

Analysis of volatile organic compounds in honey by ITEX-DHS GC-MS

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Introduction

Background:

Honey is a natural product which can be used by humans without any processing [1]. The analysis of **volatile organic compounds (VOCs)** is a faster approach to identify the honey's origin than traditional methods, which is important for the **quality control** of honeys. VOCs, like **aldehydes, hydrocarbons, alcohols, ketones, acids and esters**, can derive from the plant or nectar source, from the transformation processes of plant compounds, from heating or from microbiological or environmental contamination [2,3]. **In-tube extraction dynamic headspace (ITEX-DHS)** is a **fully automated** microextraction technique, in which the enrichment of analytes takes place by repeated aspirating and dispensing of the sample headspace through a sorbent trap. The thermal desorption takes place in the GC-injector [4].

Aims:

- **Optimization** of an ITEX-DHS GC-MS method for HS analysis of honeys
- **Validation** of the used method
- **Analysis** of VOCs in honey using ITEX-DHS GC-MS

Conclusions

- An ITEX-DHS GC-MS Method for the analysis of honey was **successfully** developed using honey samples.
- Method validation regarding **LODs, LOQs, linear range, recovery and reproducibility** was achieved.

Future work and outlook

- Different Honey samples need to be measured.
- Principal component analysis (**PCA**) shall help to group the samples based on floral and geographical origin.

