Flavour Analysis of Confectionery, Using SIFT-MS

Volatile organic compounds (VOCs) form a key component in the expression of sensory and characteristics for confectionery products. This White Paper describes the VOC analysis capabilities of Selected Ion Flow Tube Mass Spectrometry (SIFT-MS) that are relevant to flavour-release research and manufacturing of confectionery. The application of these capabilities is illustrated, with results from actual analyses presented.

Introduction

Confectionery is generally considered a luxury food item. Consequently, manufacturers continually strive to produce confectionery that enhances the consumer's eating experience, thereby building customer satisfaction and brand loyalty.

In order to understand customer perceptions of flavour, researchers and manufacturers have investigated the flavour chemistry of food. This investigation has presented the need to better understand the release of volatile flavour compounds from food as it is chewed and swallowed. In-mouth flavour release is a very complicated process, because it depends on the properties of the food itself and the physiological characteristics of the person who is eating it. Over the last decade, considerable effort has been invested in the study of *in vivo* flavour release, and substantial work has been published on real-time analysis of flavour compounds as food is eaten.

Selected Ion Flow Tube Mass Spectrometry (SIFT-MS) is a recently commercialised analytical technique that allows flavour release to be measured in real time for simple headspace and *in vivo* applications. SIFT-MS detects and quantifies the volatile organic compounds (VOCs) that are important contributors to the flavour of confectionery products. In this document we discuss the application of SIFT-MS to detection of volatile flavour compounds in several confectionery products. Results from a range of actual SIFT-MS analyses are presented.

SIFT-MS and Its Suitability for Detection and Quantitation of Flavour Compounds in Confectionery

SIFT-MS is a powerful analytical technique that uses chemical ionisation reactions coupled with mass spectrometric detection to rapidly quantify targeted VOCs. VOCs are identified and quantified in real time from whole-gas samples based on the known rate coefficients for reaction of the chemically ionising species (so-called reagent ions) with the target analytes.

The most common reagent ions used are H_3O^+, NO^+ and O_2^+, which react with trace VOCs in well characterised ways but *do not* react with the major components of air. Generally the soft chemical ionisation used in SIFT-MS yields a smaller range of product ions than is common in electron impact mass spectrometry (as used by gas chromatography mass spectrometry (GC-MS), for example). Hence the need for gas chromatographic separation of the sample is circumvented, speeding sample throughput and providing instantaneous quantification of VOCs. Use of several reagent ions to independently quantify target analytes also greatly reduces interferences, markedly increasing the specificity of SIFT-MS versus competing whole-gas analysis technologies.

These characteristics make SIFT-MS ideally suited to rapid, sensitive detection and quantification of the volatile flavour compounds in many types of confectionery. Several examples of the application of SIFT-MS to analysis of confectionery products are given below. Unless otherwise stated, Syft Technologies Voice100™ SIFT-MS instruments were used for this work.

Selected Applications of SIFT-MS in the Confectionery Market

Chewing gum

Peppermint, spearmint and fruit flavoured chewing gums (strip format) were obtained in the United States. The dominant flavour compounds are shown in Table 1.

Table 1. Chewing gum flavours and dominant flavour compounds.

Flavour	Dominant Flavour Compounds	Literature Reference
Peppermint	Menthol, menthone, menthyl acetate, menthofuran, and β-phellandrene (a monoterpene)	1
Spearmint	Carvone, limonene (a monoterpene)	2
Fruit-flavoured	Limonene, C_4 to C_7 esters	3

1. H. Maarse, Ed. (1991). Volatile Compounds in Foods and Beverages, Marcel Dekker, New York (pp. 469-472).
2. H. Maarse, Ed. (1991). Volatile Compounds in Foods and Beverages, Marcel Dekker, New York (pp. 475-476).
3. Compounds identified using Full Scan Mode of a Syft Technologies' Voice100™ SIFT-MS instrument.

For *in vivo* analysis, both in-mouth and in-nose analyses were performed. Disposable drinking straws were placed at the inlet of the SIFT-MS instrument, with the other end inserted either in the mouth or in the nose. The instrument drew air from the mouth or nasal cavity for analysis.

