Exploring the capabality of the HiSorb probe for characterization of volatile and semi-volatile compounds in brewed coffee by GC×GC-qMS

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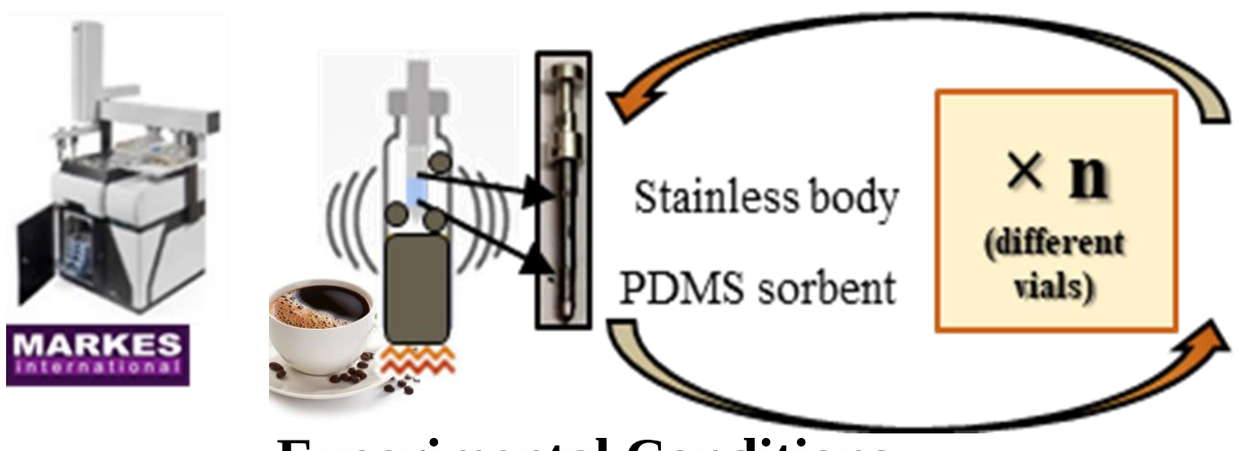
Introduction

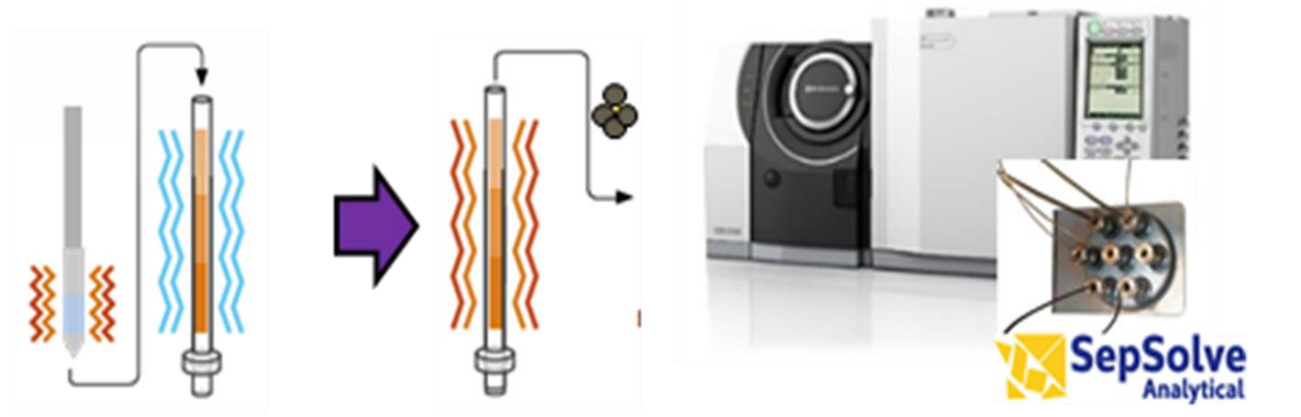
Volatile and semi-volatile components are highly relevant to characterize different samples, among which food. These compounds contribute to the peculiar aroma profile of foods and are widely used to assess quality and authenticity. The analysis of volatile and semi-volatile compounds requires the sampling of the headspace (HS) above the sample, which, now a day is mainly performed using high-concentration-capacity HS (HCC-HS) approaches, such as solid-phase microextraction (SPME), stir-bar sorptive extraction (SBSE). More recently, HiSorb has been introduced to fill the gap between SPME and SBSE. In fact, it can be easily automated (as for SPME) but. present a significantly higher sorbent volume which allows a much higher trapping capacity.

