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Flavor Release Measurement from Gum Model System

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Flavor release from a mint-flavored chewing gum model system was measured by atmospheric pressure chemical ionization mass spectroscopy (APCI-MS) and sensory time−intensity (TI). A data analysis method for handling the individual curves from both methods is presented. The APCI-MS data are ratio-scaled using the signal from acetone in the breath of subjects. Next, APCI-MS and sensory TI curves are smoothed by low-pass filtering. Principal component analysis of the individual curves is used to display graphically the product differentiation by APCI-MS or TI signals. It is shown that differences in gum composition can be measured by both instrumental and sensory techniques, providing comparable information. The peppermint oil level (0.5−2% w/w) in the gum influenced both the retronasal concentration and the perceived peppermint flavor. The sweeteners' (sorbitol or xylitol) effect is less apparent. Sensory adaptation and sensitivity differences of human perception versus APCI-MS detection might explain the divergence between the two dynamic measurement methods.

KEYWORDS: Chewing gum; flavor release; APCI-MS; time−intensity; menthol; peppermint; sorbitol; xylitol; cooling

INTRODUCTION

Chewing gum is a good model to study the release of flavor because it offers the possibility of chewing a semisolid food for a prolonged period of time, releasing flavor compounds progressively in a semicontrolled fashion. According to de Roos and Wolswinkel (*1*) the partition coefficient and the resistance to mass transfer are the major factors determining the rate and extent of flavor release. Partitioning of flavor compounds is affected by the composition of the food, and the resistance to mass transfer by its texture. Large differences in perception of flavor release among persons are reported.

Mint-flavored chewing gum is of special interest because it produces a reaction in the taste, olfactory, trigeminal, and sensory temperature systems. In this study, the chewing gum composition varies in peppermint concentration and the use of sorbitol or xylitol as sweetener. Schiffman et al. (*2*) studied structural and perceptual differences among sweeteners, concluding that sorbitol is similar to maltose and xylitol to fructose and glucose.

Peppermint oil is a plant-derived essential oil containing predominantly menthol and menthone. L-Menthol has a characteristic peppermint flavor and produces a cooling or burning sensation when applied to skin and mucosal surfaces. The cooling properties of menthol have been studied extensively (*3*, *4*).

To achieve an understanding of the dynamic process of food flavor perception, it is necessary to apply time-resolved research methods (*5*). Atmospheric pressure chemical ionization mass spectroscopy (APCI-MS) instrumental time profiles and sensory time−intensity (TI) are methods with high time resolutions. Harvey and Barra (*6*) recommended that one of the areas of application of the APCI-MS is to use it simultaneously with sensory evaluations to identify the factors most affecting flavor perception. TI consists of the continuous assessment of intensity of a certain product for a certain period of time. Data analysis of TI curves is complex due to the amount of information contained in them. A recent comparison of TI data analysis methods can be found in Ovejero-López et al. (*7*).

On the basisi of direct sampling of expired air into an APCI-MS interface, Linforth and Taylor (*8*) have described real-time measurement of volatile flavor compounds in the human breath. The resulting breath-by-breath profile is thought to be very similar to retronasal perception (*8*). Several studies have compared APCI-MS and sensory TI tests simultaneously. Linforth et al. (*9*) measured the release of volatiles from gels. They averaged the instrumental time release (TR) and sensory TI curves before making comparisons. The TR and TI curves were found to be very similar, suggesting a simple linear correlation between the chemical stimulus and the perceived sensory intensity. Baek et al. (*10*), in a model-gel study, averaged the sensory TI data using a method similar to the one

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Table 1. Factorial 2^2 Design of Chewing Gums Used in Chewing Experiments with Release Measurements by APCI-MS and Sensory TI

	product 1	product 2	product 3	product 4
peppermint oil (w/w %)	0.5	2.0	2.0	0.5
sorbitol (w/w %)	50	50		
xylitol (w/w %)			50	50

described by Overbosch et al. (*11*)—first normalizing in the intensity direction to the numeric average of maximum intensity, followed by averaging in the time dimension. They found that in their model system an adaptive (sensory saturation) phase occurs after 0.6 min. Taylor et al. (*12*) found that the concentration of menthone in expired air from the nose reached a plateau value. They also measured the concentration of sucrose in-mouth, where it peaks just before 1 min and then declines. The sensory TI for change in mint flavor intensity with time followed the same pattern as the one from sucrose in-mouth. They gave three possible explanations: the first one is adaptation, the second one is that there is a perceptual link between sucrose release and mint flavor perception, and the third one is that the panel became confused and followed sweetness rather than mint flavor. Weel et al. (*13*) performed a study in flavored whey protein gels. They extracted and analyzed the $I_{\max}$ and $T_{\max}$ parameters from the curves. They found that the "panelist" factor was highly significant and that the variance also differs between panelists according to the values obtained from the Levene test for heterogeneity. Schober et al. (*14*) calculated the mean curve of six APCI-MS replicates (triplicate measurements from two panelists) and then smoothed them by a 6-s moving average. They averaged the sensory TI curves before interpretation. Normand et al. (*15*) emphasized the need of using acetone as internal standard when analyzing APCI data. Acetone is produced by fat metabolism in the liver and then passes into the blood. It partitions from the blood stream into the lungs and therefore into the exhaled air. They claimed that a major problem in interpreting raw nosespace data is that the signal is proportional to the amount of flavor per unit time, which is the product of the air flow rate and the flavor concentration. They reported that an unusually large peak could be due to either higher flavor concentration or faster exhalation. Thus, after normalization, the peak area is proportional to the average concentration of flavor released. Using the internal standard this way removes the ambiguity in interpretation of the data.

