Volatile Release from Mint-Flavored Sweets

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The release of aroma from foods depends on the interaction of the food matrix with the volatile compounds present. However, the food matrix is not constant during eating and typically undergoes many changes, such as gel or fat melting, hydration, phase inversion, dilution and comminution. These processes can affect the amount and pattern of volatile release during eating and, consequently, the aroma profile that reaches the olfactory epithelium.

An understanding of these changes and their effects is of major importance to the food industry, which is constantly using new technologies and ingredients (such as encapsulated flavors) in its products. This has led to the development of model mouths and instrumental methods that allow us to monitor aroma compounds in the breath of people as they eat.[1] Only recently, however, have we refined those methods to allow us to follow low concentrations (part per billion by volume) of aroma compounds in the breath in real time (breath-by-breath) as we eat.[2]

These methods use Atmospheric Pressure Chemical Ionization (APCI) mass spectrometry. APCI is a soft ionization technique that typically results in the formation of an ion consisting of the intact molecule plus a proton (MH+). The ions produced from breath in the APCI source enter the quadrupole detector simultaneously, and it is possible to resolve many compounds at the same time providing they have different molecular weights.

The release of volatiles from mint-flavored sweets depends on several factors: the amount of flavoring and how it is added (encapsulated or non-encapsulated), the composition of the flavoring, the texture of the mint and the mint's solubility in saliva. However, the intensity of aroma perception also depends on taste.[3] In the case of mint-flavored sweets, the most important taste compounds will be sugars or artificial sweeteners. Sensory studies performed on chewing gum have shown that the perception of mint aroma from peppermint chewing gum was closely linked to sweetness.[4]

In this paper we report results from instrumental analysis of menthol and menthone in the breath of individuals eating a range of mint-flavored sweets. These results demonstrate the reproducibility of breath volatile analysis for assessing the aroma-release characteristics of mint-flavored products. Sensory (Time Intensity) analysis was also used to study some of the products. These preliminary findings are part of a larger program to investigate the relationship between sensory perception and the concentration of volatiles on the breath.

Experimental

Materials: We tested five commercial mint-flavored sweets from three categories:

- Chewable mints. We selected two: an extra strong mint[a] and a soft mint.[b]
- Suck-and-chew mints. These mints, which have a harder texture than soft mints or extra strong mints, are typically sucked for an initial period and then chewed once they have become thinner. We selected two varieties: Polo original mints[c] and Polo strong mints.[d]
- Chewing gum.[e]

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[a] Trebor Extra Strong Mints, Trebor Bassett Ltd, Maidstone, England
[b] Trebor Soft mints, Trebor Bassett Ltd
[c] Polo Original Mints, Nestlé Rowntree, York, England
[d] Polo Strong Mints, Nestlé Rowntree
[e] Wrigley's Spearmint Chewing Gum, The Wrigley Company Ltd, Plymouth, England

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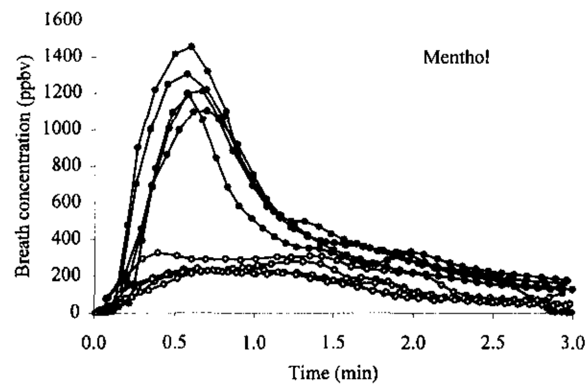

Figure 1. The concentration of menthol on the breath of an individual eating extra strong mints (●) and soft mints (○), five replicates of each

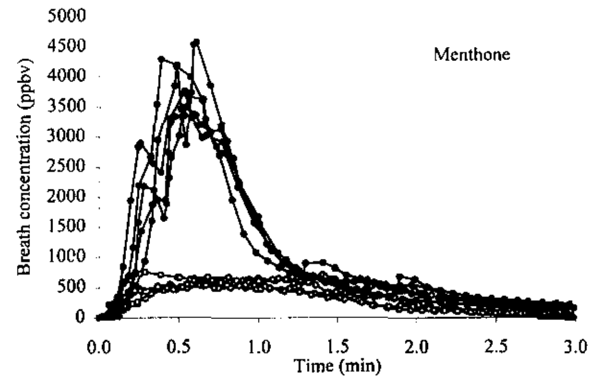

Figure 2. The concentration of menthone on the breath of an individual eating extra strong mints (●) and soft mints (○), five replicates of each