The aim of this work was to exploit the potentiality of HiSorb for the analysis of volatile and semi-volatile from brew coffee and compared to the traditional triphasic SPME fiber more widely applied. Time and temperature conditions were carefully optimized, and the possibility of performing multiple cumulative trapping was investigated as well. The analyses were performed on a multidimensional comprehensive gas chromatographic system coupled with a quadrupole mass spectrometer (GC×GC-qMS) and equipped with a flow modulator

Material and Method

HiSorb





Experimental Conditions

- Extraction time: 10, 30, 40 and 60 °C at 50 °C using 1 and 4 mL of brewed coffee
- Temperature: 30, 40 ,50, 60 and 75 °C during 30 min using 1 mL of brewed coffee
- classical extraction vs multiple cumulative extraction (HiSorb and SPME)

GC× GC-qMS

[1]D column: BPX-5MS 20 m × 0.18 mm × 0.18 µm
[2]D column: BPX-50ms 5 m × 0.25 mm × 0.25 µm
Oven: 40 °C (3 min); 6 °C/min to 250 °C
Carrier gas: He; Flow [1]D: 0.6 mL/min; [2]D 16 mL/min
Modulation period: 3.5 s; Flush time: 150 ms
2D Software: ChromSpace&ChromCompare+ (SepSolve)

Results and Discussion

Expansion of a 2D GC chromatogram is shown in ***Figure 1***. A serie of key compounds from coffee were selected to evaluate and optimize the conditions of extraction (extraction time on ***Figure 2*** and temperature on ***Figure 3***).

