Use of headspace gas chromatography – ion mobility spectrometry for fraud detection in the floral origin of honey

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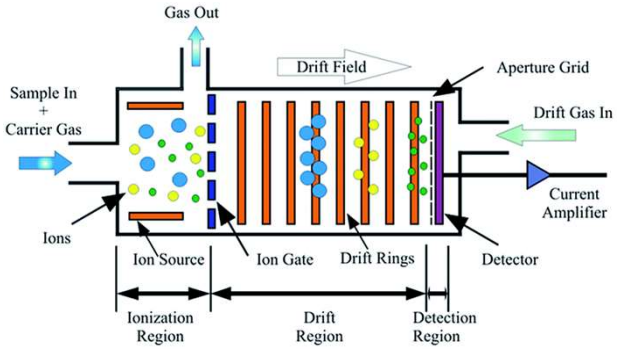
INTRODUCTION

Nowadays, many types of honey from different botanical origin are available in the market. They differ in their organoleptic properties such as color, flavor or smell, thereby the interest in honey characterization relating to floral origin is increasing. In this work, the instrumental technique headspace-gas chromatography coupled to ion mobility spectrometry (HS-GC-IMS) is used for monitoring honey volatile compounds that allow the differentiation and characterization of honey samples according to their floral origin.

ION MOBILITY SPECTROMETRY

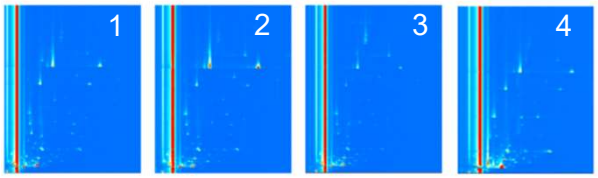


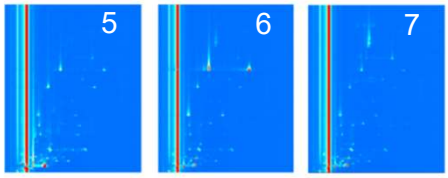
IMS separation is based on the charge, mass, size, and shape of the ions

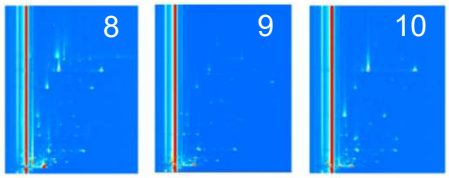
HS → GC → IMS

The HS-GC-IMS coupling allows the separation in two dimensions obtaining a spectral fingerprint of each sample.

HS-GC-IMS SPECTRA

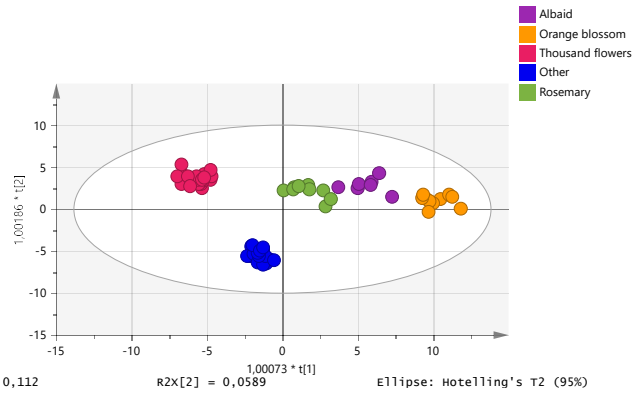






1. Thyme honey
2. Rosemary honey
3. Orange blossom honey
4. Cantueso honey
5. Oak honey
6. Thousand flower honey
7. Albaid honey
8. Heather honey
9. Broom honey
10. Melon honey

OPLS-DA CHEMOMETRIC MODEL



Due to the complex multi-dimensional data and the large variability between samples, visual differentiation is not posible. Therefore, an exhaustive chemometric processing is required.

Data processing is based on a non-targeted strategy using peak-region features (275 markers)

Success validation rate of 100%

Compound	Linear range (mg/kg)	Equation	R^2	LOQ (mg/kg)
2-Pentanone	0.19-10	y = 0.117 ln x + 0.641	0.942	0.19
2-Butanone	0.23-10	y = 0.117 ln x + 0.886	0.905	0.23
2-Nonanone	0.27-10	y = 0.077 ln x + 0.291	0.900	0.27
6-Methyl-5-hepten-2-one	0.15-10	y = 0.135 ln x + 0.355	0.927	0.15
2-Heptanone	0.25-10	y = 0.128 ln x + 0.537	0.959	0.25
Benzaldehyde	0.31-10	y = 0.131 ln x + 0.466	0.959	0.31
trans-2-hexen-1-al	0.40-10	y = 0.076 ln x + 0.250	0.966	0.40
p-Anisaldehyde	0.21-10	y = 0.030 ln x + 0.026	0.913	0.21
4-Methylacetophenone	0.22-10	y = 0.031 ln x + 0.693	0.905	0.22
trans-2-pentanal	0.36-10	y = 0.130 ln x + 0.767	0.970	0.36
Ethyl acetate	0.10-10	y = 0.085 ln x + 0.920	0.913	0.10
2-Hexanone	0.31-10	y = 0.042 ln x + 0.851	0.962	0.31
Heptanal	0.45-10	y = 0.015 ln x + 0.006	0.952	0.45
Hexanal	0.38-10	y = 0.028 ln x + 0.039	0.930	0.38
Ethyl isovalerate	0.40-10	y = 0.132 ln x + 0.410	0.981	0.40
Octanal	0.48-10	y = 0.018 ln x + 0.008	0.895	0.48
Isovaleraldehyde	0.43-10	y = 0.004 ln x + 0.002	0.966	0.43
Nonanal	0.11-10	y = 0.006 ln x + 0.108	0.969	0.11

METHOD VALIDATION

- **The proposed methodology can also be applied for the quantitative analysis of volatile compounds present in honey.**
- **A logarithmic calibration was necessary.**
- **A total of 14 of markers could be identified.**

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