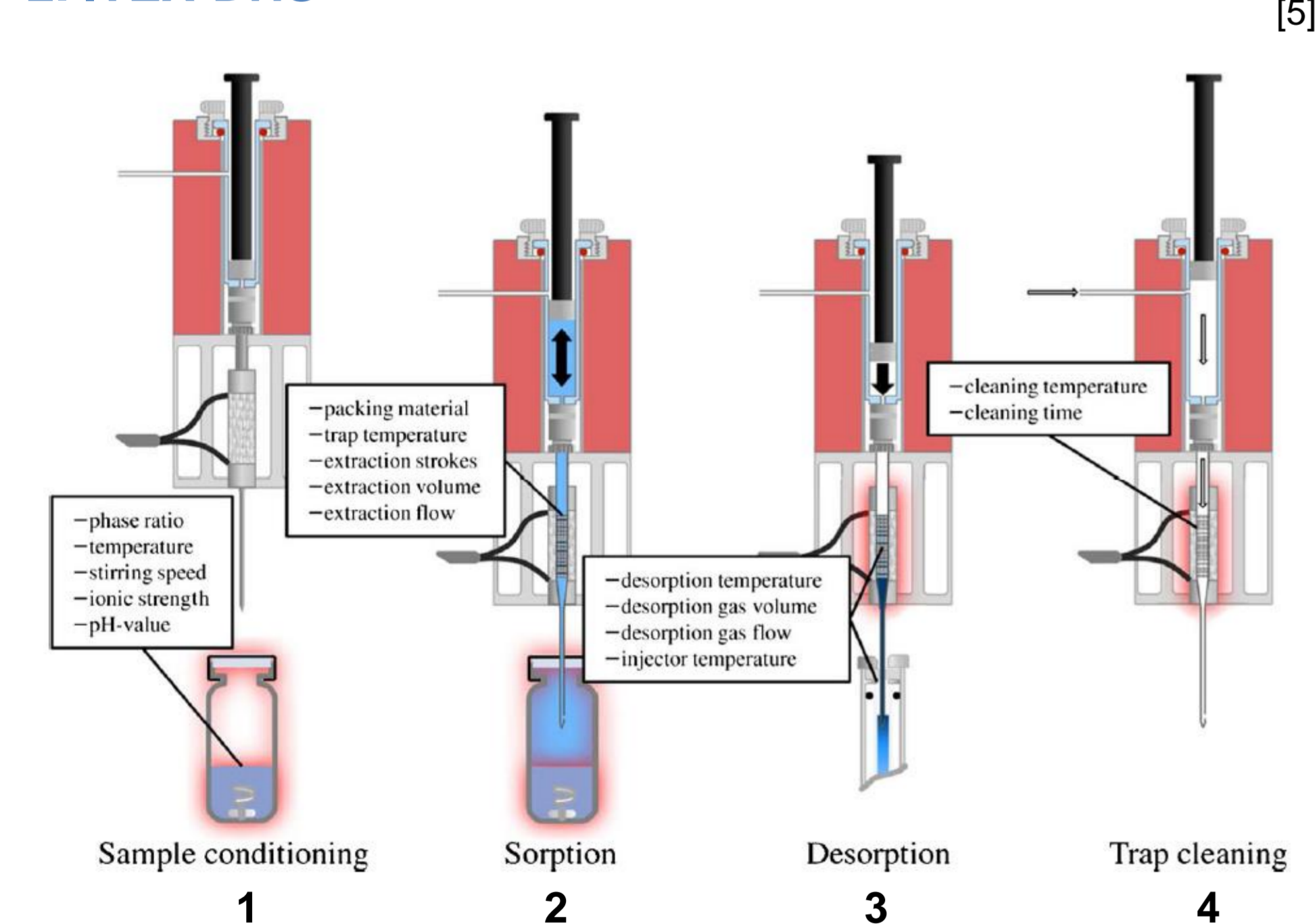
Experiments and Methods

1. Sample preparation

Stock solutions of all analytes were prepared with $c = 1$ g/L in MeOH
Standard mix containing all analytes was prepared with $c = 50$ mg/L in MeOH
Standard mix was diluted in 10 mL 25 % (w/v) NaCl corresponding to the calibration levels
Honey samples were prepared weighing 1 g of honey and diluting it with 10 mL 25 % (w/v) NaCl

GC-MS	Trace GC ultra with DSQ II (ThermoFisher Scientific) and Optic 3 (GL Science)
ITEX	PAL combi xt (CTC Analytics) with ITEX II solution
