White Paper

Application of Aroma Release Monitoring in Real Time as a Powerful Tool to Understand Consumer Perception

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Introduction

Aroma is one of the key determinants of flavour perception and therefore one of the main factors driving consumer preferences. This is the reason why the analysis of Volatile Organic Compounds (VOCs) has become a major topic for food industry. Indeed, a huge variety of analytical techniques have been developed to isolate and determine the concentration of VOCs in food. Moreover, headspace techniques have been applied to understand the effect of food components (proteins, lipids, carbohydrates, polyphenols, etc) on the volatility of aroma compounds. These interactions can modify the release of VOCs from the food matrix and affect the aromatic balance of the final product. However, these techniques cannot totally recreate the consumption experience, and thus correlation with sensory has traditionally proved difficult.

Hence, to understand the rate of VOCs reaching the olfactory receptors during consumption (and therefore taking into account the physiological factors involved in the consumption procedure), new technologies such as proton transfer reaction mass spectrometry (PTR-MS) allow the real time monitoring of VOCs directly from the human nose (nose-space analysis). Consequently, these techniques take into account both, food and consumers, and therefore are becoming key techniques to better understand the consumer experience.

The aim of this document is to review the available techniques to measure food aroma, especially focusing on the most innovative methodologies to measure aroma release during consumption. This information is critical for the industry to characterise and optimise the aroma of its product portfolio.

Chemical Characterisation of VOCs

Classic techniques (based on pre-concentration of VOCs followed by their separation and analysis by GC) have allowed identifying and quantifying thousands of VOCs. This task has constituted a challenge for industry and researchers since from a chemical point of view, VOCs are a very diverse group of compounds. They only have in common some characteristics like their low molecular weights (< 300 Da) and their high vapour pressures. However, they belong to very different chemical families (aldehydes, acids, esters, pyrazines, etc), possess different physico-chemical properties (polarity, volatility) and can be present in very different concentrations (from ng to mg/kg). Because of their low concentration in food, VOCs generally need to be pre-concentrated before being analysed.

Classic pre-concentration techniques allow isolation of VOCs from a food matrix. The most employed have been distillation, solvent extraction, and Solid Phase Extraction (SPE). These techniques or combinations of them are still being widely used because of their reproducibility and sensitivity. However, they present some major drawbacks related with the use of hazardous solvents, the risk of compound losses or artefact generation, and, mainly, because they are time consuming.

Other isolation techniques are based on monitoring the headspace, in a static or dynamic mode. The static headspace technique does not need sample pre-treatment; however, its concentration capacity is very limited, as is its sensitivity. In the dynamic headspace technique, the volatile compounds placed in the headspace are purged and concentrated in a cold trap or a sorbent under a gas flow. The trapped volatiles are transferred to the chromatographic system, generally by rapidly heating the trap. However, these two techniques are being replaced by modern headspace sampling techniques with greater concentration power, such as Solid Phase Micro-Extraction (SPME), Stir Bar Sorptive Extraction (SBSE), or Solid Phase Dynamic Extraction (SPDE). They are based in the use of different polymeric sorbents occurring in different formats (fibre, magnetic stirrer or syringe), so they can be chosen depending on the polarity of the compound of interest and on the matrix under study. Before applying them, several parameters and desorption steps during the extraction (temperature, time, agitation, etc) must be optimised to obtain the best results. Their main advantages are the minimum sample preparation, the higher sensitivity and the easy automation thanks to the use of auto samplers, results in time and money saving. However, the selectivity is dependent on the polymer of choice and they cannot provide data in real time.

After extraction, Gas Chromatography (GC) is the most employed method for the separation
and subsequent analysis of VOCs. In this regard, the use of the classic one-dimensional GC has been combined in recent years with the use of multidimensional chromatography which results in better resolution, improving the separation in complex samples. Moreover, the emergence of new detectors (Ion Trap-MS, ToF-MS) provides higher sensitivities and faster scanning than the more traditional ones.