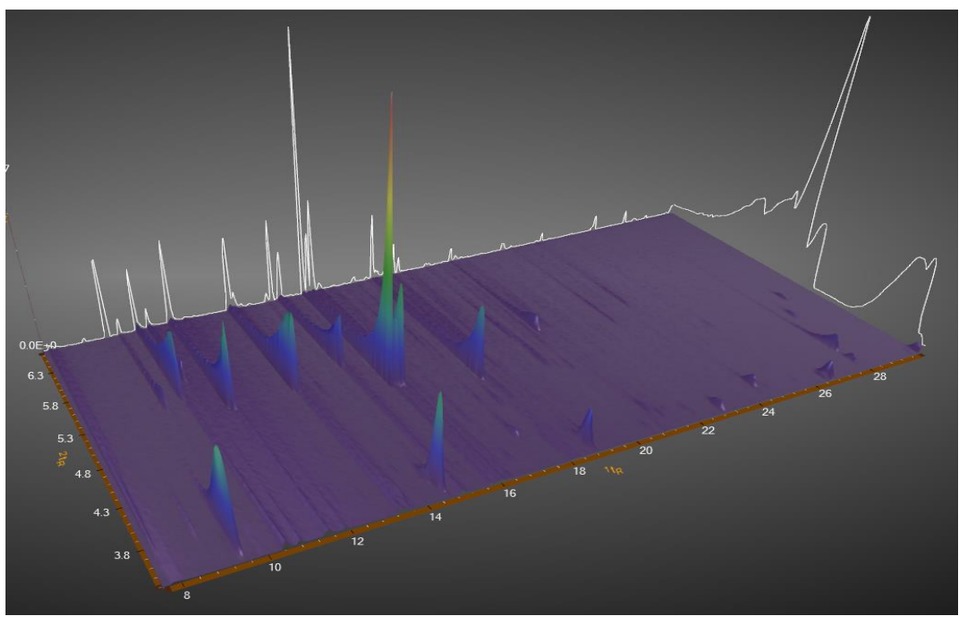


Figure 1: *Expansion of HiSorb extraction by GC×GC-MS*

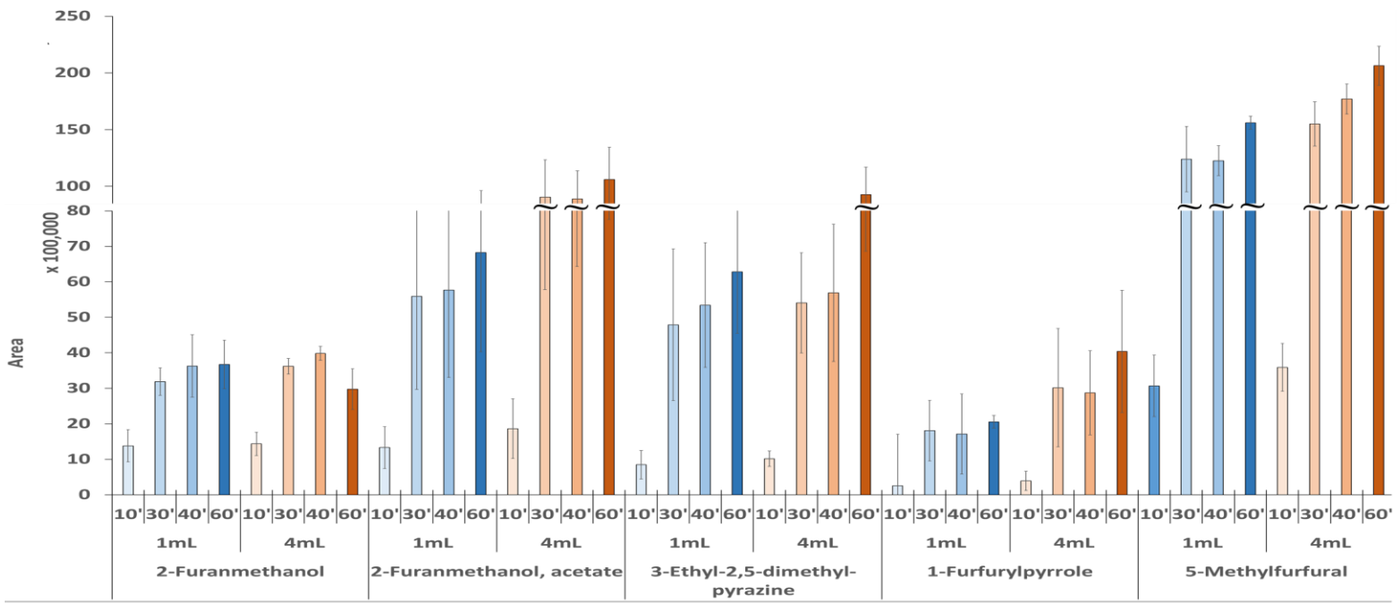


Figure 2: *Time profile at 50 °C using two different sample volumes (1 mL in blue and 4 mL in orange)*

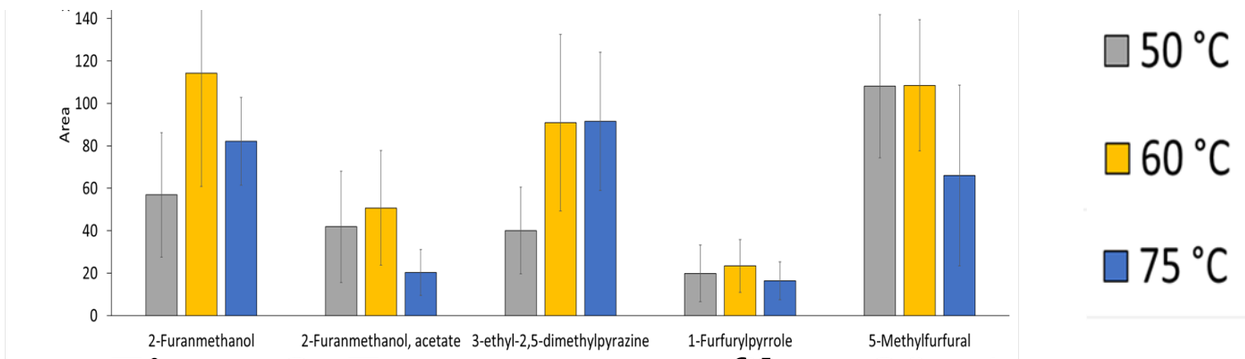


Figure 3: *Temperature profile at 30 min*

Figure 4 show the potentiality of HiSorb compared to DVB/CAR/PDMS SPME using classical extraction and multiple cumulative extraction.

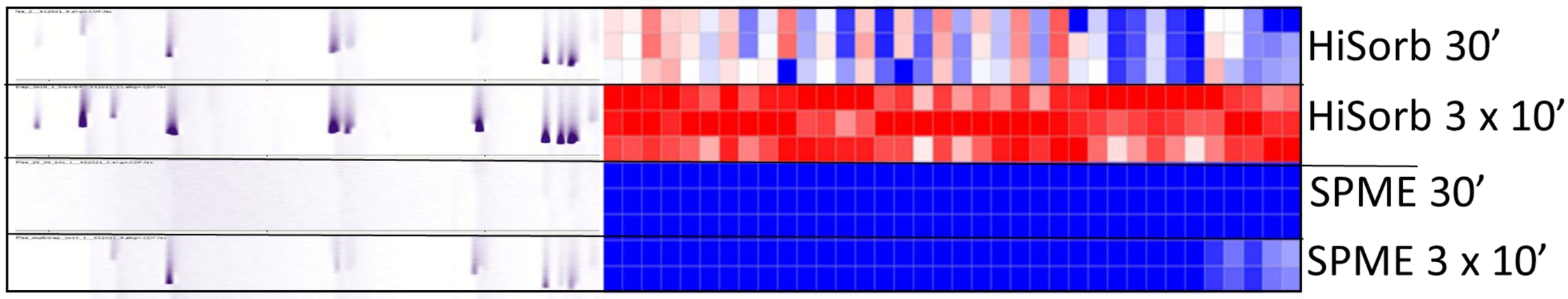


Figure 4: *Heat-maps comparison between HiSorb and DVB/CAR/PDMS using selected features. On the left example of the corresponding chromatograms*

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