Figure 1 shows the Selected Ion Mode in-mouth analysis for the peppermint gum while an untrained volunteer chewed it. The gum was chewed four-times per exhalation and four-times during inhalation. Increased concentrations of flavour volatiles are often observed when the gum is chewed. Similar results were observed for the in-nose experiments, but flavour volatiles were observed at somewhat lower concentrations. Figure 2 shows the in-nose results obtained for the fruit-flavoured gum. Enhancements of signal are observable on certain chews of the gum during the exhalation periods.

Figure 1. SIFT-MS results from in-mouth analysis of peppermint-flavoured chewing gum.

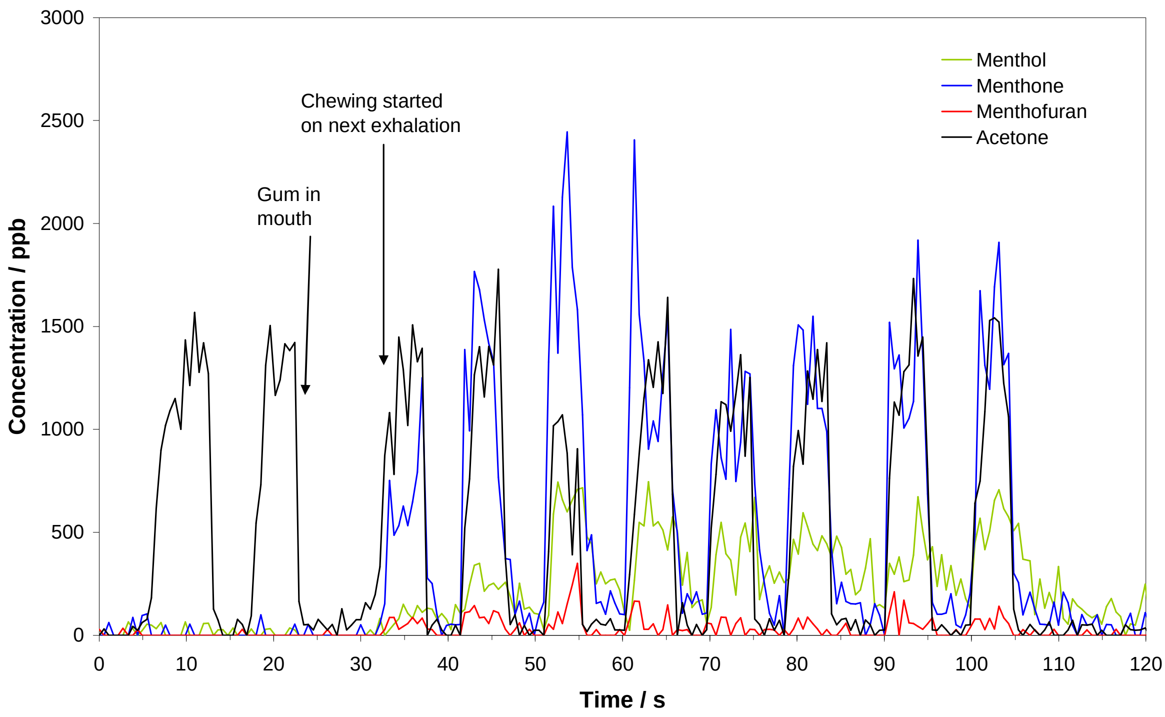
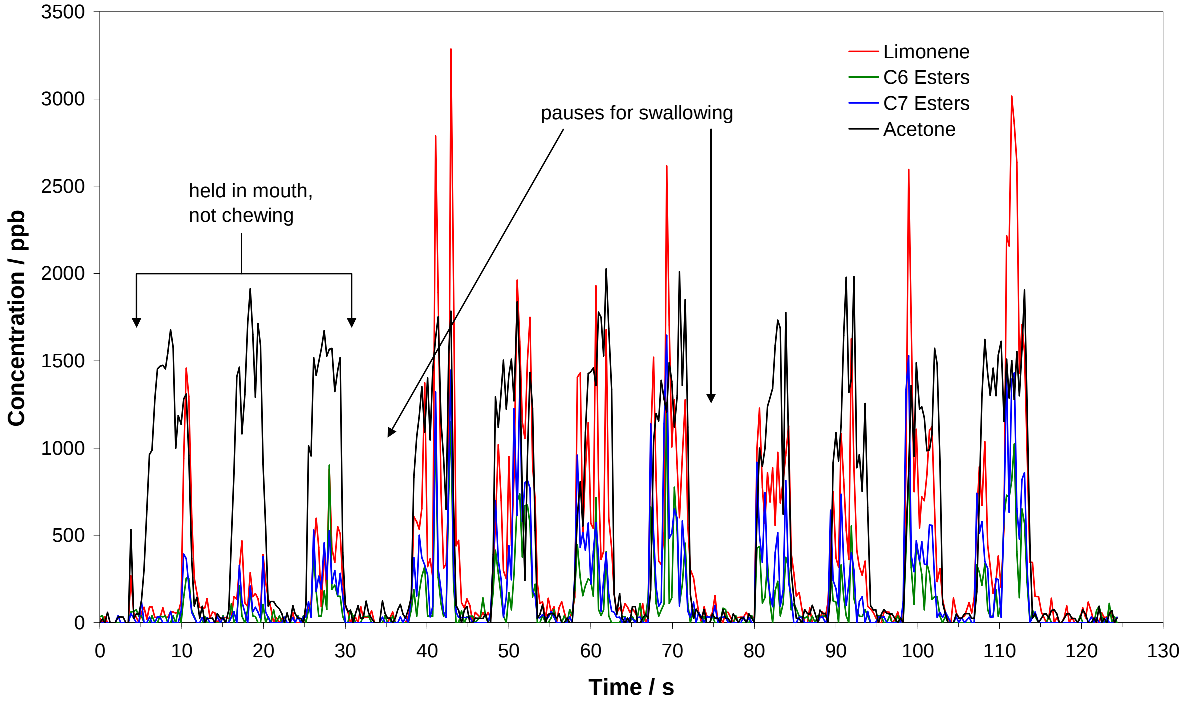