**Sensory Impact of VOCs**

It is well established that food products usually contain several hundreds of VOCs. However, only about 20 to 30 of them are important to generate its aroma. Therefore, in order to perform meaningful aroma analysis, it is important to identify these aroma impact compounds. To do that, Gas Chromatography–Olfactometry (GC-O) is the technique of choice. In this technique, the “human panellist” sniffs the effluent from the chromatograph column and, when an odour is sensed, the time and odour quality, and sometimes the intensity are recorded. There are different methods available to work with this technique (AEDA, OSME, Charm, etc.).

After identification of the odour active compounds by GC-O analysis and their quantification, the Odour Activity Value (OAV) (ratio between the concentration of one compound in the food and its threshold concentration) can be calculated. Generally, if one compound shows an OAV > 1, then this compound would exert a sensory impact.

**Aroma Release in Real Time**

Food consumption is a dynamic process during which compounds are released from the matrix into the oral cavity where they are subjected to different physiological factors before being perceived. While all conventional analytical techniques allow the determination of aroma compounds in the product itself or its headspace, they do not provide any information on how these compounds are being released during consumption. Therefore, new methods to monitor the dynamic of aroma release during consumption have been recently developed, and are moving from academic institutions towards more commercial applications. Some examples are Atmospheric Pressure Chemical Ionisation (APCI-MS), Proton Transfer Reaction (PTR-MS) or Selected Ion Flow Tube - Mass Spectrometry (SIFT-MS). These techniques are characterised by presenting short response times, which make them suitable to monitor aroma release in real time directly from the nostrils of human subjects. Therefore, this approach, also known as “breath-by-breath” or “nose-space” analysis, provides a better representation of the volatiles that reach the olfactory epithelium. This therefore gives a closer picture of what consumers can perceive. Other advantages are related to the relatively high sensitivity, and “soft” ionisation of these techniques which implies a reduced compound fragmentation. On the negative side, the challenge to distinguish isobaric compounds (compounds with the same mass) should be noted, and makes the identification of some compounds quite difficult and sometimes impossible.

One possibility to at least minimise this limitation is to combine this methodology with a Time-of-Flight (ToF) analyser, as it has been the case for PTR-MS. This set up provide higher mass range, very fast measurement and higher mass resolution which opens new horizons for research in fields such as environmental sciences, food sciences and medicine. Therefore, PTR-ToF-MS can be considered as a powerful research tool to provide rapid characterisation and understanding of VOCs release.

**PTR-ToF-MS**

In PTR-MS a hollow cathode ion source produces pure $\text{H}_3\text{O}^+$ ions, which are driven to the drift tube. Here, VOCs are introduced (usually without any pre-treatment), ionised (if proton affinity higher than water) and detected in the ToF-MS. As this technique employs air as carrier gas, the maintenance is simple.
Panellist performing a nose-space analysis in real time

**Volatile Release Data Interpretation**

As can be seen in Figure 2, with PTR-ToF-MS well-resolved time–intensity curves of the ions of interest can be obtained. Based on these curves, some key parameters which characterise the compound release can be extracted such as the area under the curve (AUC) which represents the total amount released, the maximum intensity of the release profile (I\text{max}), and the time necessary to reach the maximum intensity (T\text{max}).

![Figure 2](image)

**Aroma Release Measurement Applications**

The application of techniques like PTR-ToF-MS can enable a straightforward solution to a wide variety of flavour challenges as well as provide novel insights for new product development.

Below, two studies (one *in vivo*, one *in vitro*) outline the benefits a tool such as PTR-ToF-MS can bring to new product development.

**Study 1 – In vivo Experiment**

More and more food manufacturers try to develop healthier products with lower amounts of fat. However, changing the fat level can result in an unbalanced aroma profile and an adverse effect on sensory perception. Figure 3 shows the aroma release profile (measured from the nose of a panellist consuming the product) of two aroma compounds added at the same concentration level in two different matrices (one with high content in fat and the other one with low content). As it can be seen, the reduction of the fat level significantly affects the release of aroma compound 1 (yellow line), while aroma compound 2 (green line) seems to be unaffected.