Software	Xcalibur Data System Version 2.2 (ThermoFisher Scientific)

2. ITEX-DHS



- 1) 30 min, 70 °C, 500 rpm
- 2) 65 cycles $V_S=1$ mL
extraction speed of 100 μ L/s
70 °C, 500 rpm
- 3) $V_D=1$ mL speed 50 μ L/s at 300 °C
- 4) 15 min at 300 °C

3. GC-MS

Injection mode	Splitless
Interface T	260 °C
Ion source T	230 °C
Injector T	300 °C
Cryofocussing T	-20 °C
Carrier gas	Helium (99.999%, Air Liquide)
Gas flow	1.5 mL/min
MS mode	EI @ 70 eV, TIC m/z 40-200, 500 amu/s

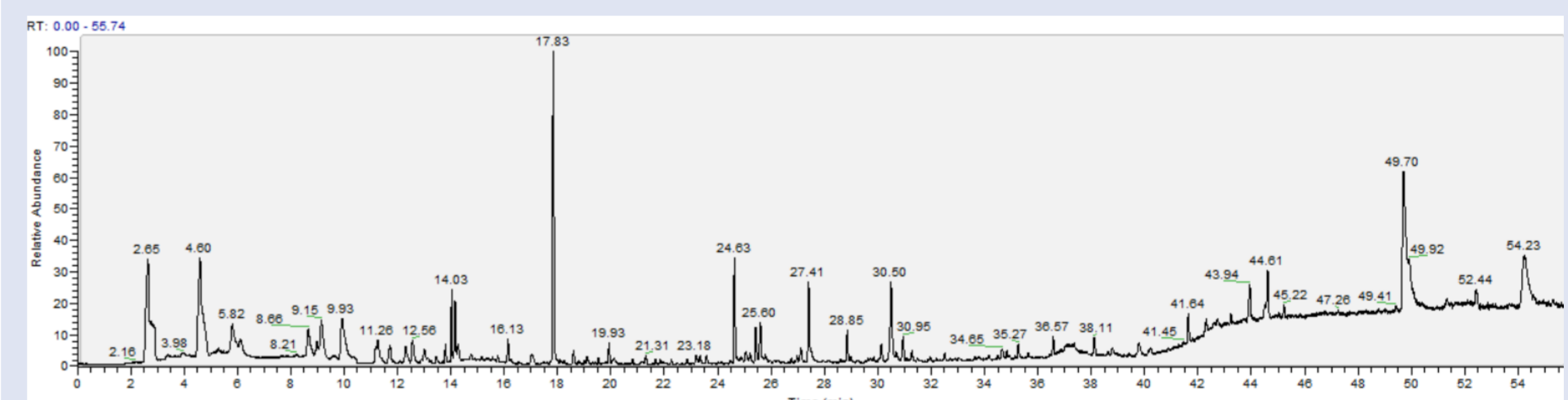
Optima FFAP plus 60m x 0.32 mm x 0.5 μ m (Macherey-Nagel)

