

# Application of conventional and rapid analytical strategies for hazelnut volatilome characterization

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• **Hazelnut** is a tree nut with relevant industrial importance. Up to **90%** of hazelnut utilization is based on **processed products** obtained from **roasted kernels**. Hazelnut paste (manteca) is a raw materials widely used in confectionery industry.

- The **roasting** is a fundamental step in hazelnut industrial processing, and it drastically modify the volatile fraction composition.
- It is important to identify characteristic trends for the volatile organic compound (VOC) formation/release and differences/similarities between cultivars in relation with thermal treatments.

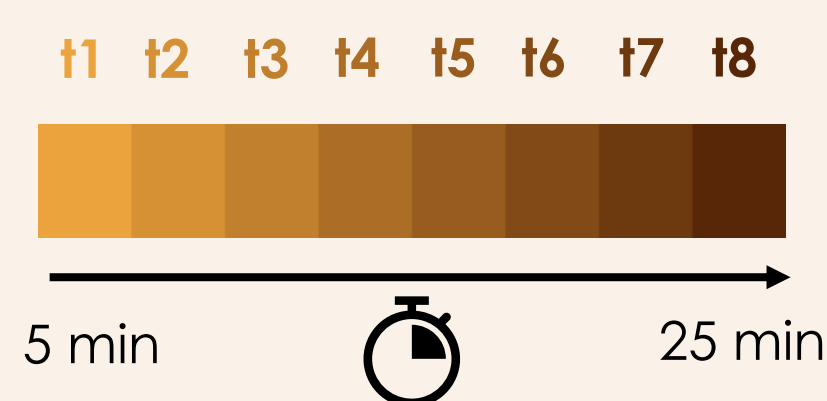
## Samples



**Hazelnut paste** samples were obtained by roasting kernels from different geographical and botanical origins.



## Lab scale infrared roasting – 140°C



**8 samplings** (150-200 g) → roasted hazelnut paste samples at increasing roasting intensity

## Aim

The application of **rapid analytical techniques** (PTR-ToF-MS and GC-IMS) and a conventional SPME-GC-MS approach to characterize the volatilome of roasted hazelnut paste.

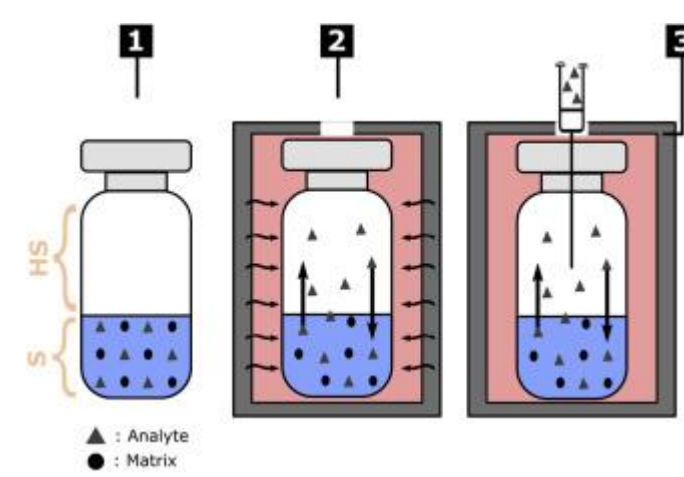
## Methods

VOC measurement were performed in parallel with the three analytical techniques:

- SHS-PTR-ToF-MS (PTR-ToF-MS 8000, Ionicon, Austria, equipped with an MPS Multipurpose Sampler, GERSTEL, Germany)
- HS-SPME-GC-MS (7890B GC system coupled to a 5977A MSD, Agilent, Little Falls, DE, equipped with an MPS Multipurpose Sampler, GERSTEL, Germany)
- SHS-GC-IMS (FlavourSpec GC-IMS system, G.A.S., Germany, equipped with a HT2000H headspace autosampler, HTA, Brescia, Italy).

