Società Chimica Italiana
Divisione di Spettrometria di Massa

FONDAZIONE EDMUND MACH

DIFFA23

DIRECT INJECTION FOOD FLAVOUR ANALYTICS

BOOK OF ABSTRACTS

Fondazione Edmund Mach

San Michele all'Adige (TN), Italy

20 - 22 September 2023

1st International Symposium on
Direct Injection Food Flavour Analytics (DIFFA)

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Proceedings of the DIFFA23 - 1st International Symposium on Direct Injection Food Flavour Analytics

Fondazione Edmund Mach – San Michele All'Adige (TN) Italy

20-22 September 2023

This book collects the conference proceedings of the 1st International Symposium on Direct Injection Food Flavour Analytics, held at the Fondazione Edmund Mach from 20th to 22nd September 2023.

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FOREWORD

Volatile organic compounds (VOCs), particularly flavour compounds, represent an invaluable noninvasive metric to follow the multi-faceted journey of food, from the farm to the fork and beyond, such as relating to the human microbiome after consumption or in addressing reduction strategies for food waste. VOCs thereby serve as a direct and swift means of measurement and notably act as a main driver of the perceived quality of food.

Mass spectrometry (MS) is an established yet increasingly pivotal tool in food and beverage characterization with a broad range of applications. When coupled with gas chromatography (GC), it stands as the predominant analytical method for exploring many aspects of food, from safety to traceability and nutritional aspects, and equally facilitates control measures in quality and process monitoring.

Recent remarkable advancements in both technology and methodology have paved the way for highly sensitive, specific, rapid, robust, and validated MS-based techniques that have become indispensable in food science and technology research and application. A subgroup of these technologies has been devised over the past two decades in the form of analytical approaches that enable the analysis of VOCs through direct injection. These methods have gained attention for their rapid, highly sensitive and high-throughput analytical capabilities.

A leading technology in this area is proton transfer reaction-mass spectrometry (PTR-MS), which has driven many innovative applications for direct flavour/food analysis. Commencing 2003, the University of Innsbruck, Austria, has organized a biennial event dedicated specifically to PTR-MS and its applications, including a focused session on food science and technology.

The **1st International Symposium on Direct Injection Food Flavour Analytics (DIFFA23)** was conceived with the backdrop of the PTR-MS conference but with a different aim, namely to embrace a broader community beyond PTR-MS uses, encompassing similar direct injection mass spectrometry (DIMS) technologies, such as atmospheric pressure chemical ionization-mass spectrometry (APCI-MS) and selected ion flow tube-mass spectrometry (SIFT-MS), with a primary emphasis on flavor compounds. It was also not exclusive to MS-based analytical techniques, but welcomed the inclusion of complementary non-MS approaches, such as solid-state sensors, fast gas chromatographic direct approaches and ion mobility spectrometry (IMS), amongst others, to ensure a wider reach and broader engagement. The meeting was established to foster scientific discussions of common interest and facilitate scientific collaborations. This book of abstract highlights the details of the event and contains the contribution summaries of both the oral and poster presentations.

The conference featured one plenary and four keynote lectures delivered by distinguished guests, as well as numerous invited and contributed talks and 25 poster presentations, with 97 attendees from different EU states, the USA, the UK, Israel and New Zealand. The event provided valuable insights into direct injection food/flavour analytics, with reviews from pioneering scientists who played key roles in developing and advancing DIMS methods in its early days, such as Andy Taylor, Patrik Španěl and Jean-Luc Le-Quéré, showcasing both historical developments and recent advancements in analytical performance and novel applications. Topics discussed included nose-space analysis of composite foods, rapid and high-throughput phenotyping, fermentation monitoring, both as an

innovative technological tool and for investigating the human microbiota, advanced data analysis and data mining tools. These are just a few examples of the themes explored during the conference.

Numerous partners contributed to the success of the event: the sponsors, whose engaging presentations and financial support sustained the quality of the meeting and ensured that the conference fees were kept to a minimum, as well as various supporting institutions and patronages. Special thanks go to the Fondazione Edmund Mach (FEM) for its scientific contributions and for hosting the conference at the Research and Innovation Centre, as well as the Division of Mass Spectrometry of the Italian Chemistry Society (DSM-SCI) for their organizational support and creation and hosting of the conference website. The invaluable support from these companies and institutions are further acknowledged through inclusion of their logos on the back cover of this book.