The present study is aimed at comparing the time–intensity curves from both APCI-MS release measurements and sensory (TI) measurements using gum-based model systems. Differences in release profiles are assessed both at the individual subject level and at the group level. Effects of peppermint oil concentration and type of sweetener (sorbitol or xylitol) on volatile release and sensory perception are presented and discussed.

MATERIALS AND METHODS

Samples. The model chewing gums were produced especially for this experiment by ChewTech A/S, Vejle, Denmark. The gums (weight = 960 ± 2 mg) were manufactured without sugar coating. The peppermint oils used to deliver flavor contained about equal amounts of L-menthol and menthone. The level of peppermint oil was 0.5 or 2.0 w/w%. The level of either sorbitol or xylitol was 50 w/w %. The experimental design of the four gums is given in **Table 1**.

Subjects. Nine people participated in the APCI-MS experiment, five females (ages 25–46 years) recruited from among the staff at the Danish Institute for Fisheries Research, Denmark, and two males and two females from the permanent sensory panel of the Royal Veterinary and Agricultural University (KVL), Denmark.

Ten people participated in the TI study (ages 24–63 years, four males and six females), all from the permanent KVL panel.

Four subjects of these two panels (identified by A, B, C, and D) thus participated in both APCI-MS and sensory TI evaluations; they will be highlighted throughout the paper. All subjects were paid for participation.

Measurement of Volatile Compounds from the Nasal Cavity (APCI-MS). Menthol and menthone from the peppermint oil in the gum were measured using an ion trap mass spectrometer (Agilent 1100 LC/MSD trap 4.0 coupled with MSD Trap Control software v. 6.08 and a Bruker Daltonics GmbH revision code A.08.03, Frankfurt, Germany) with an atmospheric pressure chemical ionization source (APCI). A modified interface was applied for the on-line measurement of the breath from the nose (*16*). Volatile compounds were ionized by a corona discharge of 4 μA. Samples were scanned from m/z 15 to 350 (mass-to-charge ratio) with an accumulation time of 300 ms in the trap. The releases of menthol, m/z 139 ($MH^+ - H_2O$) and menthone m/z 155 (MH^+) were followed over time. To ensure that the subject was breathing properly through the nose during the experiment, the release profile of acetone (m/z 59 − MH^+) was routinely monitored. The daily and weekly repeatability of the APCI-MS instrument is <10% performed in vitro for menthol and menthone at the concentration levels used in this study (*16*).

The subjects were instructed to have breakfast the day of the experiment and to refrain from drinking and eating for 1 h before arrival at the laboratory. The experimental sessions started at 9:00 a.m. and ended at 1:00 p.m. Each session included eight series of chewing gums (duplicated measurements for each of the four products) with a 20-min break between series. One series lasted 6 min, and the series differed only with respect to the type of chewing gum. Subjects were instructed to chew with their habitual chewing frequency. Furthermore, they were instructed to sit in a relaxed position with their nose in the air-sampling funnel of the APCI-MS-instrument to sample the expired air (*16*). Subjects were instructed to have their mouths closed during the chewing cycle.

One assessor did the experiment twice, resulting in a total of 10 measurement series. Three measurement series were recorded incorrectly and removed from the experiment: one time for product 1 and twice for product 2.

Measurement of Sensory-Perceived Time–Intensity (TI). The TI panel was trained in accordance with Peyvieux and Dijksterhuis, paying special attention to chewing rhythm by use of a metronome and consistently using the same chewing side (*17*).

The experiments consisted of the assessment of five different attributes in the four different chewing gums. The attributes were "peppermint flavor", "cooling without nose clip", "cooling with nose clip", "sweet taste", and "hardness".

Each chewing gum was assessed in random order for 6 min with electronic recordings every 1.5 s. Before each session the metronome was used as a reminder, but during the session the metronome was not used. After each assessment, the assessors had a 5-min pause during which they could consume crackers, water, and/or cucumbers to reduce carry-over effects. In one session the subjects would evaluate only one attribute, in blind duplicate for all four products (a total of eight tests; they got twice each chewing gum). Each session lasted about 1.5 h. Assessors were given a 10-min break in the middle of the session. The experiment took place in sensory booths designed according to ISO guidelines (*18*). All sessions for all attributes were repeated once, providing four replicates of all assessments (further details in ref *7*).

Data Analysis. The raw data from the APCI-MS measurements were preprocessed before further analysis by principal component analysis (PCA). The preprocessing was necessary to account for the differences within subjects for the acetone levels. To partially eliminate these artifacts, APCI-MS component release time profiles (menthol and menthone) were expressed by ratio scaling them on the acetone time profile: each time point of the menthol and menthone signal is divided by the corresponding time point of the acetone signal. This procedure is similar to using acetone as internal standard, strongly recommended

