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How Sweetener Type and Aroma Concentration Shape Aroma Release and Flavour Perception in Sugar-Free Chewing Gum

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Abstract

Sugar-free chewing gums (SFG) rely on added flavourings and sweeteners to compensate for the absence of sucrose. However, the interplay between aroma concentration, sweetener release, and flavour perception remains poorly understood. This study investigated how these factors shape *in vivo* aroma release and flavour perception during SFG consumption. In Experiment 1, model SFGs were prepared with five concentrations (0.2–1.0%) of a *tutti-frutti* aroma mixture. *In vivo* volatile release was monitored by nose-space Proton Transfer Reaction-Time of Flight-Mass Spectrometry (PTR-ToF-MS), while a trained panel assessed aroma and sweetness perception using discrete Time-Intensity (DTI). Higher aroma concentrations significantly increased both in-nose release and aroma perception but did not alter sweetness perception. Aroma perception peaked during the first 2 min of chewing and then declined faster than

instrumental release. In Experiment 2, the release of five sweeteners (sorbitol, mannitol, xylitol, maltitol, and aspartame) was quantified *in vitro* using a chewing simulator and ultra-high-performance liquid chromatography (UHPLC-HRMS). A biphasic release profile was observed: an initial burst (0–120 s) followed by a sustained release phase. Xylitol showed the highest relative release, sorbitol the greatest absolute amount and aspartame was released most slowly. The results show that in this case aroma concentration enhances volatile release but not sweetness perception, and that sweetener release dynamics only partially explain flavour perception. The combined use of PTR-ToF-MS, sensory analysis, and UHPLC-HRMS provides a comprehensive framework to study flavour dynamics in SFG and can guide formulation strategies for improved consumer experience.

Keywords: Flavour perception, chewing gum, aroma release, sweeteners, Proton-Transfer-Reaction Mass-Spectrometry, nose-space analysis

1. Introduction

Sugar-free chewing gum (SFG) is a widely consumed confectionery product, valued not only for its sensory appeal but also for its practical benefits, including breath freshening, stress relief and promotion of oral health. Unlike sugar-containing gum, which can promote cariogenic activity, SFGs have been shown to reduce caries increment and to lower plaque scores compared to non-chewing controls (Newton et al., 2019; Ramasubbu & Duane, 2024, Keukenmeester et al., 2013). In Europe alone, average per-capita consumption exceeds 120 pieces per year, underscoring the product's economic and functional importance (Rychlik et al., 2017). SFG consists of a water-insoluble gum base (comprising elastomers, elastomer solvents and fillers) that serves as the continuous phase, within which a water-soluble discontinuous phase – containing softeners, bulk and high-intensity sweeteners, and volatile organic compounds (VOCs) – is dispersed. Although VOCs typically account for only 0.75–3% of the

formulation, they play a critical role in determining flavour perception and, consequently, chewing duration and overall liking (Hartel et al., 2018). Flavour perception is a multimodal phenomenon that integrates olfactory (aroma), gustatory (taste), and trigeminal sensations. These modalities are influenced by a range of individual physiological and psychological variables, as well as product-related factors, making flavour perception difficult to predict. Chewing gums have been used as ideal model food for exploring the variability in aroma release and perception which has been linked to inter-individual factors such as salivary flow rate, chewing force and speed, saliva composition and bubble formation, sex, age, and ethnicity (Guo et al., 2024; Haahr et al., 2004; Pedrotti et al., 2019), as well as product-related characteristics including gum base composition (Alaçam et al., 2024), VOCs (Potineni & Peterson, 2008b), and the type of sweeteners and polyols used (Raithore & Peterson, 2018).

When focusing on VOCs, factors such as concentration, and physicochemical properties – including solubility, partition coefficient, hydrophobicity, volatility, molecular weight, polarity, and the presence of functional groups – play an important role in aroma release during consumption. Among these, octanol/water partition coefficient (Log P) and the gum-to-saliva partitioning coefficient (Log cP) have been frequently used to predict chewing gum *in vitro* aroma release. In general, more hydrophobic VOCs (*i.e.* higher Log P) are retained longer in the gum base, whereas hydrophilic compounds migrate more rapidly into saliva (Konar et al., 2016; Potineni & Peterson, 2008a; Van Ruth et al., 2000). Despite a substantial body of research on SFG, the effect of aroma concentration on *in vivo* aroma release and perception remains largely unexplored. Potineni and Peterson (2008), have investigated two concentrations of cinnamaldehyde in SFG, focusing solely on release kinetics without any sensory evaluation. To complicate the picture, the relationship between *in vivo* aroma release, perception and concentration is modulated by a range of compound-specific and contextual factors. Key determinants include mass transport from the gum base to saliva and partitioning between saliva

and the air phase, both governed by the physicochemical properties of the aforementioned VOCs (Fisk, 2015). Additionally, odour thresholds and inter-individual physiological variability further complicate the prediction of sensory responses (Repoux et al., 2012). Moreover, *in vitro* mastication models demonstrated that over 80% of VOCs can remain entrapped in gum matrix after 20 minutes (Krause et al. 2011). Similarly, Hinderink and colleagues (2019) showed that less than 4% of the aroma was released after 15 minutes of mastication with an artificial mouth. For *in vivo* mastication, between 81-93% of the initial concentrations of different aroma compounds remained in the gum bolus after 12 minutes of mastication (Raithore & Peterson, 2016). Finally, sugars and sweeteners in chewing gum further influence flavour dynamics: bulk polyols (70-85% of the SFG formulation) and high-intensity sweeteners (HIS) (0.005-1%) each influence aroma release and perception primarily through their relative sweetness intensity and solubility, which affect aroma release during consumption (Hartel et al., 2018). For instance, xylitol yielded a stronger perceived mint intensity from L-menthol and menthone (2%) than sorbitol (A.-M. Haahr et al., 2003) while the polyol choice (mannitol, sorbitol or xylitol) and particle size resulted in different concentrations of aroma compounds in the nose cavity of participants consuming chewing gum (Fisker & Nissen, 2006; Raithore & Peterson, 2018). Beyond physicochemical effects, specific odour-taste interactions may also occur modulating sweetness perception (Thomas-Danguin et al. 2016). However, while most SFG studies have focused on individual factors, the combined effect of aroma concentration and the simultaneous release of aroma compounds and sweeteners on flavour perception remains poorly understood. Therefore, the aim of this study was to examine how the concentration and release of a "*fruity*" aroma, as well as the release of different sweeteners from SFG, influence aroma release and flavour perception. Nose-space analysis by Proton Transfer Reaction Mass Spectrometry (PTR-MS) was combined simultaneously with dynamic sensory analysis to characterize *in vivo* aroma release and perception of SFG with five