The conference started a fruitful exchange of results, ideas and issues amongst scientists working with direct tools to monitor VOCs in food science and technology, with broad attendance from sensory and applications scientists from academia and industry.

We would like to thank all those who, through their participation and support, made this event possible, which exceeded our most ambitious expectations.

Thank you all, and we look forward to seeing you at the next edition.

On behalf of the Scientific Committee

Franco Biasioli, Jonathan Beauchamp, Pat Silcock

CONFERENCE PROGRAM

20th September 2023

12.30-14.00 Registration and welcome buffet

Conference opening

14.00-14.10	Welcome addresses
	Fulvio Magni - *Società Chimica Italiana-Divisione Spettrometria di Massa*
	Mario Pezzotti - *Fondazione Edmund Mach*
14.10-14.20	Why DIFFA23?
	Franco Biasioli - *Fondazione Edmund Mach*
14.20-15.05	Plenary lecture: *DI-MS – A game changer for flavour research?*
	Andy Taylor - *University of Nottingham*

Session 1 | Unlocking Flavour with DIMS

Chairs: Pat Silcock & Nina Cleve

15.05-15.35	Jonathan Beauchamp - Fraunhofer Institute for Process Engineering and Packaging IVV
	The long and winding road: a flavoursome tale of PTR-MS
15.35-15.55	Graham Eyres - *University of Otago*
	What is Flavour and how can DIMS help untangle the puzzle?
15.55-16.15	Andreas Mauracher - *IONICON*
	Advantages of Next-Gen PTR-ToF instruments for food and flavour sciences

16.15-17.00 Tea break and poster session

Session 2 | DIMS in Health and Wellbeing

Chairs: Donatella Caruso & Eirini Pegiou

17.00-17.20	Josep Rupert - *Wageningen University & Research*
	Signalling volatile compounds in the human gut microbiota: new avenues offered by direct analytical methods.
17.20-17.40	Chris Mayhew - *University of Innsbruck*
	Real-Time Trace Analysis of Breath Volatiles using Proton Transfer Reaction Mass Spectrometry: implications for in-vivo flavour release measurements
17.40-18.00	Enrico Davoli - *Istituto Mario Negri*
	Direct analysis of sex-wellness products using a field deployable MS equipped with a Direct Sampling Atmospheric Pressure (DSAP) source
18.00-18.20	Corrado Di Natale - *University of Rome Tor Vergata*
	Direct injection mass spectrometry and gas sensors: a teacher-pupil relationship
18.20-18.40	Luca Cappellin - *University of Padua*
	Improved compound identification in direct VOC analysis using an EI&CI-TOFMS

19.00 Welcome cocktail - cloister of the monastery and historical cellar

21st September 2023

Session 3 | Linking DIMS Data to Sensory Perception

Chairs: Graham Eyres & Iuliia Khomenko

9.00-9.30	Jean-Luc Le-Quéré - *INRAE-CSGA Dijon* *Twenty years of Direct Injection Mass Spectrometry for aroma research in Dijon*
9.30-9.50	Catrienus De Jong - *Wageningen University & Research* *Exploring new in vivo and in vitro methods to integrate sensory and instrumental analysis to get insight and improve the flavour of plant-based food products during oral processing and drinking*
9.50-10.10	Markus Stieger - *Wageningen University & Research* *In vivo aroma release and sensory perception of composite foods*
10.10-10.20	Michele Pedrotti - *Wageningen University & Research* *Characterization of plant-based milks by combining sensory analysis with headspace and nose-space direct injection mass spectrometry*
10.20-10.30	Karina Gonzalez-Estanol - *Wageningen University & Research* *In vivo analysis of nose-space concentration by direct injection mass spectrometry to study the effect of chewing rate on aroma release during food consumption*
10.30-10.40	Laura Hill - *University of Nottingham* *Understanding the relationship between lipids, capsaicin and aroma release in confectionery*

10.40-11.10 Coffee break and poster session

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Chairs: Fulvio Magni & Caroline Perltier