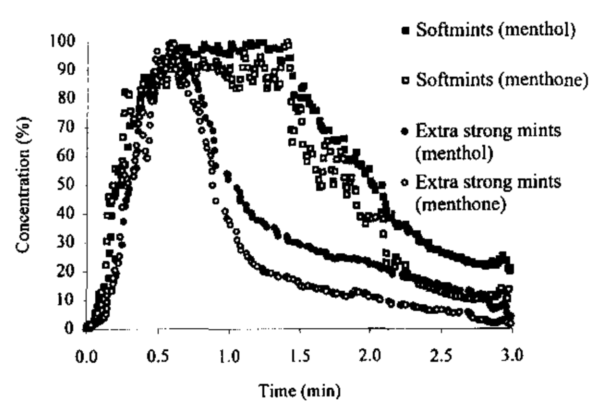

Figure 3. The average, normalized breath profiles of menthol and menthone from Figures 1 and 2

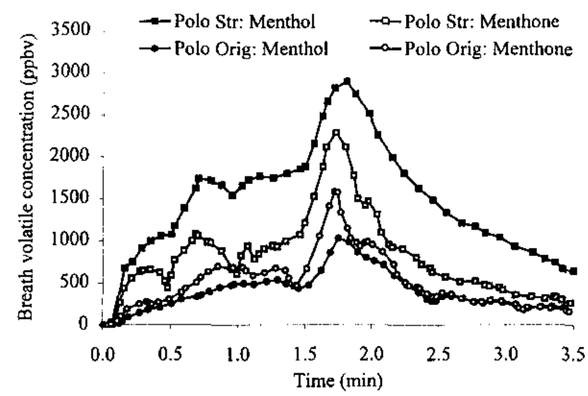

Figure 4. The concentration of menthol and menthone on the breath of one of the six panelists eating an original (Orig) Polo or a strong (Str) Polo mint

All products were purchased from a local supermarket in Nottingham.

Instrumental analysis: The panelists placed the samples in their mouths and ate according to specified eating patterns appropriate to each type of mint. While they ate, an open-ended plastic tube was in place in one nostril to guide the breath over the sampling port of the interface. The plastic tube did not interrupt the normal breathing pattern. During exhalation, breath entered the plastic tube. During inhalation, laboratory air entered the tube.

The gas phase in the tube was sampled continuously at 10 mL/min. through the heated (60°C) interface into the APCI source of the mass spectrometer.[f] There, the volatile compounds were ionized by a 4kV corona discharge before they were sampled into the high vacuum region of the mass spectrometer.

The mass spectrometer was used in selected ion mode to monitor masses specific for the compounds of interest. Calibration was achieved by comparing the peak heights of the compounds present in the breath with the peak heights produced by introducing and vaporizing a hexane solution containing known amounts of the compounds of interest.

Sensory analysis: Six panelists trained in Time Intensity (TI) analysis were instructed to rate the intensity of the overall mint flavor of the sweets by moving a joystick. Prior to the main experiment, the panelists were given samples of extra strong mints to provide them with an example of the maximum intensity of mint flavor they would be likely to experience. The TI data were collected simultaneously with the instrumental data. The output from the joystick was fed directly into one of the analog channels of the mass spectrometer. The resulting TI curves were processed using the software provided with the mass spectrometer.

Volatile Release from Chewable Mints

APCI-MS data: The profiles for menthol and menthone in the breath of one panelist chewing extra strong mints and soft mints are shown in Figures 1 and 2. In these profiles, 0.0 minutes corresponds to the time when the mints were placed in the mouth, and the breath volatile concentration was followed for 3 minutes thereafter. Using ions characteristic for menthone and menthol, these two

[f] Platform II, Micromass, Manchester, England

compounds were monitored simultaneously as five replicate samples of each mint were eaten.