different aroma concentrations. *In vitro* release of sweeteners from one of the samples was investigated by combining an artificial chewing device and ultra high-performance liquid chromatography (UHPLC-HRMS) to evaluate the influence of sweeteners solubility and total sweetener release on flavour perception. The research hypotheses were that increasing aroma concentration in the chewing gums would proportionally increase *in-vivo* aroma release, due to a greater mass transfer from the gum matrix to saliva and through the retronasal pathway to the olfactory epithelium. Consequently, a higher aroma perception was expected, although modulated by perceptual thresholds. In contrast, sweetness perception was expected to remain relatively constant, with only minor increases at higher aroma concentrations due to potential taste–aroma interactions. Regarding sweetener release, distinct kinetic patterns were anticipated based on the known physicochemical properties and molecular structures of the sweeteners studied, particularly between bulk sweeteners such as sorbitol and high-intensity sweeteners such as aspartame.

2. Materials and methods

2.1 Experimental design

This research consisted of two experiments. Firstly, the influences of aroma concentration and physicochemical characteristics of aroma compounds on aroma release and perception were explored (Experiment 1). Expert panellists (n=6) evaluated through dual-attribute discrete time intensity (DTI) the aroma and sweetness intensity of the same chewing gum base containing a fruity aroma (TF: "*tutti frutti*") in five different concentrations (0.2%, 0.4%, 0.6%, 0.8%, 1.0%). Simultaneously, aroma release was measured by PTR-MS *in vivo* nose-space analysis. Samples were tested in triplicates in three different sessions organized over three days.

In the next experiment, sweeteners release was studied for the sample containing 0.6% TF aroma to evaluate the influence of sweeteners solubility and total sweetener release on aroma perception (Experiment 2). Saliva samples were collected using an artificial chewing device, at

different time intervals (15, 30, 60, 120, 180, 300 and 420 sec). Sorbitol, mannitol, xylitol, maltitol and aspartame were quantified through UHPLC-HRMS. Each sample for each time interval was collected and analysed in triplicates.

2.2 Materials

Tailor made chewing gum samples without sugar coating were obtained from the industrial partner. Experiment 1 samples consisted of gum base (30%), sweeteners (sorbitol 48%, mannitol 5%, xylitol 5%, maltitol 10%, aspartame 0.2%), glycerol (0.2%), citric acid (1.0%), and TF aroma at five concentrations (0.2%, 0.4%, 0.6%, 0.8%, 1.0%). The 0.6% TF aroma sample was also used in Experiment 2. This sample was selected as it represented an intermediate concentration level and made it more reflective of typical industrial samples. The main molecules of TF aroma consisted of ethyl acetate (11%), ethyl butyrate (34%), isobutyl acetate (8%), isoamyl acetate (34%), and vanillin (7%). In Table 1, an overview on the physical, chemical, and physicochemical properties of the aroma compounds is given. The manufacturing of the chewing gum samples consisted in melting and softening gum base pellets in a forced-air oven at approximately 60 °C. The softened base was transferred into a kettle mixer and processed with mechanical blades. Once adequately softened, the remaining ingredients were incorporated into the matrix. The TF was then added at a controlled temperature of approximately 40 °C to preserve volatile properties. The resulting mixture underwent a cooling phase, was rolled with a rolling pin, and cut into individual sticks of ~2 g each. Samples were stored at 4 °C until further use.

Table 1 here

2.3 Participants

In Experiment 1, three men and three women (mean age ± SD = 30.5 ± 5.8 y.o., mean weight ± SD = 68.8 ± 15.5 kg) with prior experience in PTR-MS nose-space measurements, took part

in the study. All were healthy non-smokers, not pregnant, fully dentate and free from oral inflammatory conditions such as gingivitis and periodontitis. To further control for major sources of interpersonal variability known to influence nose-space signals, participants were screened for stimulated salivary flow and oral cavity volume and completed the Italian version of the state anxiety subscale of the State–Trait Anxiety Inventory Form Y (Pedrabissi and Santinello, 1989) immediately prior to the analysis. Panel homogeneity was further supported by the absence of outlier values (defined as ±1.5 SD from the mean) across all screening measures and by all participants reporting "(e.g., no or low)" state anxiety levels (scores 20–37) (Pedrabissi and Santinello, 1989).

As the current protocol involved the same participant tasks as our previous study (Pedrotti et al., 2019), which had been formally reviewed by the Wageningen University Ethics Committee and deemed free of any potential discomfort or ethical concerns, no additional evaluation was requested. Informed written consent was obtained from all participants in accordance with the European General Data Protection Regulation (EU 679/2016), and the study was conducted in accordance with the principles of the Declaration of Helsinki (most recently amended in Fortaleza, Brazil, 2013).

2.4 Sensory assessment

The dual-attribute DTI method was used to evaluate sweetness and aroma perception. DTI is a temporal sensory method designed to assess one or multiple attributes simultaneously at fixed intervals within a defined timeframe (Delarue & Loescher, 2004), making it particularly suitable for long-lasting products such as chewing gum (Hort, 2017). Sweetness and aroma intensity (fruity) were selected as the most appropriate and relevant attributes to describe their sensory profile.

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All panellists joined an information meeting and received three trainings prior to the experiment about the sensory method, the scales and their usage (including some references for the anchors: for aroma: a chewing gum without flavour and a commercial chewing gum with a strong fruity flavour; for sweetness: water and a water solution with 1 gr sucrose/mL of water) for the two evaluated attributes and about the fixed chewing protocol that was adopted to reduce inter-individual variability. A metronome counting 60 beats per minute (bpm) was used to practice the chewing rhythm of one chew/sec. For breathing, no restrictions were given.

Participants were asked to refrain from drinking alcohol 12 hours prior to the experiment and from brushing their teeth, wear perfume, eat or drink anything besides water at least two hours prior to the experiment. During Experiment 1, dual-attribute DTI sensory analysis was carried out in this study simultaneously to PTR-MS nose-space analysis. Samples were presented with three-digit random codes and were randomized across participants and sessions. At discrete time points, panellists were asked to rate the perceived intensity of sweetness and aroma on a linear scale ranging from 0-100 (Experiment 1). Both attributes were evaluated after 15, 30, 60, 120, 180, 300, 420 and 480 sec. At every evaluation the order of appearance of the two attributes was randomized. Panellists were asked to swallow excess saliva prior to rating the intensities. After each sample, participants rinsed their mouth with water and a plain cracker. A break of at least 20 minutes was taken before performing other evaluations to avoid carryover effects and to mitigate sensory fatigue. Data were collected using the EyeQuestion Software (Elst, The Netherlands, version 4.11.19). Each sample was evaluated in triplicates by each participant.