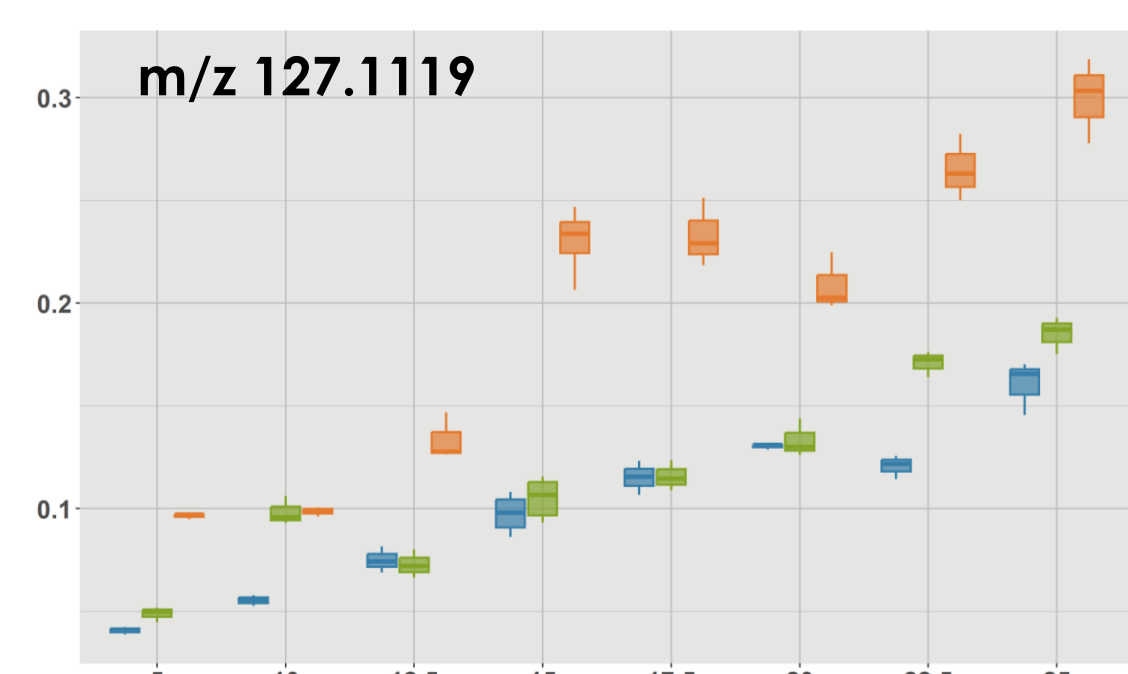
	Conventional GC-MS	Rapid GC-IMS    PTR-ToF-MS	
<b>Sampling</b>	HS-SPME	SHS	SHS
<b>Analysis time</b>	60 min	30 min	3 min

For HS analysis (both static and SPME) 1 g of paste was placed in a 20 ml HS vial. Each sample was analyzed in triplicate. All the sample vials were stored at -20°C until the measurement.