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11.30-11.45	Nina Cleve - *Fraunhofer Institute for Process Engineering and Packaging IVV* *Milk matters: Unraveling retronasal aroma release and perception of coffee by combining in vivo nosespace analytics with dynamic sensory methods*
11.45-12.05	Tomasz Majchrzak - *Gdansk University of Technology* *What happens when food goes into oil during deep frying? Monitoring the first minutes of frying using PTR-MS*
12.05-12.20	Gregory Schmauch - *Rational F&E GmbH* *Influence of product quantity, cooking parameter and flow tube pressure on the measurement with Sift-MS in a cooking oven*
12.20-12.40	Vaughan Langford - *Syft Technologies* *Application of SIFT-MS to chemical and sensory screening of packaging materials*

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14.00-14.15	Terry Bates - *Cornell University* *Rapid headspace solid-phase microextraction with sheets with direct analysis in real time mass spectrometry (SPMESH-DART-MS) of derivatized volatile phenols in grape juices and wines*
14.15-14.30	Matteo Tonezzer - *University of Cagliari* *PTR-MS as a tool to understand and improve the performance of electronic noses*
14.30-14.45	Andrea Warburton - *University of Otago* *Application of PTR-ToF-MS to monitor development of flavour in sourdough*
14.45-15.05	Paolo Redegalli - *Shimadzu Italia S.r.l.* *Characterization of isoflavones and its metabolites in foods by direct probe ionization mass spectrometer (DPiMS) with high resolution detection*
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Chairs: Riccardo Flamini & Michele Pedrotti

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17.40-18.00	Ana Rita Monforte - *AFB INTERNATIONAL* *Modelling the kinetics of flavour formation & relaese as a function of ingredients addition in real food systems*
18.00-18.20	Pietro Franceschi - *Fondazione Edmund Mach* *Mining datasets from untargeted direct analytical methods: a data analyst point of view*
18.20-18.35	Mickael Le Bechec - *Institute of Analytical Sciences and Physico-Chemistry for Environment and Materials (IPREM)* *Volatile fingerprints of food thanks to the untargeted use of SIFT-MS raw data*

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22nd September 2023

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Chairs: Catreinus de Jong & Brian Farneti

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10.15-10.30	Pedro Martinez Noguera - *University of Copenhagen* *Using PTR-ToF-MS to quantify microbial off-flavors geosmin and 2-methylisoborneol in water. Method development, performance assessment and comparison with established GC-MS methods*
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10.45-11.05	Rupert Holzinger - *Utrecht University* *Using SI traceable gas standards to improve the accuracy of untargeted PTR-MS measurements*

11.05-11.45 Coffee break and Poster Session

Session 8 | Floral, Biogenics and Phenotyping

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12.05-12.25	Brian Farneti - *Fondazione Edmund Mach* *DI-MS as high performing VOC phenotyping tool to support the horticultural production chain management*
12.25-12.40	Alberto Roncone - *Fondazione Edmund Mach* *Validation of gas chromatographic methods for the botanical characterization and authentication of lavender essential oil by stable isotope analysis of its organic volatile compounds*
12.40-12.55	Eugenio Aprea - *University of Trento* *Contribution of volatile organic compounds to multifloral honey flavor*
12.55-13.15	Daniele Zatta - *University of Padua* *Comparative analysis of VOC purification techniques in complex cooking emission: adsorption, photocatalysis and combined systems.*

13.15-13.30	Closing remarks Fulvio Magni - *Società Chimica Italiana-Divisione Spettrometria di Massa* Franco Biasioli - *Fondazione Edmund Mach*

13.30 Farewell buffet

LIST OF CONTRIBUTIONS

P.09 Application of conventional and rapid analytical strategies for hazelnut volatilome characterization

Mazzucotelli Maria[1,3]*, Pietro Franceschi[1], Iuliia Khomenko[1], Brian Farneti[1], Emanuela Betta[1], Elena Gabetti[2], Luca Falchero[2], Andrea Cavallero[2], Eugenio Aprea[3]

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Summary: Food volatilome characterization has an increasing important role in food quality assessment. Hazelnut volatilome is influenced by the botanical and geographical origin of kernels, and by the industrial processing steps, such as roasting. Different analytical approaches can be used, individually or in combination, for food volatilomics. In this study, two rapid analytical techniques (GC-IMS and PTR-ToF-MS) were applied in parallel with conventional HS-SPME-GC-MS analysis for the characterization of hazelnut VOCs.
Keywords: hazelnut, volatilome, PTR-ToF-MS, GC-MS, GC-IMS