2.5 PTR-MS nose-space analysis

A proton transfer reaction mass spectrometry instrument equipped with a time of flight (PTR-ToF-MS) mass analyser was used (PTR-ToF 8000, Ionicon Analytik GmbH, Innsbruck, Austria). H_3O^+ was used as precursor ion and a PEEK inlet system was used for nose-space analysis from a heated (95 °C) nose-space air sampling extension (N.A.S.E., Ionicon Analytik,

GmbH, Innsbruck, Austria) apparatus. Ionisation conditions were 538 V drift voltage, 110 °C drift temperature and 2.8 mbar drift pressure, which resulted in an E/N ratio of 130 Td. Acquisition was set to one spectrum/sec and the inlet flow was 35 sccm.

The PTR-MS sampling protocol was adapted from a previous PTR-MS in nose study (van Eck et al., 2021). Briefly, after measuring 20 sec of laboratory air, panellists were asked to breathe for 60 sec into the machine by inserting two Teflon tubes in their nostrils, with their mouth closed to obtain a baseline breath. Next, panellists received the sample and chewed on it for 420 sec, at a speed of 60 bpm with the help of a metronome. After 420 sec, the chewing gum was discarded, and panellists' breath was analysed for another minute. PTR-MS data were acquired and calibrated with the TOFO Office (Department of Food Quality and Nutrition, Edmund Mach Foundation) following Cappellin *et al.* (2011).

2.6 UHPLC-HRMS analysis

The 0.6% TF aroma chewing gum samples were also used for Experiment 2. Each sample was introduced into a chewing apparatus (AB FIA, Lund, Sweden) (Kvist et al., 2000) together with 25 mL of artificial saliva. The artificial saliva was prepared accordingly to the DIN53160-1 and contained water, potassium and sodium salts, calcium and magnesium chloride and hydrochloric acid (pH of 6.8±0.1). The chewing device was set at 60 rpm (60 chew/min) and different saliva samples were obtained for each of the time intervals (15, 30, 60, 120, 180, 300, 420 and 540 sec), simulating the *in-vivo* nose-space experiment intervals. Saliva samples were extracted at regular intervals according to in-vivo protocol and differently diluted (1:100 to 1:1000) with acetonitrile before the UHPLC-HRMS analysis. The UHPLC-HRMS system was equipped with an Ultimate 3000 chromatograph coupled with a Q Exactive™ hybrid quadrupole-Orbitrap™ mass spectrometer (Thermo Fisher Scientific, Waltham, Massachusetts, USA). Samples (5 µL) were injected in a Luna Omega Sugar, 150 × 2.1 mm x 3 µm (Phenomenex, Bologna, Italy), thermostated at 5 °C. Water 10 mM

ammonium acetate (A) and acetonitrile (B) were used as eluents with the gradient:0–5 min 98% B, 5–20 min, 98%–93% B; 20–35 min, 93%-70% B; 35–36 min, 70%–98% B; 36–50 min, 98% B. The flow rate was set to 0.700 mL/min. MS data were acquired in electrospray (ESI) negative ionization mode (scan range: 90–500 m/z). Quantitative evaluations were made by means of a matrix-matched (artificial saliva) seven points external calibration curve in the range 0.1–10 mg/L. Peak areas for each analyte, were normalized for the area of the internal standard (mannitol-D9, Sigma Aldrich, Milan, Italy), added at the final concentration of 1 mg/L. Linearity was evaluated by the least squares regression and $R^2 > 0.99$ was obtained for all the analytes. LODs were 1 mg/L for aspartame and 5 mg/L for sorbitol, mannitol, xylitol and maltitol, respectively. Samples and calibration solutions were analysed in triplicate.

2.7 Data processing and analysis

2.7.1 Processing and treatment of sensory and PTR-MS data (Experiment 1)

Prior to downstream analysis, panel reliability and repeatability were verified through an ANOVA model, following standard procedures for panel performance evaluation (Table A1). PTR-MS data were pre-processed according to Pedrotti et al. (2020). To retain only relevant mass peaks, we excluded those associated with calibration, ^{13}C isotopes, and noise signals identified through visual inspection of concentration values (ppbv) over time. Peaks exhibiting baseline breathing patterns were classified as breath compounds and discarded. The remaining signals, defined as aroma compounds, were averaged over the time intervals corresponding to sensory evaluation. Subsequently, background noise and participants' breath were removed by subtracting the average concentration recorded during the baseline interval from each time point (15, 30, 60, 120, 180, 300, and 420 sec) (Charles et al., 2015).

Sensory and PTR-MS data were then averaged per replicate as a function of Sample and Time. To ensure adherence to normality assumptions, we ran the bestNormalize function from

the *BestNormalize R* package (Peterson 2021) and ordered quantile normalization was identified as the optimal transformation based on data fit. Normality and homogeneity of variance were assessed using the Shapiro–Wilk and Levene's tests, respectively. A two-way factorial analysis ANOVA was subsequently performed with Sample and Time as fixed factors. For the sensory data, when significant effects were detected, pairwise comparisons across aroma concentrations and time points were conducted using Tukey's HSD post hoc test. For the PTR-MS data, where no significant interaction was detected, one-way ANOVAs with Bonferroni correction were employed to assess differences among samples within each time interval and among time intervals within each sample. Data are reported as the mean ± SD, unless otherwise specified, and experimental m/z values are presented to three decimal places. All statistical tests were two-tailed, and significance was set at $p < 0.05$.

2.7.2 Data analysis of sweetener release (Experiment 2)

For Experiment 2, the release of each sweetener (mg/L) into saliva was obtained. The release in % was calculated by multiplying the concentration of each sweetener (in mg/L) by the amount of artificial saliva (15 g) to calculate the release in grams (Krause et al., 2011) and then dividing by the initial amount of sweetener. To better compare the sensory data with the instrumental data from the *in vitro* experiment, the release of each sweetener was calculated for each time interval as follows:

$$R_x = \frac{\Delta S_x}{\Delta t} \qquad \textit{Formula 1}$$

Where ΔS_x represents the difference between two concentrations of a specific sweetener at successive times divided by the time elapsed between the two measurements (Δt). These concentrations in time were then multiplied by their relative sweetness reported in Table 1 to obtain the sucrose equivalence which was plotted against time intervals.