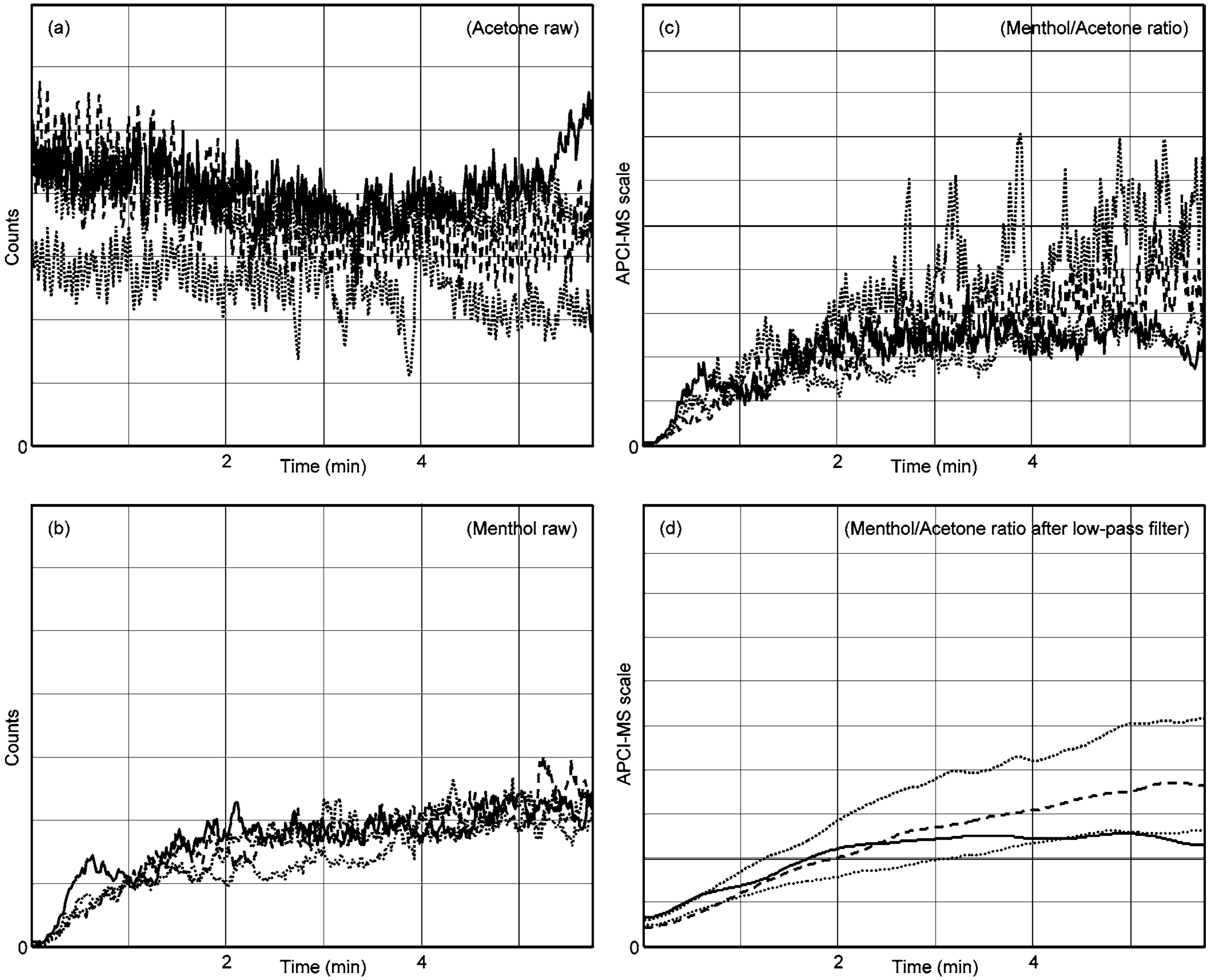

Figure 1. Preprocessing of a typical APCI-MS signal: product 3 for the replicate measurements of subject one (— and ..) and two (.. and - -): (**a**) acetone signal; (**b**) raw menthol; (**c**) menthol/acetone ratio; (**d**) menthol/acetone ratio after smoothing filter. See Material and Methods.

by Normand et al. (*15*). Because the subjects' breathing pattern was undirected, and to further facilitate the interpretation of the data, a further processing of the time signals by a low-pass filter was performed before the two replicates for each individual were averaged.

Before averaging over the four replicates, the panel TI profiles were smoothed by a weak low-pass filter. The basic working principal of low-pass smoothing filters used in this paper can be illustrated by the following equations, where signal vector $\mathbf{z}$ represents the raw data from a time series and vector $\mathbf{z}_f$ is its smoothed or filtered counterpart (*19*):

$$\text{minimize } (|\mathbf{z} - \mathbf{z}_f|^2 + \lambda|\text{differentiate}(\mathbf{z}_f)|^2)$$

$$\text{minimize (“distance” + “roughness”)}$$

The filter tries to minimize the distance between the original signal and the smooth version, penalizing fast changes in the signal (noise/roughness). The tuning parameter λ can be adapted to get a satisfactory result.

PCA finds the best least-squares low-rank approximation of a data matrix $\mathbf{X}$ (*20*)

$$\mathbf{X} = \mathbf{u}_1 s_1 \mathbf{v}_1^T + \mathbf{u}_2 s_2 \mathbf{v}_2 + \mathbf{E} = \mathbf{USV}^T + \mathbf{E}$$

$$\text{minimize}||\mathbf{X} - \mathbf{USV}^T||^2$$

where the matrix product $\mathbf{USV}^T$ is the (in this paper two-factor) singular value decomposition, $\mathbf{S}$ is a diagonal matrix, and $\mathbf{E}$ is the unmodeled part of $\mathbf{X}$. In this work $\mathbf{X}$ is assumed to be column mean-centered before analysis. To eliminate redundancy in the decomposition, the singular values are usually included in the object score matrices

$$\mathbf{X} = \mathbf{t}_1\mathbf{p}_1^T + \mathbf{t}_2\mathbf{p}_2^T + \mathbf{E} = \mathbf{TP}^T + \mathbf{E}$$

where object score vectors fulfill $\mathbf{t}_i\mathbf{t}_i^T = s_i$ and $\mathbf{t}_i\mathbf{t}_j^T = 0$ and variable loadings define criteria $\mathbf{p}_i\mathbf{p}_i^T = 1$ and $\mathbf{p}_i\mathbf{p}_j^T = 0$. Hence, the first set of scores and loadings is the best approximation of the original data, and the percentage explained variance captured from the original data matrix by this first pair expresses how well this approximation succeeded. Similarly, the second pair is the next best approximation. The scores can be seen as new pseudo-values for the objects (panel members for different products in this paper). The loadings show the role of the original variables (time points on the APCI-MS and TI time axis for this paper). The variable loading can be interpreted as a weighted average of the time profiles, averaged over all of the panel members.