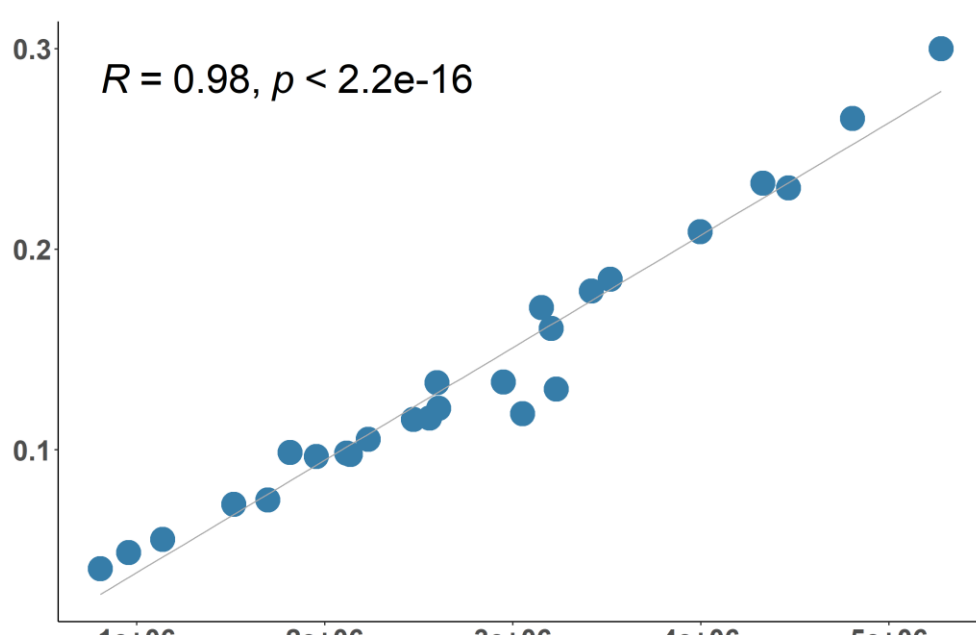
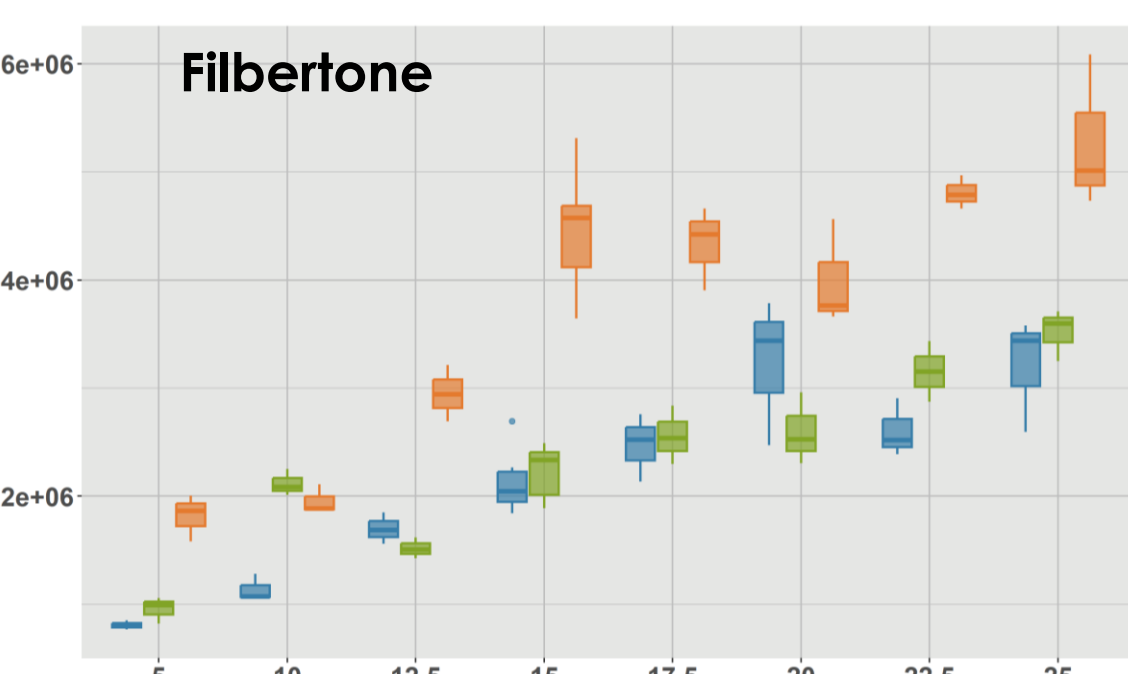
## Combining PTR-ToF-MS and GC-MS