2.7.3 Software

All data were analysed using R, version 3.6.2 (R foundation for statistical computing, Vienna, Austria, 2019), IBM SPSS Statistics for Windows, version 26 (IBM Corp., Armonk, N.Y., USA) and Microsoft Excel (Office 365). In R, panel performance was checked via the *SensoMineR* package. For visualisation of data, *ggplot2*, *ggpubr* and *FactoMineR* were used.

3. Results

3.1 Effect of aroma concentration on release and flavour perception

Dynamic aroma and sweetness perception of TF aroma of different concentrations is shown in Figure 1A and 1B, whereas *in vivo* nose-space aroma release for the five TF aroma concentrations tested is shown for four relevant mass peaks (Figure 2A, B, C and D).

When assessing aroma intensity (Figure 1A, Table 2), samples containing 0.4%, 0.8%, and 1.0% aroma concentration exhibited a higher perceived aroma after 30 sec compared to those with 0.2% and 0.6% aroma concentration. For the samples containing the highest levels of aroma (0.6, 0.8 and 1.0%), after 2 min of consumption, a decrease in aroma perception was found. The other samples, decreased already after the first 30 sec. The same was found for sweetness intensity, but with a much smaller deviation, suggesting that sweetness perception was more stable and less sensitive to changes in aroma concentration. The 2-way ANOVA found no significant interaction effect of aroma concentration and time ($F = 0.22$, $p = 0.93$). Both aroma concentration and time had a significant effect on aroma perception ($F = 6.06$ $p < 0.001$ and $F = 128.52$, $p << 0.001$, Table A2), indicating that the panel detected differences in aroma intensity among the samples and throughout their consumption. The post hoc test, revealed that during the consumption, the chewing gums with 0.4, 0.8, 1.0% of aroma were perceived as significantly higher ($p < 0.05$) than the sample with 0.2% of aroma. In terms of kinetics, significant differences ($p < 0.01$) among the samples were found between the first 3 min of consumption and the later stage of consumption (300, 420 and 480 sec). No significant differences in the first 2 min of consumption were observed (Table A2).

For sweetness intensity the 2-way ANOVA showed no significant interaction effects and aroma concentration did not affect sweetness perception (F=1.03. p = 0.39). As for aroma intensity, for the main effect of time, a significant difference was observed (F=117.60, $p < 0.001$) between the first part of the consumption (up to 180 sec) and the last part of consumption (420 and 480 sec) (Table 2, Table A2). In terms of performance, the panel demonstrated both reliability and repeatability, as supported by the lack of significant *sample:panellist* and *sample:replicate* interaction effects for all descriptors (Table A1 Supplementary Materials) and suggesting that each panellist evaluated the products consistently across attributes and replicates.

Table 2 here

Figure 1 here

Figures 2A, 2B, 2C and 2D show the release of *m/z* 89.060, *m/z* 117.091, *m/z* 131.107 and *m/z* 153.057, tentatively identified as ethyl acetate (plus a fragment of ethyl butyrate), a mixture of ethyl butyrate and isobutyl acetate, isoamyl acetate and vanillin, respectively. The release behaviour of *m/z* 89.060 (Figure 2A) had a similar trend to the one of *m/z* 117.091 and *m/z* 131.107 (Figure 2B and 2C), where a high release was observed initially, with a peaking around 60 sec and followed by a slow but steady decline. *M/z* 153.057 (Figure 2D) on the other hand showed a delay as its peak occurs at 120 sec. Statistical analysis (Table 3) confirmed these observations, showing significant differences ($p < 0.05$) in aroma release across all selected mass peaks and tested aroma concentrations during consumption. All the esters (*m/z* 89.060, 117.091 and 131.107) exhibited their maximum release within the 1 min of mastication, regardless of the initial aroma concentration in the chewing gums. Due to the high variability given by inter-individual differences, only few sample (aroma concentration 0.2%, 0.6% and 0.8%) showed a statistically significant difference in aroma release between the 60 sec interval and subsequent intervals with differences up to 63% towards the end of the consumption. Most

of the significant differences in aroma release kinetics for the esters were observed after chewing gums were discarded (*i.e.* after 7 min of consumption). For vanillin (*m/z* 153.057), the release was more constant during the consumption, with the highest concentration reached between one and 3 min of consumption across the different samples. Significant differences in vanillin concentration were observed only after the sample was discarded.

Table 3 here

For what concerns the different aroma concentrations in the chewing gums, an increase in nose-space aroma release was observed with increasing aroma concentrations levels (Figure 2, Table 3). For all the four mass peaks, a significant difference ($p<0.05$) between samples with different aroma concentrations was detected at all time points except for the last time interval (480 sec). At the end of consumption, a significant difference in aroma release was detected only for vanillin. The post-hoc tests revealed that TF4 and TF5 samples (0.8 and 1.0% of aroma) had a significant higher release for all mass peaks than TF1 and TF2 samples (0.2 and 0.4% of aroma) during the whole consumption with differences in release up to 15%. After the first 60 sec of consumption till 300 sec, a significant difference in aroma release was found also between samples containing 0.6% and 1.0% of the aroma for most of the mass peaks.

Figure 2 here

3.2 Sweetener release

The release for the five different sweeteners at various time intervals using an artificial chewing device is presented in Table 4. When considering absolute release (mg/L), sorbitol had the highest release at the end of the mastication (17177±1836 mg/L), followed by xylitol and mannitol, while aspartame showed the lowest release (26.61±1.9 mg/L). Sorbitol and maltitol displayed a relatively constant linear increase throughout mastication. In contrast, aspartame and mannitol exhibited a more pronounced increase (over 200%) at later stages of consumption (180–300 s), likely reflecting their lower solubility in saliva.

In terms of relative release (%), after 8 min of mastication, xylitol exhibited the highest release, followed by sorbitol and mannitol. In contrast, maltitol and aspartame showed a relatively low release of sweetener after 8 min – of 14.8 and 16.6% respectively. On average, total sweetener release reached approximately 33% of all the contained sweeteners after 8 min, which corresponded to the point at which the chewing was discarded in Experiment 1.