Figure 2. SIFT-MS results from in-nose analysis of fruit-flavoured chewing gum.



Chocolate

Analysis of the flavour and aroma compounds found in chocolate's headspace provides important product information for researchers and manufacturers in the chocolate industry. Figure 3 shows the concentrations of a range of important flavour compounds in several types of chocolate made by different manufacturers and/or having different countries of origin (Table 2). The different chocolates have quite different volatile profiles for this group of compounds.

Moreover, the application of SIFT-MS in the chocolate industry is not limited to the finished product. It can provide manufacturers with benefits in the cocoa bean grading and roasting processes, as well as real-time monitoring of the conching process.

Table 2. Chocolates for which headspace analyses were performed.

Chocolate Type	Country of Origin	Manufacturer	Label on Graph	Cocoa Solids[1]
White	New Zealand	Manufacturer A	White (NZ)	23%
Milk	New Zealand	Manufacturer A	Milk (NZ)	27%
Milk	France[2]	Manufacturer A	Milk (Fr.)	20% min.
Milk	Germany	Manufacturer B	Milk (Ger.)	30% min.
Dark	Australia[3]	Manufacturer A	Dark (Aus.)	70% min.
Dark	Germany	Manufacturer B	Dark (Ger.)	50% min.

1. As stated on packet *2. Purchased in the United Kingdom* *3. Purchased in New Zealand*

Chewy sweets

The volatile compounds released in-mouth from five flavours of a New Zealand brand of chewy sweet were analysed using a Syft Technologies Voice200^{TM} SIFT-MS instrument. Air in the mouth space was sampled via a drinking straw while the sweet was chewed by an untrained volunteer. Figure 4 shows the release of limonene, the dominant flavour, as the orange sweet was chewed, together with the endogenous acetone arising from the volunteer. The high concentration volatiles arising from the orange and banana fruit chews persist in the mouth for several minutes after the confectionery itself had been consumed.