### Improvement of identification



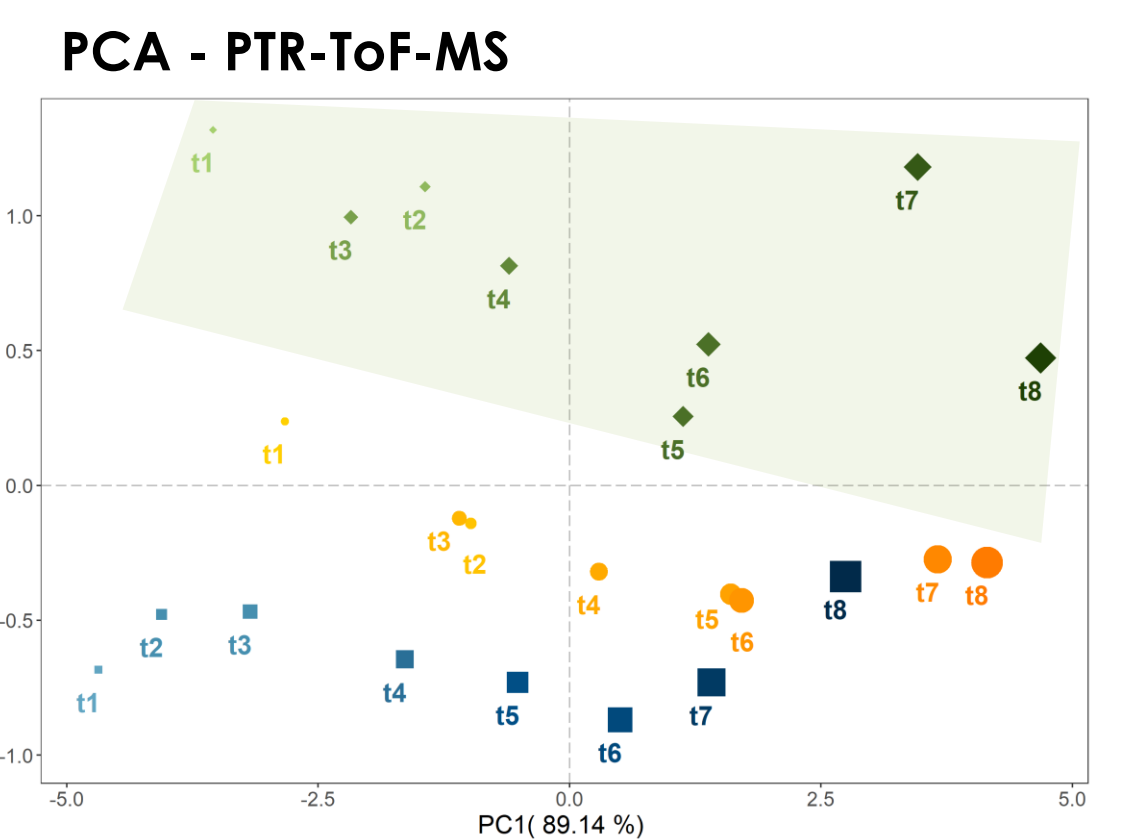
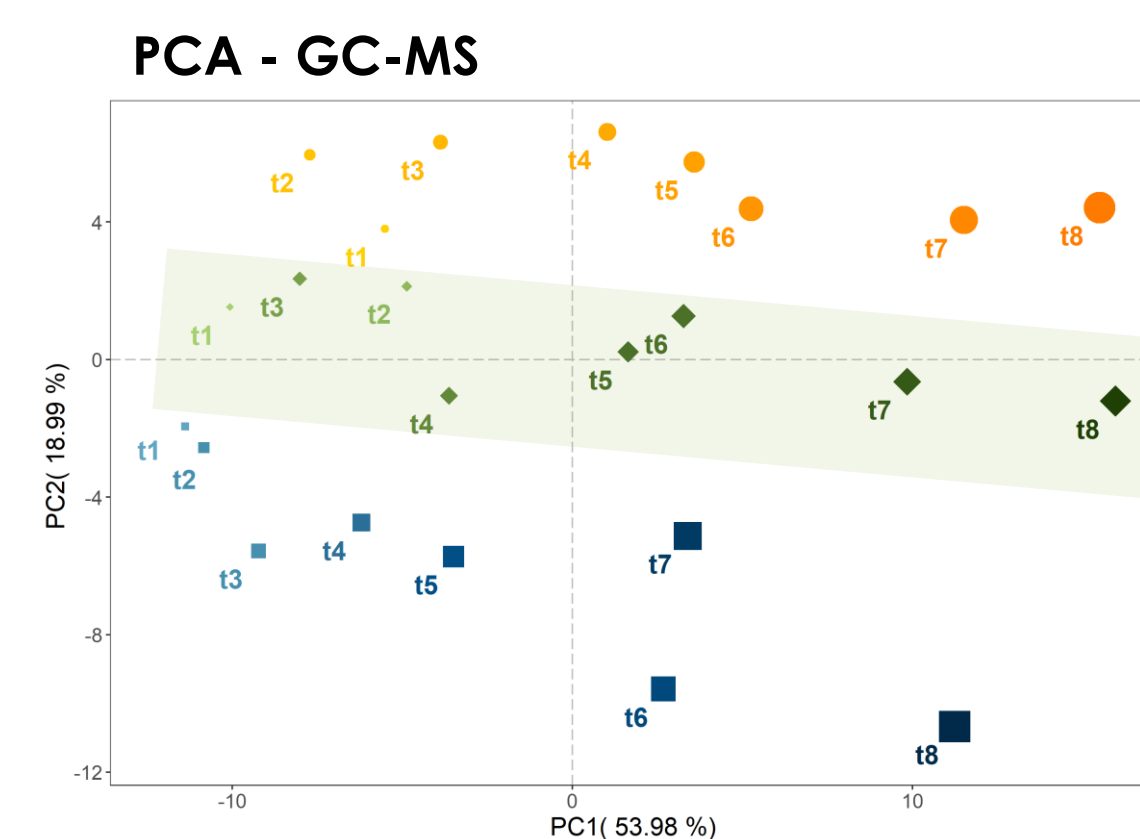
The **m/z 127.1119** was tentatively identified as **filbertone** ((E)-5-methyl-2-hepten-4-one), an important key odorant of hazelnuts.  
 $C_8H_{14}O \rightarrow C_8H_{14}OH^+$

- m/z 127.1119 and filbertone (detected by HS-SPME-GC-MS) show the **same trend** during the roasting process.
- The intensities of m/z 127.1119 and filbertone are **highly correlated**.