In the supervised classification method of linear discriminant analysis (LDA) a line is sought to maximally separate two groups (the so-called Fisher discriminant function; *21*). As an example the separation between sorbitol (s) and xylitol (x) gums in the PCA two-factor score space is used: split the products in the two classes $\mathbf{T}_s$ and $\mathbf{T}_x$ and compute the averages (avg) for groups $\mathbf{t}_{s,avg}$ and $\mathbf{t}_{x,avg}$ and the combined variance–

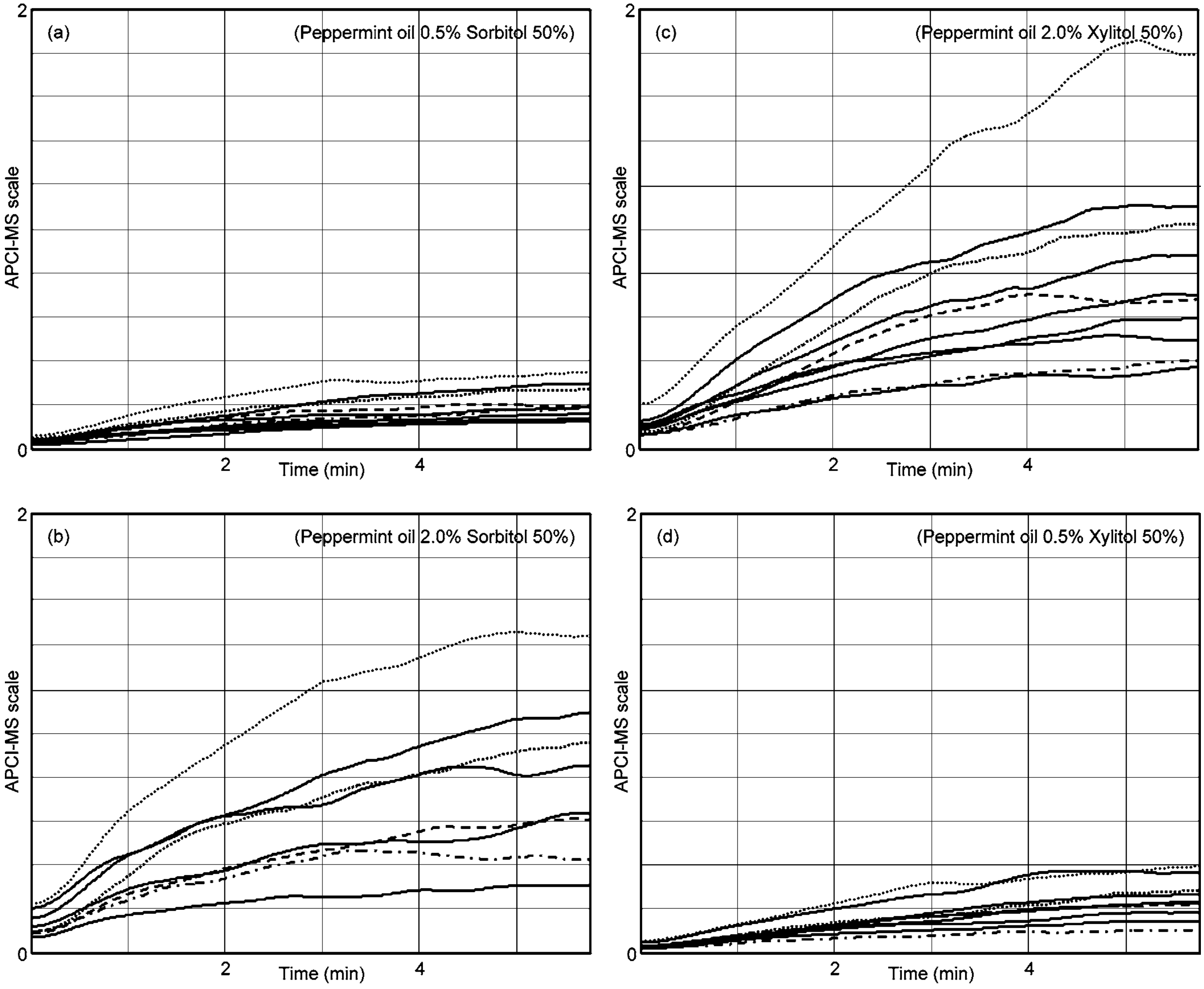

Figure 2. APCI-MS signal for menthol from individual subjects sorted by gum type: (**a**) product 1; (**b**) product 2; (**c**) product 3; (**d**) product 4. Subject coding: (..) A; (**..**) B; (- -) C; (-.) D.

covariance matrix of the object scores $\mathbf{C}_{s+x}$. From these entities compute the classification or membership line m

$$m = \mathbf{t}_{\text{unknown}}\mathbf{w}^{\mathrm{T}} + w_0$$

$$\mathbf{w} = (\mathbf{t}_{\text{s,avg}} - \mathbf{t}_{\text{x,avg}})(\mathbf{C}_{s+x})^{-1}$$

$$w_0 = -0.5(\mathbf{t}_{\text{s,avg}} - \mathbf{t}_{\text{x,avg}})(\mathbf{C}_{s+x})^{-1}(\mathbf{t}_{\text{s,avg}} + \mathbf{t}_{\text{x,avg}})^{\mathrm{T}}$$

If m for the unknown sample is >0, it is classified as "sorbitol"; if m is <0, it is classified as "xylitol". Reclassifying the known/training samples gives an indication of how well the classes can be grouped by a simple straight line; the line $c = 0$ can be plotted together with the object sores for visual clarification.