Figure 3. Concentrations of important flavour compounds in the headspace of several chocolates at 50 °C, determined using SIFT-MS. Refer to Table 2 for a fuller description of the chocolate samples.

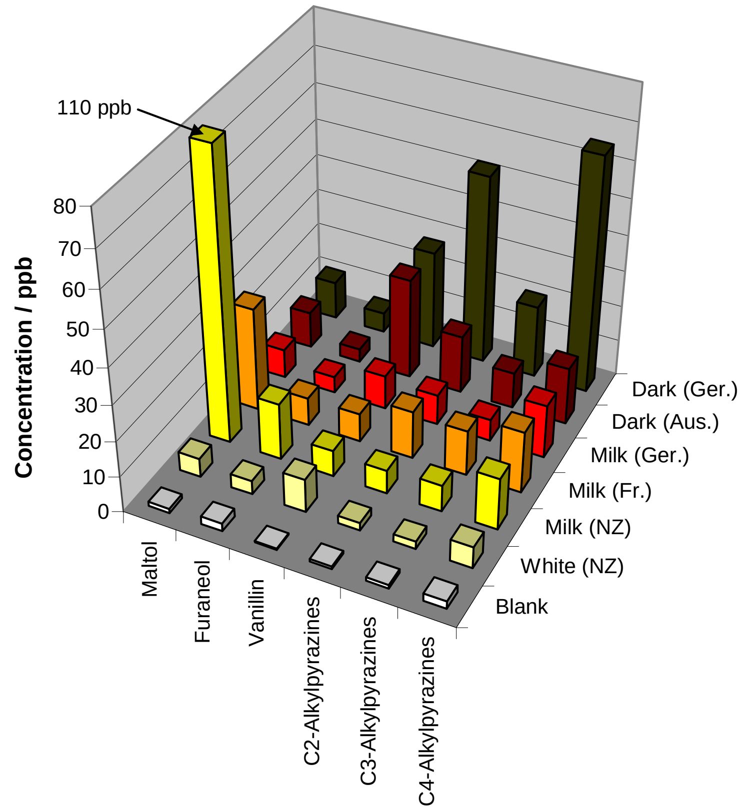


Figure 4. Results of a SIFT-MS analysis of mouth space air while chewing an orange flavoured fruit chew. The first two exhalations provide a control for the following exhalations, which exhibit intense limonene concentrations.