Table 4 here

To be able to better compare sweeteners and aroma release with the perceived sweetness and aroma of the TF3 sample (0.6% aroma concentration), the data are presented together in Figure 3. The concentration of only two compounds, *m/z* 131.107 and *m/z* 153.057 (identified as isoamyl acetate and vanillin) was plotted (Figure 3). These compounds were chosen since they represent the two release patterns that were observed in all the chewing gum aroma compounds, shedding some light on the possible taste-aroma interaction. A dichotomy is evident between the first (0-120 sec) and the second (after 120 sec) part of the consumption.

Figure 3 here

The perception of maximum aroma and sweetness was reached within the first 30 sec of consumption and remained stable until 120 sec before steeply declining (Figure 3A). A similar trend was observed for the *in-vivo* nose-space release of esters which reached the maximum release almost immediately (Figure 3B). However, in this case, the decline was more gradual than in sensory perception, particularly for vanillin. A different pattern was observed for sweeteners and relative sweetness (Figure 3C). While most compounds reached their maximum release early in consumption similarly to what was observed for aroma release and sensory perception, their presence in saliva remained stable into the saliva during the mastication. Notably, aspartame, the primary drive of relative sweetness due to its high sweetening power

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(Table 1), exhibited a slower increase, reaching peak release around the midpoint of consumption before gradually declining.

4. Discussion

For both sweetness and aroma perception, participants' evaluations are in line with previous studies where a maximum after few minutes was reached, followed by a decrease in intensity (Ovejero-López et al., 2004; Pedrotti et al., 2019; Potineni & Peterson, 2008b). Aroma release and perception are influenced not only by aroma concentration, but also by the physical and chemical properties of the aroma compounds and sweeteners. From a sensory perspective, the observed differences at low aroma concentration (0.2%) compared to higher concentrations, support the general relationship between higher aroma concentration-higher aroma release and perception (Prazeller et al., 2006). Notably, the differences emerged more clearly after the first 60 sec of mastication, suggesting that aroma concentration plays a more critical role in aroma persistence than in the initial aroma burst. As well, this indicates that 0.2% difference in aroma concentration may be below the aroma difference threshold perceivable by the panel. In contrast, no significant differences in sweetness perception were observed among the samples. Since all samples shared the same sweetener composition, any differences would likely stem from cross-modal interactions with aroma. Previous studies have shown that congruent food-like aromas such as vanilla, caramel, or fruit can enhance sweetness perception through multi-modal interactions (Wang et al., 2019, Bertelsen et al., 2020). In the present case, however, the absence of detectable differences may suggest that in SFG the enhancement of sweetness is more linked to the presence or absence of a congruent aroma rather than to small concentration variations. It is also possible that within the concentration range tested, the increments in aroma were below the perceptual threshold required to elicit additional sweetness enhancement. These findings therefore support the idea that multi-modal interactions and specifically, aroma–taste

interactions, are highly product- and context-specific, and that their magnitude may depend also on perceptual thresholds.

In vivo aroma release is more complicated than just the sum of the single aroma compound characteristics. For instance, adsorption and resorption of volatile compounds by the mouth mucus membrane has been shown to hamper the prediction of aroma compounds released in the nasal mucosa (Buettner & Schieberle, 2000; Raithore & Peterson, 2016). In addition, both the composition of the oral microbiota and interaction with salivary proteins contribute to inter-individual variability. Salivary proteins may bind or facilitate the release of volatile aroma compounds via chemical reactions, thereby modulating their partitioning behaviour (Ployon et al., 2017). Furthermore, the presence of odorant-metabolizing enzymes in the olfactory epithelium, which participate in odorant signal termination and modulation, may also influence PTR-MS nose-space response (Neiers et al., 2023).

Despite these complexities, our study found a moderate positive correlation (*p.value* < 0.05, $R \approx 0.6$) between added aroma concentration (ranging from 0.2% to 1%) and in-nose concentration during consumption (Figure A1 & A2 - Supplementary Materials). This suggests that, within this range, higher concentrations resulted in proportionally higher *in vivo* aroma release across all tested VOCs. Similar relationships have been previously documented. For example, Krause (2010) reported a 1.7-fold increase in-nose volatile concentration measured via PTR-MS, when the aroma concentration in chewing gum was doubled using an artificial chewing device. Likewise, a linear correlation between menthone concentration and perceived intensity has been demonstrated in both aqueous solutions (Tietz et al., 2008) and during *in vivo* consumption of beverages (Hodgson et al., 2005).

Regarding release kinetics, two distinct patterns were observed (Figure 2): esters displayed an early burst release with a sharp peak in concentration, whereas vanillin exhibited a slower, more sustained release throughout mastication. These differences can be attributed to the compounds'

physicochemical properties. Esters are typically low in molecular weight, hydrophobic, and highly volatile, with weak interactions with the gum matrix. Their rapid release is facilitated by high diffusion coefficients and minimal binding, while their fast decline is likely due to depletion from the gum surface and the water phase (Hinderink et al., 2019). Our findings on ethyl acetate align with those of Sostmann et al. (2009), who reported peak in-nose intensity within 3 min using non-coated chewing gum. In contrast, vanillin has a higher molecular weight, lower volatility, and polar characteristics due to its hydroxyl and aldehyde functional groups, which can form strong interactions with the gum base and the saliva. Its lower diffusion rate and possible hydrogen bonding with gum or salivary components contribute to its slower release, which is further influenced by the progressive liberation of oil and flavour droplets during mastication (Hinderink et al., 2019). Moreover, its delayed peak in PTR-MS measurements may be partially due to its 'sticky' nature - its tendency to condense within instrumental tubing - potentially exacerbated by its relatively high boiling point (285 °C, Table 1 and 3) compared to other VOCs (Hartungen et al., 2013). This divergent behaviour between compounds illustrates how aroma release kinetics during mastication are shaped not only by chemical structure and volatility but also by molecular interactions.