The so-called RV coefficient is one way to quantitatively express the (dis)similarity between two matrices (*22*). This number is similar to the correlation coefficient for two vectors, working on column-centered matrices of equal size

$$\text{RV coeff} = \text{trace}(\mathbf{X}^{\mathrm{T}}\mathbf{Y})/[\text{trace}(\mathbf{X}^{\mathrm{T}}\mathbf{X})\text{trace}(\mathbf{Y}^{\mathrm{T}}\mathbf{Y})]^{0.5}$$

It is a number between 0 and 1, where 1 indicates perfect correlation/agreement between the different sets of observations.

To maximize alignment of column mean-centered object score matrices from two different PCA models $\mathbf{T}_a$ and $\mathbf{T}_b$, ithe so-called orthogonal Procrustes rotation (*23*) can be used:

$$\text{minimize } ||\mathbf{T}_a - \mathbf{T}_b\mathbf{Q}||^2$$

$$\mathbf{T}_a{}^{\mathrm{T}}\mathbf{T}_b = \mathbf{U} \times \text{diagonal}(\mathbf{S})\mathbf{V}^{\mathrm{T}}$$

$$\mathbf{Q} = \mathbf{U}\mathbf{V}^{\mathrm{T}}$$

The rotation matrix can be used to process matrix $\mathbf{T}_b$ to create maximum overlap with the matrix $\mathbf{T}_a$.

RESULTS AND DISCUSSION

The raw data retrieved form the APCI-MS measurements were preprocessed before they were submitted to PCA modeling (see Materials and Methods for details on all calculations). Assuming the acetone signal in the breath to be a constant background signal, considerable differences are observed between individuals and replicate runs (**Figure 1a**). These differences can originate both from individual variation, day to day effect, or even from the relatively complicated instrumental interfacing and measurement techniques (**Figure 1b**), and