ramp	temperature	hold time
-	35 °C	5 min
7 °C/min	110 °C	2 min
5 °C/min	200 °C	4 min
10 °C/min	230 °C	2 min

Results and Discussion

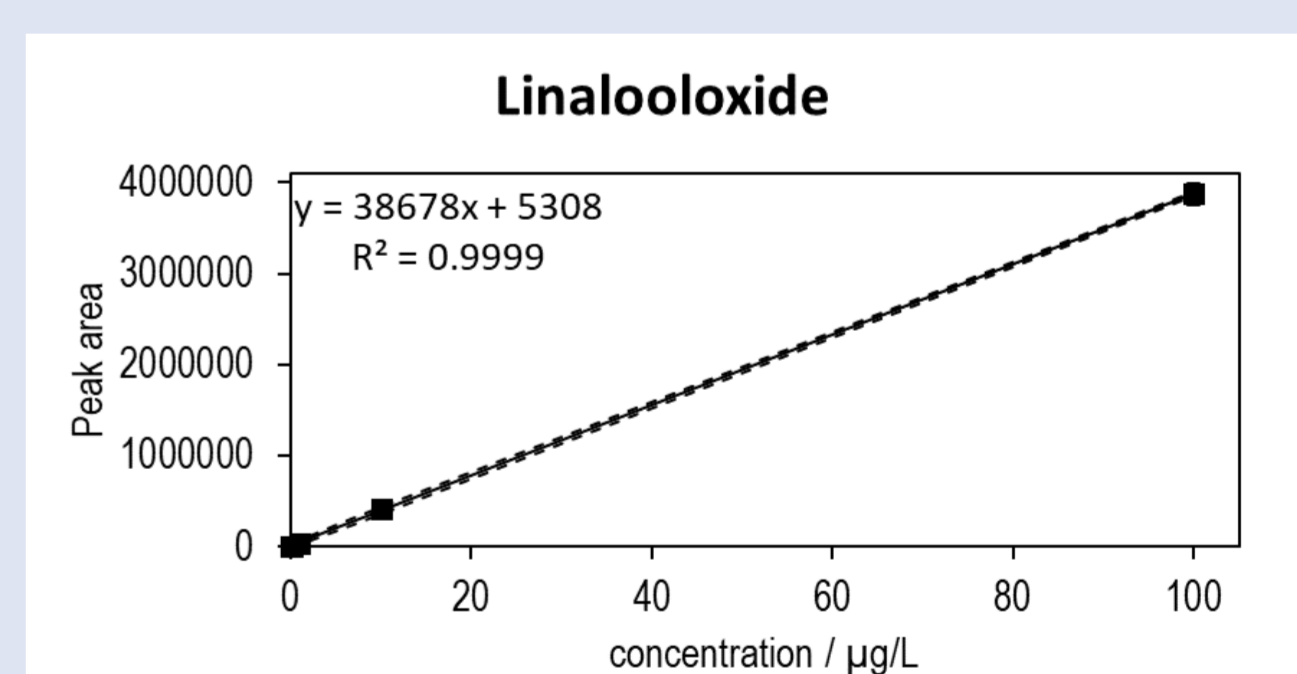
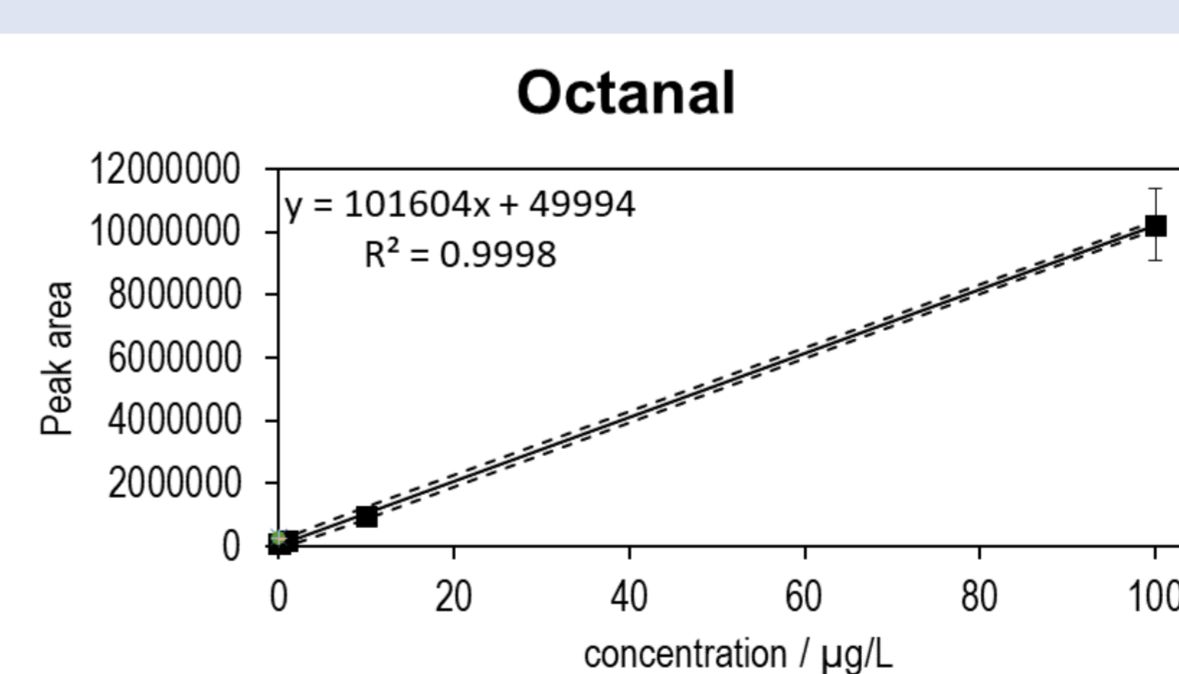
1. Optimization of ITEX-DHS GC-MS

Exemplary chromatogram of a honey sample to show peak capacity



- Honey matrix doesn't effect chromatographic results negatively
- Extraction time of sample overlaps with GC runtime of previous samples
→ very **time efficient**
- **Cryofocussing** at -20 °C is beneficial but not necessary

2. Calibration Curves for 2 representative analytes



3. Validation results

compound	R ²	LOD / ng/L	LOQ / ng/L	RSD / %	recovery / %
Dimethylsulfide	0.9995	5.71	19.05	6	83
Octane	0.9993	1.14	3.81	14	89
Octanal	0.9998	2.22	7.41	12	86
Linalool oxide	0.9999	8.57	28.57	11	102
Benzaldehyde	0.9997	1.04	3.48	4	99
Thymol	0.9999	1.76	5.88	11	100
Carvacrol	0.9999	1.69	5.63	8	96

- 25 % (w/v) NaCl-solution spiked with standard mix
- **Linearity** from 0.01-100 μ g/L
- **R² > 0.9993**
- **LOD** (S/N = 3) and **LOQ** (S/N = 10)
- **RSD < 14 %**
- **Recovery** between 83 and 102 %

References

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