1 Introduction

The volatilome of a food matrix is defined as the pool of volatile organic compounds (VOCs) present in the system and it is a complex mixture of compounds belonging to different chemical classes. From one hand, it is influenced by many aspects, from the geographical and botanical origin of the raw matrix to the technological processes to which it is subjected. From the other hand, the VOCs present in foods play an important role in determining their sensory quality and acceptability. Thus, food volatilome investigation represents an important tool for food quality assessment, traceability, and process monitoring purposes [1].
Hazelnut (*Corylus avellana L.*) is a tree nut with relevant industrial importance. The wide use of hazelnuts in the confectionery industry entails a complex supply chain. Furthermore, up to 90% of hazelnut utilization is based on processed products obtained from roasted kernels, such as hazelnut paste [2]. It is therefore important to identify characteristic trends of VOC formation/release and differences/similarities between cultivars and geographical origins in relation with industrial processing.
Food volatilomics studies are performed applying different analytical platforms and approaches, individually or in combination. Gas Chromatography coupled with Mass Spectrometry (GC-MS) is the reference and conventional technique for VOC characterization. However, a time-consuming pre-concentration step is usually required, and the chromatographic separation entails relatively long analysis time. These drawbacks can be particularly severe in case large samplings, such as for quality control and phenotyping. In this case, direct injection mass spectrometry (DIMS) techniques, such as Proton Transfer Reaction Mass Spectrometry (PTR-MS), offer alternative analytical tools because of their sensitivity and high throughput. However, in complex matrices, such as roasted hazelnuts, the presence of isomeric compounds, and the formation of some fragments and clusters, make the compound identification challenging or impossible. The use of DIMS on large sample sets with a

simultaneous GC-MS based analysis on selected samples provides comprehensive results in terms of both analytical information and large sampling capability [3].
The second rapid analytical technique applied in this study is Gas Chromatography coupled with Ion Mobility Spectrometry (GC-IMS). This technique is rapidly gaining popularity for food volatilomics application, in particular untargeted fingerprinting approaches [4] due to the ease-of-use of the instrumental platform and the relatively short analysis time per sample.
The aim of this contribution is the comparison of PTR-MS, GC-IMS and GC-MS for roasted hazelnut volatilomics investigation, and a critical evaluation on the potential of a comprehensive analytical strategy based on their combination.

2 Experimental

Hazelnuts from three geographical and botanical origins were employed in this study: "Tonda Gentile Romana" monocultivar hazelnuts from Lazio region (Italy), "Tonda Gentile delle Langhe" monocultivar hazelnuts from Piemonte region (Italy), and "Akçakoca" hazelnuts from Turkey. The roasting process was carried out in a pilot scale infrared roaster. Small aliquots (150-200 g) of kernels were collected regularly throughout the thermal treatment, obtaining samples at increasing roasting intensity. Each aliquot was processed to obtain the paste samples, which were characterized for the VOC profile applying the three techniques.
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) and SHS-GC-IMS (FlavourSpec GC-IMS system, G.A.S., Germany, equipped with a HT2000H headspace autosampler, HTA, Brescia, Italy).

3 Results

The VOC profile of hazelnut paste samples was determined applying three analytical platforms: HS-SPME-GC-MS, SHS-PTR-ToF-MS, and SHS-GC-IMS. A targeted approach was applied for the monitoring of key-odorants (filbertone) and roasting markers (alkyl pyrazines), and different evolution trends were observed among the geographical/botanical origins. For example, filbertone, an important hazelnut key odorant, resulted more abundant in "Tonda Gentile delle Langhe" hazelnuts throughout the entire roasting process.
In parallel, untargeted strategies provided insights on the complexity of roasted hazelnut volatilome and its evolution during the thermal treatment. In this case, the combined application of analytical techniques enabled to increase the reliability of analyte identification and provides more comprehensive results in terms of extracted information.

4 Conclusions

The volatilome of roasted hazelnut was characterized using rapid and conventional analytical techniques.
The study indicates, on one side, the potential of a combined approach for the volatilome characterisation, and, on the other, the possibility of the use of rapid analysis tools when preliminary investigation by GC-MS enables the annotation of the relevant features.

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