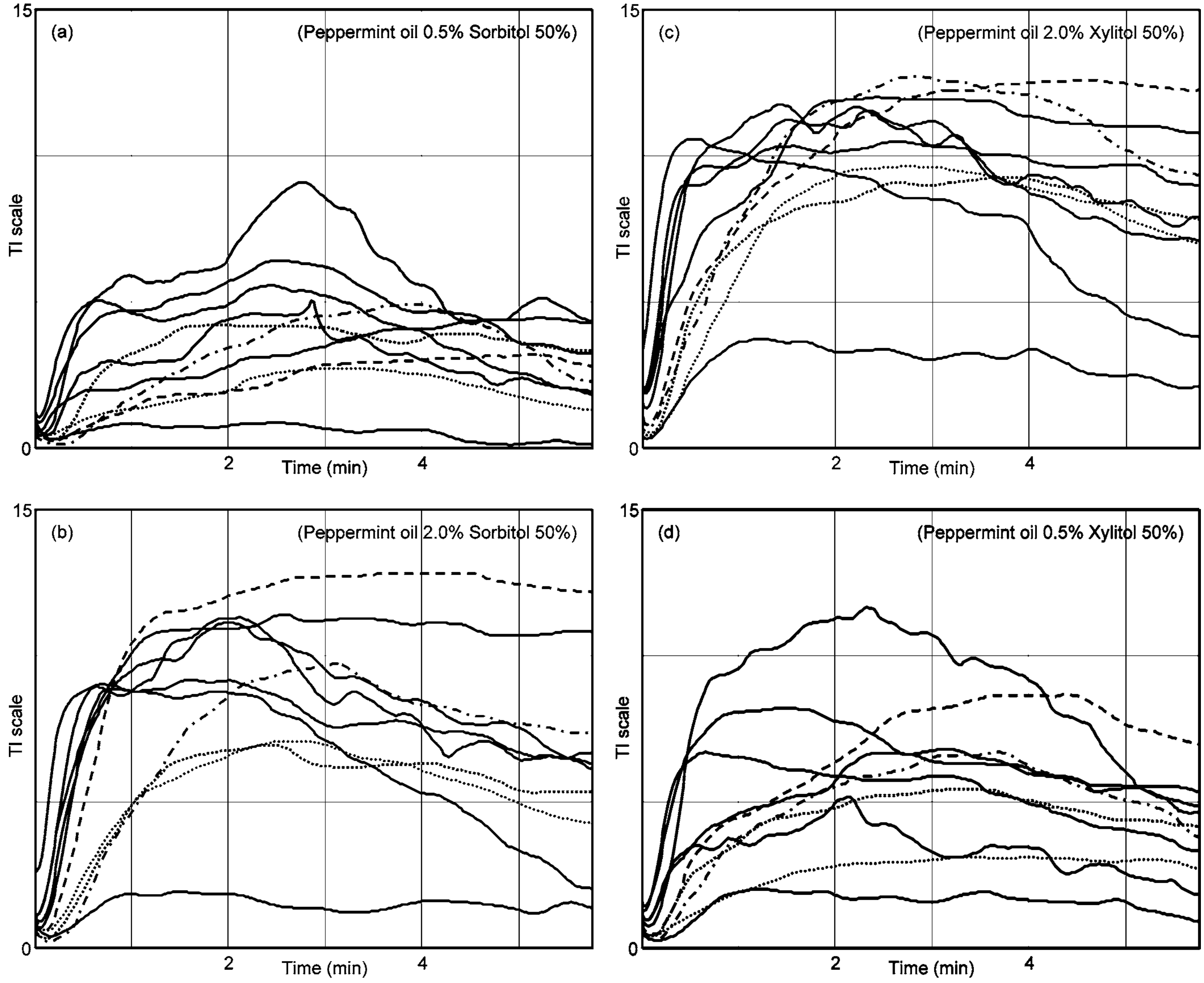

Figure 3. Time–intensity profiles of the peppermint attribute perceived by individual subjects sorted by gum type: (**a**) product 1; (**b**) product 2; (**c**) product 3; (**d**) product 4. Subject coding: (..) A; (**..**) B; (–) C; (-.) D. See Material and Methods.

unfortunately it is not possible to distinguish between these sources of variation. To partially eliminate these artifacts, APCI-MS component release time profiles (menthol and menthone) were expressed by ratio scaling them on the acetone time profile (**Figure 1c**). Because subject breathing was not guided, and to further facilitate the interpretation of the data, an additional processing of the time signals by a low-pass filter (**Figure 1d**; $\lambda = 1000$) (*19*) was performed before the two replicates were averaged. **Figure 2** shows the menthol signals, sorted by gum type, in the form used for further analysis. In a similar plot for APCI-MS, menthone gives similar profiles (not shown here). The difference between low and high peppermint oil concentrations is obvious from the plot. It is also observed that for high concentrations the differences between individuals are considerable for the instrumental APCI-MS.

Before averaging over the four replicates, the sensory time–intensity profiles were smoothed by a weak low-pass filter ($\lambda = 10$). **Figure 3** shows the averaged individual panel member profiles for the peppermint attribute sorted by gum type. Large individual differences can be observed in the sensory TI response. Despite the considerable number of data treatments suggested in the literature for these profiles, no obvious candidate was found for this dataset. It was therefore decided to use the smoothed profiles shown in **Figure 3** in PCA.

Assessors' individual profiles for APCI-MS and TI are, in general, in agreement (**Figures 2** and **3**). Assessor A is scoring high and assessors C and D are around average for both methods. Assessor B's results vary between tests. Furthermore, there is no common tendency at the end of the individual TI profiles in **Figure 3**. Some assessors recorded a plateau after a maximum, wheres others experienced a very strong decrease. This effect is in agreement with findings by de Roos et al. (*1*), who stated that there are large differences in flavor release among people, and also with Weel et al. (*13*), who reported considerable differences in variance between assessors. However, more training of the assessors could help to clarify whether there are differences in perception. In conclusion, differences among assessors are observed by both TI and APCI-MS measurements. This indicates that individual differences in food perception are due to a combination of physiological and psychological factors.

One assessor systematically scored low for the sensory TI results, as can be observed in **Figure 3**. This could possibly be due to insufficient training or incorrect use of the scale. Despite this observation, it was decided not to eliminate this person's results from the dataset to avoid biasing our findings.

The object scores of PCA (*20*) on the APCI-MS release profiles are shown in **Figure 4**. A clear separation between low-

