("Pack")

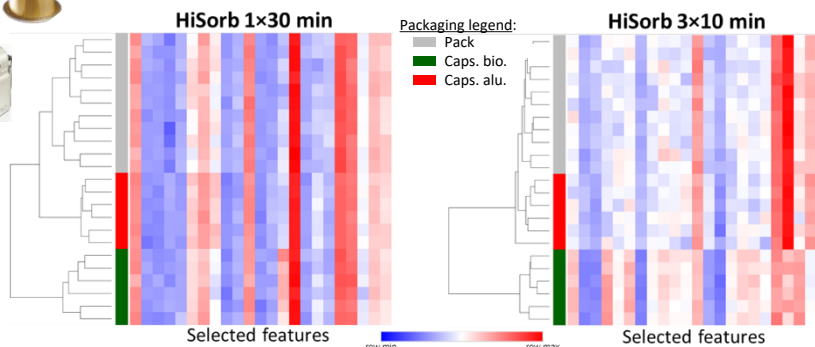


Fig 5. Heatmaps of model selected features for 1 mL coffee extracted 1×30 min (left) or 3×10 min (right) at 60 °C by HiSorb™

- Both 1×30 and 3×10 min extractions allow perfect clustering of packages according to their aroma (Fig. 5).
- MCT extractions allow for better separation of samples according to their package groups for the same extraction time (Tab. 1).

Compared groups	Euclidian distance for	
	1×30 min	3×10 min
Caps. alu. vs caps. bio.	1.1	2.9
Pack vs caps. alu.	0.8	0.9
Pack vs caps. bio.	1.0	3.0

Tab 1. Euclidian distance between packaging groups