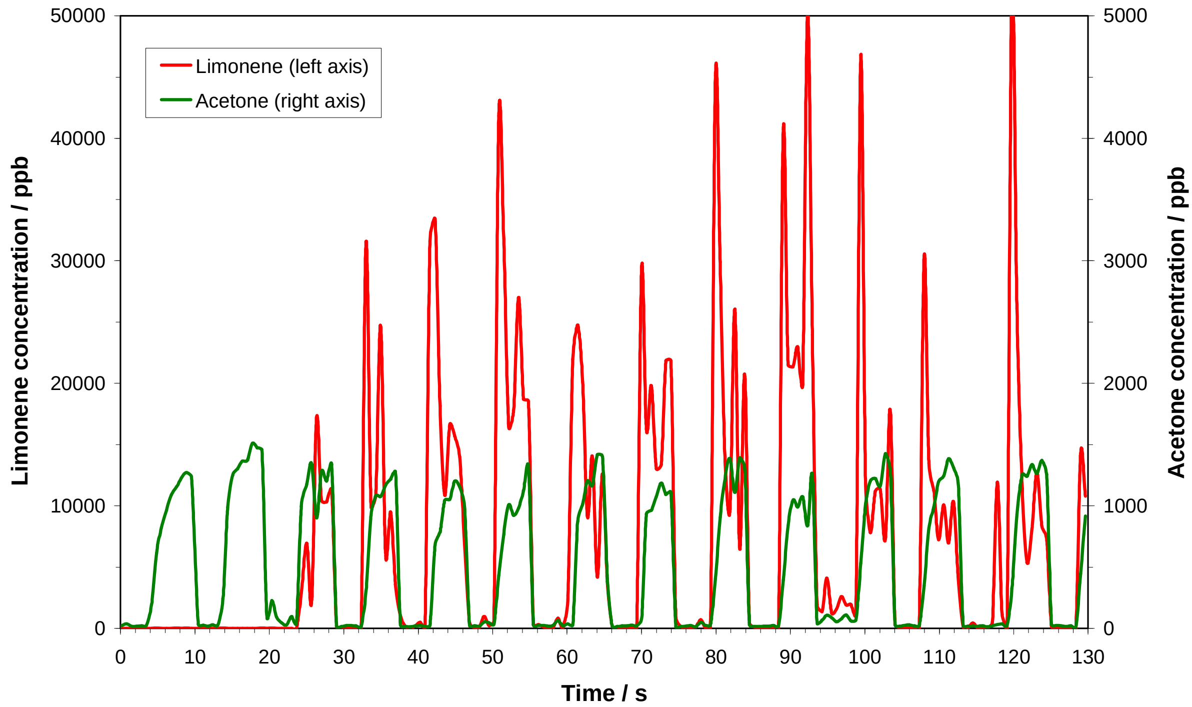
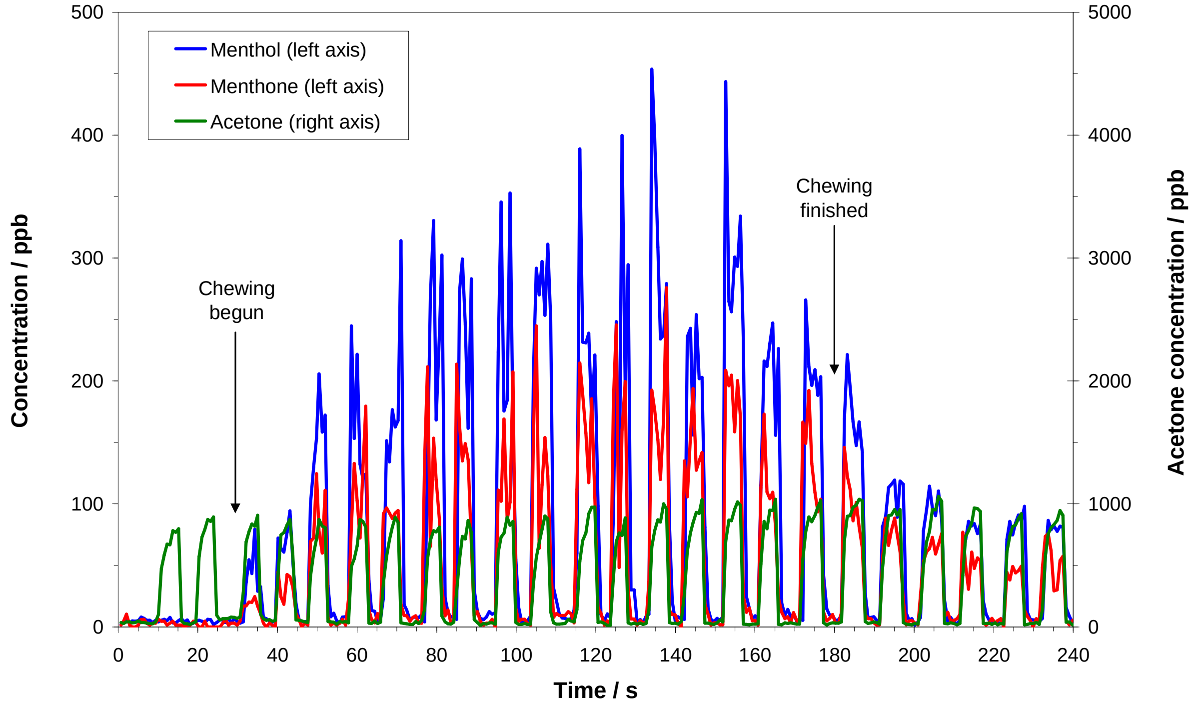