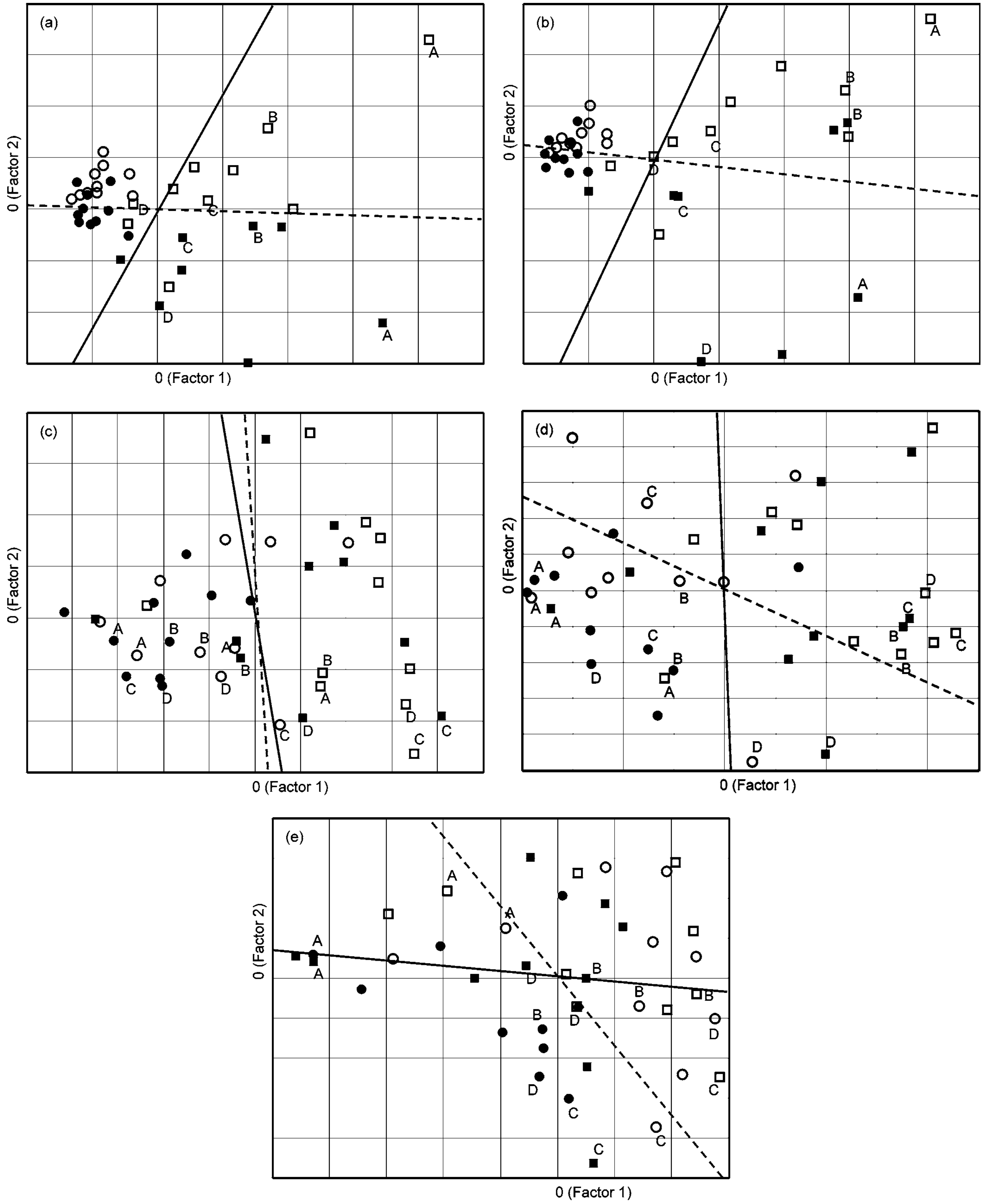

Figure 4. PCA for APCI-MS and time–intensity profile data: (**a**) factor 2 versus factor 1 object scores APCI-MS menthol (●, product 1; ■, product 2; □, product 3; ○, product 4); (**b**) object scores for APCI-MS menthone; (**c**) object scores for peppermint attributes; (**d**) object scores for cooling attribute: (**e**) object scores for sweetness attribute. Subject coding A, B, C, and D placed where possible; LDA separating lines for L–H (—) and S–X (- -) superimposed (see Material and Methods and **Table 2**).

and high-dose peppermint oil can be seen on the first factor for both menthol (**Figure 4a**) and menthone (**Figure 4b**; after Procrustes rotation, see below). The variable-loading vectors for PCA on the APCI-MS data are shown in **Figure 5**. As expected, the variable-loading vectors resemble the average of the signal profiles shown in **Figure 2**, and the object scores

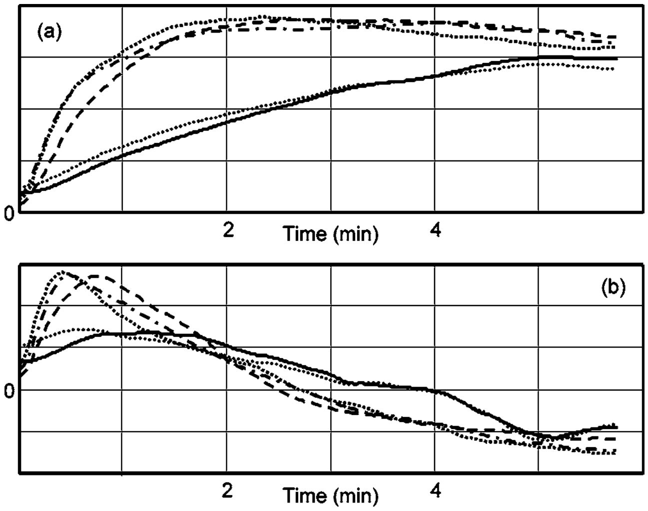

Figure 5. PCA for APCI-MS signals and time–intensity profiles (and cumulative percentage explained variance per extracted PCA factor): (**a**) factor one variable loadings for APCI-MS menthol (—, 99.1%), APCI-MS menthone (.., 99.0%), peppermint attribute (**..**, 88.6%), cooling attribute (- -, 94.4%), and sweetness attribute (-., 78.5%); (**b**) factor two variable loadings for APCI-MS menthol (—, 99.8%), APCI-MS menthone (.., 99.6%), peppermint attribute (**..**, 97.5%), cooling attribute (- -, 98.4%), and sweetness attribute (-., 94.8%).

Table 2. Correct Classification of Low-versus-High Peppermint Oil Concentration[a] and Sorbitol-versus-Xylitol[b] for Different APCI-MS Measurements and TI Sensory Attributes for Two-Factor PCA Object Score Matrices Based on Simple Linear Discriminant Analysis (LDA) Separation Line: Number Correctly Classified/Total Number

measurement (total samples)	L–H (correctly classified)	S–X (correctly classified)
menthol (38)	35	33
menthone (38)	35	30
peppermint (40)	33	25
cooling (40)	33	26
cooling with nose clip (40)	34	25
sweetness (40)	22	29

[a] L–H, products 1 and 4 versus products 2 and 3. [b] S–X, products 1 and 2 versus products 3 and 4.

can be interpreted as an intensity score for this average profile. The second factor (**Figure 4a**,**b**) splits, albeit less clearly, the sorbitol from xylitol gums. Notice that the picture is very similar for menthol and menthone.