In this study, an artificial chewing device was used to help understand sweetener release during mastication by excluding inter-individual variability. The primary mechanism for sweeteners release is dissolution, which involves the transfer of sweeteners from their solid state within the gum matrix into the liquid salivary medium. As expected, sorbitol and xylitol, due to their high solubility in water, were rapidly released upon contact with saliva, resulting in both the highest absolute and relative release during the whole mastication (Raithore & Peterson, 2018). These highly water-soluble bulk sweeteners are responsible for the rapid "burst" release during the initial chewing phase and may contribute to the observed initial sweetness perception. Despite its higher aqueous solubility, maltitol exhibited lower release levels compared to mannitol. This

behaviour may be attributed to its greater molecular weight and the presence of multiple hydroxyl functional groups, which could facilitate stronger intermolecular interactions (such as hydrogen bonding) with components of the gum base. These interactions are likely to impede its diffusion into the saliva phase during mastication. Nevertheless, our results show that sweeteners release plays an important role in perception especially in the first 2 min of mastication, (Figure 3). While *in vivo* aroma release and aroma perception followed a similar pattern, the most striking incongruity found by this study was the decline in sweet perception over time in comparison to the *in vitro* sweeteners release. Even by considering the values obtained by Formula 1, while sweet perception started to decrease after the first 2 min of consumption following aroma perception, the maximum intensity in relative sweeteners was reached after about 3 min of mastication due to the slower aspartame release (Raithore & Peterson, 2018). The release of this high intensity sweetener did not stop the decrease in sweet perception, which may be partially attributed to adaptation phenomena involving physiological and cognitive pathways. Sensory adaptation occurs as the human brain tries to protect itself from an 'information overload' by attenuating stimuli from the senses (O'Mahony, 1986). In addition, the decreasing aroma perception may have influenced sweet perception by cross-modal interactions. However, it should be noted that the sweeteners released measured in this study was obtained *in vitro* by using a chewing simulator, which – while highly controlled – cannot fully replicate the complexity of the physicochemical oral environment and oral processing. The used chewing simulators lack key physiological processes such as dynamic salivary flow, swallowing, mucosal absorption, enzymatic activity, and microbial metabolism, all of which may significantly influence real in vivo release dynamics and sensory perception (Doyennette et al., 2014). Therefore, the *in vitro* results should be interpreted with caution. Future studies should include *in vivo* measurements of sweetener release, to validate our findings and to obtain a more continuous and physiologically representative understanding of

release behaviour over time. ~~Due to the high inter-individual variability, it is also recommended to have a larger number of panellists to gain a better representation of the aroma release and perception..~~ Another limitation of this study that should be acknowledged is the relatively small number of panelists which may limit the generalizability of the findings, especially given the high inter-individual variability typically observed in *in-vivo* aroma release and perception experiments. Future studies should therefore involve a larger and more diverse panel to improve statistical power and representativeness. Finally, a more detailed characterization of participants (*e.g.*, saliva composition, flow rate, breathing patterns) would help better explain individual differences in aroma release and perception. Integrating both product-related and subject-related factors will allow a more comprehensive understanding of in-vivo flavour dynamics.

5. Conclusion

This study elucidates how aroma concentration and sweetener release jointly govern *in vivo* flavour perception in SGF. While increasing aroma levels (0.2–1.0%) produced proportionally higher nose-space aroma concentrations, perceived aroma intensity did not increase beyond 0.4% aroma. Furthermore, increasing the aroma concentration within the same chewing gum samples had no significant effect on perceived sweetness. Two temporal phases emerged: an early phase (0–120 s) where aroma and sweetness perceptions closely track sweetener release, and a later phase characterized by sensory decline despite ongoing aroma and sweetener liberation. These results highlight the importance of considering both compound physicochemical properties and multicomponent release dynamics when designing SFG formulations for targeted sensory profiles. From an applied perspective, manufacturers may benefit from optimizing aroma levels to enhance the initial flavour burst, while tailoring sweetener composition and release behaviour to sustain sweetness perception beyond the first two minutes of chewing.

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Methodologically, coupling PTR-MS nose-space analysis with dual DTI sensory evaluation and *in vitro* UHPLC-HRMS quantification of sweeteners offers a framework for dissecting complex flavour interactions when tailoring SFG aroma concentration. Future work should extend *in vitro* sweetener measurements to *in vivo* contexts, incorporate larger and more diverse panels, and explore the role of salivary cues and mucosal interactions on aroma and sweetener bioavailability. Such advances will refine predictive models of flavour perception and guide the development of optimized confectionery products.

CRediT authorship contribution statement

M.P.: Conceptualization, data curation, formal analysis, investigation, methodology, project administration, software, visualization, writing-original draft, writing – review & editing. **S.v.d.L.:** Formal analysis, data curation, writing - original draft, writing - review & editing, visualization. **L.M.**: Investigation, data curation, writing – review & editing, **L.C.**: Resources, software, methodology, supervision, writing – review & editing. **G.R.**: Resources, conceptualization, investigation, formal analysis. **M.R.**: Investigation, methodology, writing – review & editing. **S.B.**: Resources, methodology, supervision, writing – review & editing. **S.P.:** Investigation, methodology, writing – review & editing. **V.F.:** Conceptualization, resources, writing – review & editing, supervision, project administration. **F.B:** Conceptualization, resources, writing – review & editing, supervision, funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AI Statement

During the preparation of this work the author(s) used ChatGPT (v5.1) in order to improve readability of the introduction and discussion sections. After using this tool/service, the

author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the published article.

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Tables:

Table 1: Physicochemical properties of the aroma compounds, of the bulk sweeteners and high intensity sweetener (HIS) used in the chewing gum samples. Boiling points were obtained from PubChem, Log P were obtained from ECHA website and odour threshold values from the American Industrial Hygiene Association.

Aroma compounds					
Molecule	**Chemical Formula**	**Mw (g/mol)**	**Bp (°C)[a]**	**Log P[b]**	**Odour threshold value (ppm)[c,d]**
Ethyl acetate	C4H8O2	88.11	77	0.73 c	9.0 E-2 – 1.9 E2

Ethyl butyrate	C6H12O2	116.16	122	3.4 (25 °C)	1.0 E-3
Isobutyl acetate	C6H12O2	116.16	117	2.3 (25 °C)	8.0 E-3 – 1.3 E2
Isoamyl acetate	C7H14O2	130.18	142	2.7 (35 °C)	7.5 E-4 – 3.7 E2
Vanillin	C8H8O3	152.15	285	1.2 (20 °C)	1.6 E-7 – 9.3 E-2
			Sweeteners		
Molecule	**Chemical Formula**	**Mw (g/mol)**	**Relative sweetness (sucrose =1)**	**Solubility (g/ 100 g H_2O at 25°C)[e]**	**Sweeteners type**
Sorbitol	C6H14O6	182.17	0.6	235	Sugar alcohol
Xylitol	C5H12O5	152.15	0.95	200	Sugar alcohol
Mannitol	C6H14O6	182.17	0.5	22	Sugar alcohol
Maltitol	C12H24O11	344.31	0.8-0.9	165	Sugar alcohol
Aspartame	C14H18N2O5	294.30	246-247	≈1[a]	HIS

a PubChem | b ECHA (2019) | c American Industrial Hygiene Association (1989) | d Leffingwell and Leffingwell (2004) | e (Marie & Piggott, 1991)

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Table 2: Average aroma and sweetness (± SD) for each chewing gum sample and time interval (Experiment 1). Since no sign. interaction effect was found by the two-way ANOVA, p.values for the main effects (aroma concentration of the chewing gum samples and time intervals) are reported in a separate table (Table A2).