The combined application of PTR-ToF-MS and GC-MS increase reliability of identification

### Increase of extracted analytical information



**RO** samples have different relative position in the Cartesian plane of PCA.

The loadings which contribute most to the separation of RO samples on PC2 correspond to:

- **m/z 46.9950** →  $C_2H_5SH^+$
- **m/z 63.0274** →  $CH_2SH^+$

These sulphur compounds were not detected by SPME-GC-MS

The combined application of PTR-ToF-MS and GC-MS provides more comprehensive results in terms analytical information

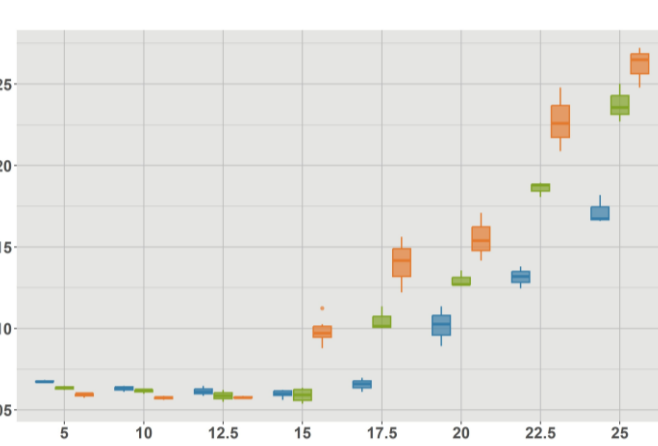
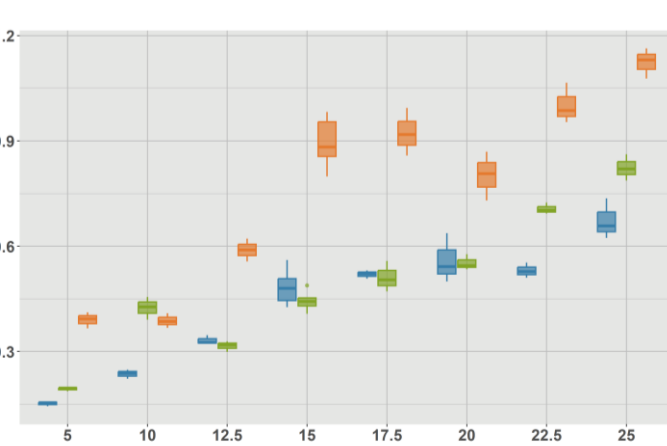
## Exploring the potential of GC-IMS

### 1) From targeted..

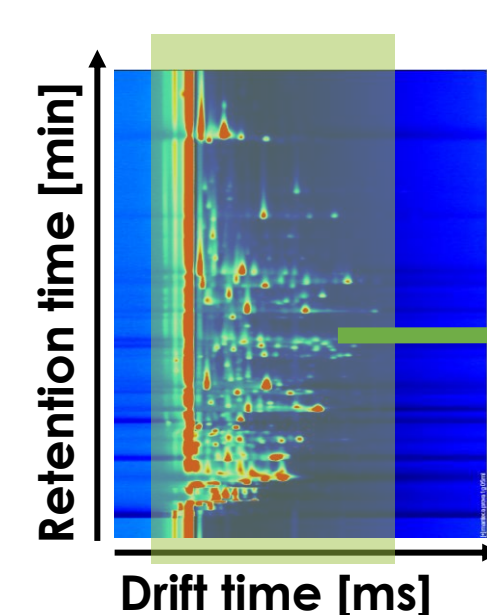
Monitoring of target analytes (manual peak selection)