The results (Figures 1 and 2) show that the maximum breath volatile concentrations were reasonably consistent for the different replicates of each type of mint. A person would therefore appear to produce reproducible breath volatile profiles for a single substance during eating. There were, however, substantial differences in the breath volatile concentrations observed when two types of chewable mint were eaten.

Normalized data: Normalizing the combined data to 100% allowed the comparison of the overall shape of the volatile release profiles (Figure 3).

When the two types of mints were eaten, the concentration of menthol and menthone increased in the breath at similar rates (as a percentage of the maximum volatile concentration), until they reached a maximum at 0.5-0.6 minutes. Thereafter, the profiles for the two mints showed very different trends. The experience of the panelist testing the mints was that the extra strong mint had broken down readily on chewing and could be swallowed quickly (in 30-40 seconds), whereas the soft mints were more persistent in the mouth (up to 1.5 minutes). As a result of this persistence, the breath volatile concentration reached a steady state and only declined after 1.4-1.5 minutes when the bulk of the soft mint had been swallowed.

The differences in the shape of the overall volatile release profiles were related to the textural properties of the sweets, which affected the way in which they were eaten.

Volatile Release from Suck-and-Chew Mints

Six panelists were used in the experiments on Polo mints. They were instructed to suck the mints for 1.5 minutes and then to chew the mints, swallowing as necessary. Figure 4 shows an example of the menthol and menthone breath profiles for the two types of Polo mints analyzed (original and strong).

Differences in the concentration of menthol and menthone in the breath of panelists eating the two types of mints were compared at 1 minute after the start of eating (sucking phase) and at the point of maximum volatile concentration (chewing phase). At both of these points in the eating process, the menthol and menthone breath concentrations were significantly higher for the strong Polo mints than for the original Polo mints (t-test, differences in menthol $P<0.01$ and menthone $P<0.05$). The differences were not obscured by the variation among panelists, which might have resulted from different chewing efficiencies, saliva flow rates or breathing patterns. The analysis of samples with more subtle differences would, however, require greater replication or alternative statistical analyses to minimize error associated with such variation.

Changes in both menthol and menthone breath concentration over the timed course of eating were similar. Sucking the two different mints resulted in an initial increase in

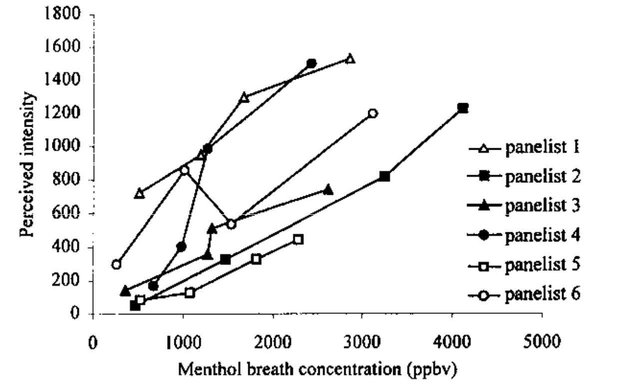

Figure 5. The relationship between the perceived intensity of mint flavor (TI data) and the concentration of menthol in the breath (at selected time points) for six panelists eating original and strong Polo mints

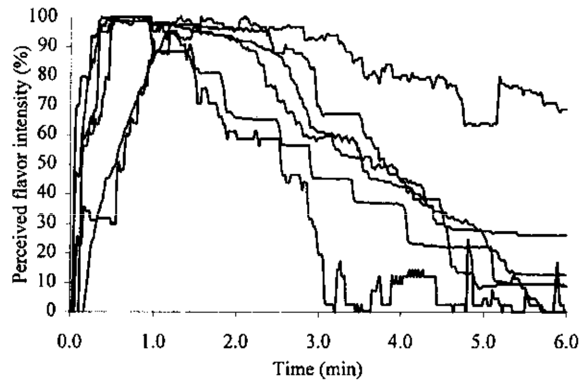

Figure 6. The perceived intensity (TI curves) of overall mint flavor recorded by six panelists chewing a commercial spearmint gum

breath volatile concentration (0.0 to 0.8 minutes) followed by a steady state phase (Figure 4). This ended when the mints were chewed (at 1.5 minutes), causing the breath volatile concentration to increase again. Thereafter the breath volatile concentration declined as the residue of the mints was swallowed.