Aroma								
Concentration [%] / Time [sec]	**15**	**30**	**60**	**120**	**180**	**300**	**420**	**480**
0.2	39.71±25.68	43.95±25.38	39.29±21.32	36.91±21.05	32.15±16.16	22.08±16.93	16±11.95	8.66±6.82
0.4	47.91±21.8	54.13±22.48	52.48±20.85	48.44±20.21	45.56±18.61	32.11±18.86	22.52±18.72	13.68±13.01
0.6	41.86±20.31	43.46±19.91	39.71±19.36	41.94±18.87	34.51±19.82	27.39±17.99	18±14.13	8.94±6.22
0.8	44.16±21.24	50.51±19.77	52.74±19.6	54.55±17.73	46.33±18.13	33.56±20.72	28.52±20.07	17.84±17.58
1	43.01±20.92	50.34±18.48	52.02±14.89	53.61±17.17	44.4±14.41	35.05±16.86	26.18±14.63	12.6±10.58
Sweetness								
Concentration [%] / Time [sec]	**15**	**30**	**60**	**120**	**180**	**300**	**420**	**480**
0.2	47.42±25.26	52.79±22.77	51.36±21.37	50.06±19.7	45.54±20.37	37.22±22.44	29.48±20.09	18.31±15.33
0.4	48.31±23.15	55.82±21.38	55.59±17.64	52.73±17.05	46.51±16.54	34.65±18.06	24.65±17.31	13.28±12.91
0.6	40.12±20.32	45.9±20.66	42.03±21.34	44.23±21.42	40.32±20.83	33.11±20.56	26.07±18.17	13.39±12.88
0.8	41.33±20.78	47.47±20.18	49.86±19	48.36±18.35	43.56±18.7	31.68±18.44	25.82±18.6	12.65±10.95
1	42.38±18.93	50.75±17.14	51.17±16.52	53.77±16.39	43.68±16.7	36.08±16.92	27.14±15.74	12.42±12.73

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Table 3: Average aroma release (± SD) for each chewing gum sample and time interval (Experiment 1). One-way ANOVA results for concentration and time effects are shown. Different superscripts (a–d) within rows indicate significant differences over time for each sample; different subscripts (w–z) within columns indicate differences among samples at each time point (Bonferroni-corrected, $p < 0.007$). Data for each mass peak are presented in separate sections.

m/z 89.06								
Concentration [%] / Time [sec]	**30**	**60**	**120**	**180**	**300**	**420**	**480**	**p-value Time**
0.2	$14.5\pm10.2^{b}_{w}$	$24.6\pm6.2^{c}_{w}$	$17.3\pm7.3^{b}_{w}$	$15.8\pm5.8^{b}_{w}$	$13.2\pm4.2^{b}_{w}$	$12.1\pm4.0^{b}_{w}$	1.7 ± 1.7^{a}	1.70E-12
0.4	$37.6\pm27.9^{b}_{wx}$	$51.6\pm30.3^{b}_{w}$	$47.1\pm22.6^{b}_{wx}$	$46.3\pm27.6^{b}_{w}$	$35.9\pm27.4^{b}_{wx}$	$31.9\pm19.4^{b}_{wx}$	8.7 ± 7.7^{a}	2.20E-05
0.6	$56.3\pm45.1^{bc}_{wy}$	$78.2\pm42.3^{c}_{wx}$	$68.5\pm38.9^{bc}_{xy}$	$54.3\pm 28.3^{bc}_{w}$	$46.9\pm30.1^{b}_{x}$	$49.1\pm31.4^{bc}_{xy}$	6.3 ± 11.7^{a}	3.10E-07
0.8	$81.6\pm59.2^{bc}_{xy}$	$124.7\pm79.8^{c}_{xy}$	$97.5\pm58.2^{bc}_{yz}$	$95.0\pm51.2^{bc}_{x}$	$78.8\pm37.7^{bc}_{y}$	$66.2\pm35.1^{ab}_{y}$	22.5 ± 44.1^{a}	2.30E-06
1	$106.7\pm132.6^{b}_{y}$	$144.6\pm125.8^{b}_{y}$	$139.6\pm88.1^{b}_{z}$	$112.2\pm78.2^{b}_{x}$	$86.8\pm45.9^{ab}_{y}$	$72.4\pm33.1^{ab}_{y}$	12.2 ± 11.7^{a}	1.10E-04
p.value Concentration	1.48E-03	5.71E-06	4.17E-09	3.37E-08	1.07E-09	4.87E-09	8.57E-02	
ms.117.091								
Concentration [%] / Time [sec]	**30**	**60**	**120**	**180**	**300**	**420**	**480**	**p-value Time**
0.2	$19.8\pm14.8^{b}_{w}$	$31.6\pm10.9^{c}_{w}$	$20.8\pm8.5^{b}_{w}$	$19.0\pm6.7^{b}_{w}$	$16.1\pm5.3^{b}_{w}$	$15.2\pm5.2^{b}_{w}$	1.8 ± 1.9^{a}	3.72E-12
0.4	$43.4\pm32.6^{b}_{wx}$	$57.9\pm35.5^{b}_{w}$	$51.3\pm26.1^{b}_{wx}$	$50.7\pm32.4^{b}_{w}$	$40.6\pm33.5^{b}_{wx}$	$36.3\pm23.9^{b}_{wx}$	8.6 ± 20.7^{a}	6.85E-05
0.6	$66.4\pm48.1^{bc}_{wy}$	$90.0\pm48.3^{c}_{wx}$	$76.1\pm41.8^{bc}_{xy}$	$58.2\pm29.8^{bc}_{wx}$	$52.4\pm33.6^{b}_{x}$	$56.8\pm37.2^{bc}_{xy}$	7.1 ± 16.7^{a}	1.26E-07
0.8	$99.8\pm76.2^{bc}_{xy}$	$145.7\pm100.6^{c}_{x}$	$107.4\pm65.6^{bc}_{yz}$	$103.0\pm58.9^{bc}_{xy}$	$88.1\pm45.4^{b}_{y}$	$75.6\pm43.5^{ab}_{y}$	25.2 ± 54.2^{a}	1.06E-05
1	$133.4\pm179.7^{b}_{y}$	$166.5\pm155.9^{b}_{x}$	$156.8\pm103.7^{b}_{z}$	$127.8\pm102.0^{b}_{y}$	$96.4\pm53.9^{ab}_{y}$	$83.1\pm41.4^{ab}_{y}$	11.3 ± 11.1^{a}	0.0005
p.value Concentration	3.14E-03	3.02E-05	1.22E-08	5.88E-07	1.36E-08	6.92E-08	1.36E-01	
m/z 131.107								
Concentration [%] / Time [sec]	**30**	**60**	**120**	**180**	**300**	**420**	**480**	**p-value Time**
0.2	$1.2\pm0.9^{b}_{w}$	$1.9\pm1.0^{c}_{w}$	$1.4\pm0.5^{b}_{w}$	$1.3\pm0.5^{b}_{w}$	$1.2\pm0.4^{b}_{w}$	$1.2\pm0.4^{b}_{w}$	0.2 ± 0.2^{a}	1.30E-09