**Filbertone**  
(proton bound dimer)

**2,5-dimethyl pyrazine**  
(proton bound dimer)

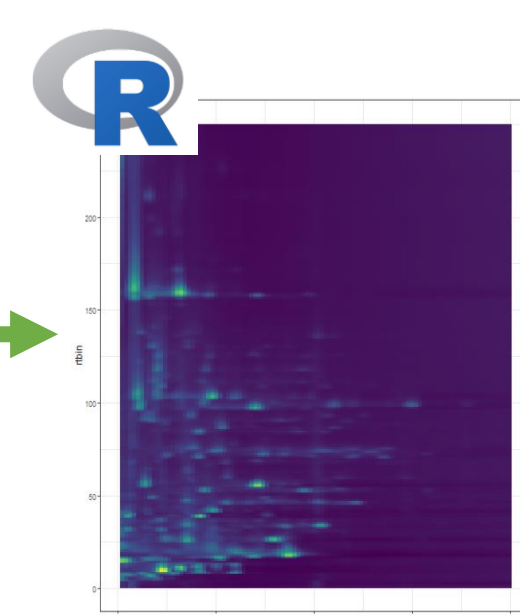


### 2)...to untargeted

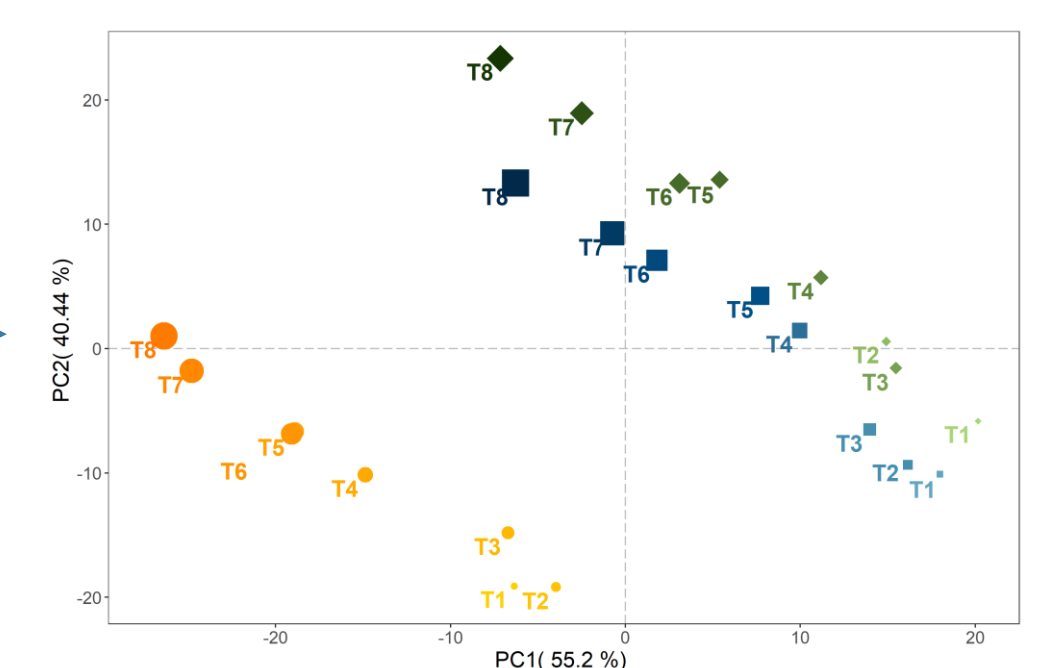


1) Selection of the region with peaks

2) Binning of the bidimensional matrix



3) Background removal (by thresholding)



An untargeted approach enable to automatize GC-IMS data elaboration, avoiding manual peak selection.

## Conclusion

A characterization of roasted hazelnut volatilome was performed combining rapid (PTR-ToF-MS and GC-IMS) and conventional (GC-MS) techniques. Outcomes of this study demonstrated the complementarity of these analytical techniques and the prospect to apply them for hazelnut volatilomics.

## Bibliography

- Lytów, A. E., Panagou, E. Z. & Nychas, G. J. E. Volatilomics for food quality and authentication. *Curr. Opin. Food Sci.* **28**, 88–95 (2019).
- Romero-Aroca, A., Rovira, M., Cristofori, V. & Silvestri, C. Hazelnut Kernel Size and Industrial Aptitude. *Agriculture* **11**, 1115 (2021).
- Farneti, B. *et al.* Volatilomics of raspberry fruit germplasm by combining chromatographic and direct-injection mass spectrometric techniques. *Front. Mol. Biosci.* **10**, (2023).
- Wang, S., Chen, H. & Sun, B. Recent progress in food flavor analysis using gas chromatography-ion mobility spectrometry (GC-IMS). *Food Chem.* **315**, 126158 (2020).

## Acknowledgements

**SOREMARTEC**