Sensory analysis data: The panelists also were asked to make simultaneous TI recordings of their perception of the overall intensity of the mint flavor. Each panelist was given a Polo original and a Polo strong. The perceived aroma intensities were compared with the instrumental breath menthol concentration after 1 minute of eating (during sucking) and at the point of the maximum breath volatile concentration (during chewing) for each of the six panelists. Consequently, two instrumental values of breath volatile concentration and the corresponding two sensory perceived intensities were obtained for each panelist (Figure 5). There was an approximately linear correlation between the two sets of data. Mint, however, is a complex multi-component system and consequently menthol would have been only one of many volatile compounds in the breath. The correlation between the two sets of data is therefore only tentative. However, menthol could be considered an indicator of the overall release of volatiles from the mints, and it would have been this total aroma that produced the perceived stimulus.

Volatile Release from Chewing Gum

Chewing gum is typically eaten over a longer time period (5 minutes to several hours) relative to other mint-flavored sweets. Consequently, the rate of aroma release, persistence of aroma and ultimately the development of chewing gums with longer-lasting flavor are of significant commercial interest.

Sensory analysis data: Samples of the spearmint gum were chewed for 6 minutes and the volatiles measured instrumentally while the panelists (six in total) assessed the overall mint flavor by TI. The TI curves showed broadly similar trends (Figure 6). The perceived flavor intensity reached a maximum after 0.5-1.5 minutes and then declined. After 5.0-6.0 minutes of chewing the gum, only one of the six panelists considered the flavor intensity to be greater than 30% of the maximum.

Table I. The concentration of menthol and menthone in the breath (ppbv) for six panelists chewing a commercial spearmint chewing gum. The concentration of compounds in the breath was determined for the 0.6-1.4 min, 2.6-3.4 min and 4.6-5.4 min time windows of the eating time course.

	Menthone (ppbv)			Menthol (ppbv)		
Panelist	0.6-1.4	2.6-3.4	4.6-5.4	0.6-1.4	2.6-3.4	4.6-5.4
1	239	214	222	211	190	167
2	190	228	257	157	185	205
3	171	231	249	179	199	200
4	133	132	163	131	122	125
5	292	319	249	234	281	230
6	201	181	178	217	209	167
Average	204	218	220	188	198	182

APCI-MS data: The instrumental data revealed a trend that differed from that of the TI data. The instrumental data in Figure 7 shows that after the initial increase in the concentration of menthol and menthone (0.0-0.5 minute), the average breath volatile concentration reached a steady state and effectively remained constant (Table I). Similar results have been observed by other researchers,[5] who found that many chewing gums release aroma at a steady rate during eating, unless formulated to generate a flavor burst.

An unanswered question: The exact cause of the differences between the sensory and instrumental data remains unclear. It has been reported that the sensory perception of mint flavor from chewing gum is closely associated with the release of sugars from the gum.[4] Alter-

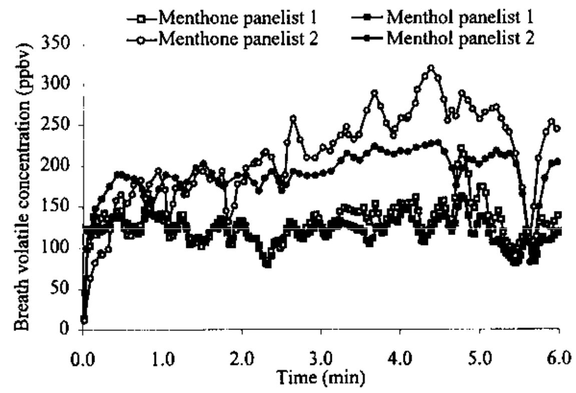

Figure 7. The concentration of menthol and menthone in the breath of two of the six panelists chewing a commercial spearmint gum

nately, processes such as olfactory adaptation[6] may have occurred; that is, the panelists may have gradually adapted to the constant menthol and menthone stimulus present in the breath, thus reducing their ability to perceive them. It is likely that these factors as well as a number of others combined to diminish the perception of the aroma compounds.

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