![Figure 3](image)

**Study 2 – In vitro Experiment**

Besides the *in vivo* measurements, PTR-MS is a very versatile technique that allows performance of *in vitro* experiments. The *in vitro* experiments could be fast and non-invasive headspace analyses to monitor fundamental and industrial processes (quality control), or more sophisticated by the employment of *in vitro* devices (also known as artificial mouths, release cells, or retro-nasal aroma simulators). These are simple mechanisms more similar to a dynamic headspace analysis, but include some physiological conditions in order to recreate the release of aroma compounds in the mouth or in the throat. This experimental approach, although it cannot reproduce exactly the complexity of the eating/drinking process (as happens during the *nose-space* analysis), has the advantage of allowing the study of specific oral physiological variables involved in this process. Moreover, the increase in sensitivity and high reproducibility are other advantages when compared to approaches involving human panellists.

In a second study (Figure 4), the different behaviour of two Earl Grey teas (loose leaf and tea bag) from the same manufacturer shall be demonstrated using an *in vitro* experimental set-up. Interestingly, the loose tea leaves themselves present high aroma intensity above the cup (as measured by the concentration of a key aroma impact compound of bergamot oil). However, when the tea beverage is prepared, i.e. water is introduced in the system, the aroma concentration decreases dramatically,
probably because of the formation of micelles retaining the hydrophobic aroma compounds. Interestingly, as it is shown in Figure 4, the behaviour observed in the tea bag is the contrary: weak aroma above the cup that increases during the tea preparation due to the presence of encapsulation systems within the product (allowing a controlled release over time). Therefore, the application of this methodology allows industry a better understanding of the real consumer experience, predicting how the products are going to behave during the preparation and consumption procedure, which is really close to the consumer experience.

However, to date the employment of these techniques has been mainly applied in academic research and for the study of model foods. Thus, in the future a growing development in this area is expected to clarify the mechanisms driving consumer preferences.

**Summary**

Food manufacturers are under pressure to reduce certain ingredients like salt, fat and sugar, as well as achieve productivity savings through more affordable ingredients or employ more sustainable raw material sourcing. There can in turn be a significant impact on flavour perception of the product, ultimately affecting the acceptability of the product.

Therefore, the application is shifting from more traditional techniques which only take into account the composition of the food towards new techniques that also consider the food oral processing to which the food is subjected during the consumption procedure. The combination of real time methodologies (such as nose-space analyses and dynamic sensory measurements), will become a powerful tool for industry to successfully achieve product reformulation and new product development in line with consumer preferences. Its employment will be associated with patent application and development of intellectual property based on objective analytical data.

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**Figure 4**

Effect of encapsulation on aroma release during tea preparation

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**Link Analytical and Sensory Methods**

Product consumption is a complex multisensory experience which changes during the time of consumption itself. Therefore, the link of dynamic analytical and sensory methods is a promising tool that could help industry in the development of new products based on the reformulation of the existing ones (e.g. products with low fat, salt and/or sugar level).
Andreas Czepa – Technical Specialist

Andreas holds a Master of Science in Food Chemistry as well as a PhD in Flavour Chemistry from the Technical University of Munich, Germany. He has over 10 years of professional experience in analytical flavour research, including the implementation and successful application of innovative flavour analysis methodologies across the food and tobacco industry. Andreas is a member of multiple international Chemical Societies as well as a reviewer of several scientific journals.

Carolina Munoz – Technical Specialist

Carolina is Ph.D in Biology and Food Science. Her research activities involve the development of analytical methods to better understand flavour perception. In this regard, her specialty focuses on the study of the effect of food matrix and physiological factors affecting the release of aroma compounds. She possesses an impressive research track record, with a large number of publications in scientific journals. She has held different positions in both, the public and the private sector, and so far, she has worked in three different countries (Spain, France and United Kingdom). She is passionate about the study of flavour and its relation to food consumption and consumer preferences. This information is essential to improve the flavour characteristics of food products in the healthiest manner possible, which is her career’s goal.

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