Figure 5 shows the in-mouth release of menthol and menthone from a chewy peppermint-flavoured sweet over a period of four minutes. The concentrations in exhaled breath increase steadily from 30 to 60 seconds to an approximately constant value. There are several extended gaps between exhalations, which were when swallowing occurred. The breaths immediately following swallowing at 115, 130 and 150 seconds on the chart show spikes in the menthol concentration which suggest that the action of swallowing increases the concentrations of certain flavour compounds in the mouth. Chewing was stopped at 180 seconds, when the size of the mint had reduced considerably. It was held in the mouth at this point, and a decay is noticeable to a lower, yet approximately constant concentration.

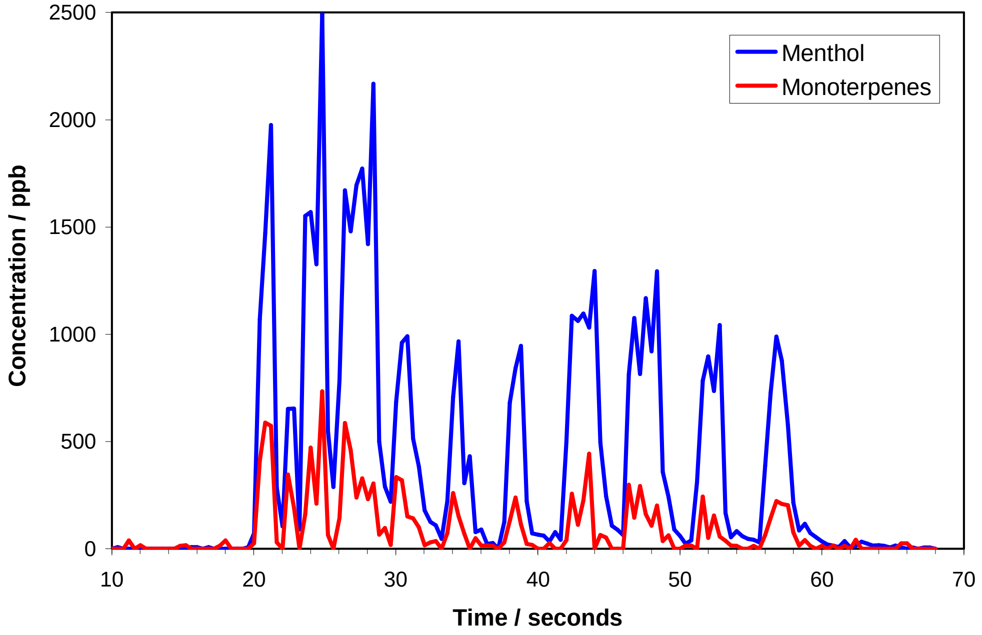
Figure 5. SIFT-MS analysis of the in-mouth release of menthol and menthone from a chewy peppermint-flavoured sweet.



Hard sweets

Flavour release can also be studied for harder forms of confectionery. Figure 6 shows the results of in-mouth analysis of several flavour compounds when a volunteer chewed a small, hard peppermint sweet. The peaks correspond to release of the menthol and monoterpene flavours in unison with the chewing cycle. There is a general trend of diminishing flavour with time for the duration of the sampling (starting at 20 seconds and concluding at 60 seconds). This corresponds to the experienced perception with such sweets, where flavour is strongest in the first few chewing cycles.

Figure 6. SIFT-MS in-mouth analysis for menthol and monoterpene flavour compounds released from a hard peppermint-flavoured sweet.



Summary

The Syft Technologies Voice100^{TM} and Voice200^{TM} SIFT-MS instruments very effectively detect and quantify important volatile flavour compounds in confectionery. Applications include static and dynamic headspace analysis, as real-time analysis of in-mouth and in-nose flavour release.

Further information on SIFT-MS, including background, technical and application specific information, is available on the Syft Technologies website www.syft.com. Alternatively, questions and enquiries can be sent directly to info@syft.com.