Samples with higher peppermint concentration are widely distributed with respect to both factors one and two (**Figure 4a**,**b**). Perception scoring of these samples among and within subjects is more diverse than the samples with low peppermint concentration. To get an objective indication of the separation in gum types, the LDA (*21*) optimal classification or separation lines for low-versus-high peppermint oil dose and sorbitol-versus-xylitol sweetener are superimposed in **Figure 4**. LDA is the simplest classification method, chosen here as an indicative/visual aid only. The dataset is too small to justify more powerful classification techniques. The separation performance is given in **Table 2**, where it is shown that APCI-MS menthol and menthone PCA score values result in a similar, reasonable separation of the gums.

Individually, assessor A shows a clear differentiation between the presence of sorbitol or xylitol in the high-peppermint chewing gums with respect to menthol and menthone. This effect is less pronounced in the recordings of assessor D and almost not present for assessors B and C.

Table 3. RV Correlation Coefficients after Procrustes Alignment between APCI-MS Measurements and Different TI Sensory Attributes for Two-Factor PCA Object Score Matrices

	menthone	cooling without nose clip	cooling with nose clip	sweetness
menthol	0.94			
peppermint		0.66	0.66	0.33
cooling without nose clip			0.85	0.26
cooling with nose clip				0.19

To evaluate the menthol and menthone results, the two-factor object score spaces found by PCA are compared. To eliminate trivial differences in the two-dimensional score spaces of the different models, a Procrustes rotation is used to maximally align each of the PCA models before the plotting and comparison (*22*). The comparison itself is based on the RV coefficient to determine correlation/similarity between the rotated object score matrices (*23*); the RV coefficient is reported in **Table 3**. Menthol and menthone measured concentrations are highly correlated. This could be due to the fact that both compounds come from the peppermint oil added to the chewing gum and that peppermint oil concentration added is the main source of differentiation of the different chewing gums. Although it cannot excluded that the high correlation also is an effect of inefficient signal preprocessing, the approach used in this work was corroborated by the conclusions from Normand et al. (*15*).

The PCA on the panel TI attributes peppermint, cooling, and sweetness is shown in **Figure 4** (after Procrustes correction). Details on the attribute "cooling with nose clip" will not be shown or discussed because they are very similar to the attribute "cooling". The object score plots give similar high-versus-low peppermint dose results as did the APCI-MS data, albeit less well pronounced. The variable-loading vectors for the first factor are again representative of the average TI curves (**Figure 5**).

For the second factor the split in sorbitol versus xylitol is much less clear. By close examination it was further observed that panel member scores for different gums cluster together for the peppermint attribute (**Figure 4c**), more so than for the attributes cooling (**Figure 4d**). This might indicate that panel members were not capable of properly characterizing this attribute. To compare the results over the panel for different attributes, the RV coefficients are reported in **Table 3**. The highest correlation among the attributes is for cooling with and without nose clip, even though both attributes are known to show partly different phenomena (*7*). Sweetness is not correlated to any of the other attributes, thus confirming it to be a different type of attribute. This effect is in disagreement with one of the possible explanations of the results obtained by Taylor et al. (*12*). They proposed there is a perceptual link between sucrose release and mint flavor perception. From the LDA results for sweetness it is observed that—as expected—the separation is primarily induced by sorbitol- versus xylitol-based gums.

Cooling and peppermint taste are partially correlated, in agreement with Ovejero-López et al. (*7*), but still the assessors seem to have clearly different and individual responses for these attributes.

A visual comparison for APCI-MS menthol plus menthone and the TI attributes peppermint, cooling, and sweetness can be made from the variable loadings in **Figure 5**. With respect to the APCI-MS profile, the intensities of both menthol and menthone are increasing slowly during the first 5 min, after which they form a plateau. Taylor et al. (*12*) found a fast increase of menthone concentration during the first minute followed by a plateau value. This difference could just reflect the difference on the chewing gum material composition and properties. The sensory profiles show a fast increase in intensity during the first minute, after which the slope levels out gradually until it reaches a maximum. The increase in perception of the cooling attribute is "slower" than that of sweetness and peppermint taste. Cooling perception is generally slower than other taste or smell attributes as reported in the literature. Peppermint taste reaches maximum intensity between 2 and 3 minutes and then decreases. Sweetness and cooling maximum intensities are reached at around 4 min. Afterward, the decreases of intensity are less pronounced than for peppermint taste.

In general, a fast perception of the TI sensory attributes and then a plateau or weak decrease of the perception are observed, whereas the APCI-MS profiles show progressive increase of menthol and menthone. It seems that human perception is reacting quickly and sensitively to menthol and menthone to a certain level. From then on the sensory perception appears to be in a state of saturation or adaptation. Recovery is not observed in the 6-min recording time for this study. During the recorded 6 min there is no dramatic decrease on sweetness or peppermint taste perception. Thus, the effect observed by Taylor et al. (*12*) when apparently sucrose release drives down the mint flavor intensity when intensity is decreasing is not apparent here.

The APCI-MS data processing proposed in this paper might not be conclusive to eliminate all instrumental artifacts introduced. For example, the strong correlation between APCI-MS menthol and menthone found in **Table 3** might be an effect of inefficient signal preprocessing. However, in the present setup it is not possible to differentiate between (genuine) panel subject differences and experimental errors other than averaging over replicates. This confounding warrants further investigation. Assessors were not recording specific scores for the intensity of menthol and menthone. A higher correlation between APCI-MS and TI may be obtained by simultaneous measurements, longer sensory training, and choosing of alternative attributes to evaluate intensity of menthol and menthone.

Measurement of flavor release from mint chewing gum can be studied by instrumental and sensory tests. The results obtained by both techniques are based on individual profiles rather than group/panel averages as commonly presented in the literature. In general, agreement is found between APCI-MS and TI observations.

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