0.4	$2.3\pm1.8^{b}_{wx}$	$3.4\pm2.2^{b}_{w}$	$3.0\pm1.5^{b}_{wx}$	$3.2\pm2.0^{b}_{w}$	$2.6\pm2.0^{b}_{wx}$	$2.5\pm1.5^{b}_{wx}$	0.6 ± 1.2^{a}	7.90E-05
0.6	$3.8\pm2.8^{b}_{wy}$	$5.5\pm2.9^{b}_{wx}$	$4.8\pm2.6^{b}_{xy}$	$3.9\pm1.9^{b}_{wx}$	$3.6\pm2.7^{b}_{x}$	$4.2\pm2.7^{b}_{xy}$	0.6 ± 1.2^{a}	1.20E-07
0.8	$5.6\pm4.2^{ab}_{xy}$	$8.8\pm6.0^{b}_{x}$	$6.8\pm4.0^{b}_{yz}$	$6.7\pm3.7^{b}_{xy}$	$6.1\pm3.0^{b}_{y}$	$5.5\pm3.0^{ab}_{y}$	2.1 ± 4.4^{a}	5.20E-06
1	$7.5\pm9.0^{b}_{y}$	$10.6\pm9.5^{b}_{x}$	$9.7\pm5.8^{b}_{z}$	$8.4\pm5.8^{b}_{y}$	$6.9\pm3.7^{ab}_{y}$	$6.3\pm3.1^{ab}_{y}$	1.2 ± 1.2^{a}	3.10E-04
p.value Concentration	9.71E-04	1.20E-05	1.36E-09	4.37E-08	1.19E-09	1.03E-08	8.95E-02	
m/z 153.057								
Concentration [%] / Time [sec]	**30**	**60**	**120**	**180**	**300**	**420**	**480**	**p-value Time**
0.2	$0.1\pm0.1^{a}_{w}$	$0.3\pm0.1^{b}_{w}$	$0.3\pm0.1^{b}_{w}$	$0.3\pm0.1^{b}_{w}$	$0.3\pm0.1^{b}_{w}$	$0.3\pm0.1^{b}_{w}$	$0.1\pm0^{a}_{w}$	1.70E-13
0.4	$0.3\pm0.2^{a}_{wx}$	$0.6\pm0.3^{b}_{w}$	$0.6\pm0.3^{b}_{w}$	$0.7\pm0.3^{b}_{wx}$	$0.6\pm0.3^{b}_{wx}$	$0.6\pm0.3^{b}_{wx}$	$0.2\pm0.2^{a}_{w}$	2.70E-05
0.6	$0.4\pm0.2^{ab}_{xy}$	$0.9\pm0.5^{c}_{wx}$	$0.9\pm0.6^{c}_{wx}$	$0.9\pm0.5^{c}_{xy}$	$0.7\pm0.4^{bc}_{x}$	$0.8\pm0.4^{bc}_{xy}$	$0.2\pm0.1^{a}_{w}$	3.20E-08
0.8	$0.6\pm 0.3^{ab}_{y}$	$1.3\pm0.6^{c}_{xy}$	$1.2\pm 0.7^{c}_{y}$	$1.4\pm 0.7^{c}_{yz}$	$1.2\pm0.6^{c}_{y}$	$1.0\pm 0.5^{bc}_{yz}$	$0.5\pm0.4^{a}_{x}$	2.90E-06
1	$0.7\pm0.6^{ab}_{y}$	$1.7\pm1.2^{c}_{y}$	$1.9\pm1.2^{c}_{y}$	$1.8\pm1.0^{c}_{z}$	$1.4\pm0.8^{bc}_{y}$	$1.4\pm0.6^{bc}_{z}$	$0.6\pm0.4^{a}_{x}$	5.04E-06
p.value Concentration	1.53E-05	4.12E-08	6.83E-09	1.06E-09	3.06E-10	1.81E-11	2.55E-07	

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Table 4: Mean sweetener release (in mg/L and as percentage of the total concentration), along with standard deviations, for the five sweeteners measured during *in vitro* mastication (Experiment 2). Data represent the average of three replicates at specific measurement time points.

		Time [seconds]						
Sweetener	**Variable**	**15**	**30**	**60**	**120**	**180**	**300**	**420**
Sorbitol	Release [mg/L]	2166±38	2562±433	3694±100	5955±131	8306±54	12404±217	17177±1836
	Release [%]	5.6±0.1	6.7±1.1	9.6±0.3	15.5±0.3	21.6±0.1	32.3±0.6	44.7±4.8
Mannitol	Release [mg/L]	112±1	147±20	281±25	444±11	526±25	1132±92	1375±133
	Release [%]	2.8±0	3.7±0.5	7.0±0.6	11.1±0.3	13.2±0.6	28.3±2.3	34.4±3.3
Xylitol	Release [mg/L]	700±253	655±243	683±123	1092±142	1364±232	2014±278	2336±250
	Release [%]	17.5±6.3	16.4±6.1	17.1±3.1	27.3±3.6	34.1±5.8	50.4±7.0	58.4±6.3
Maltitol	Release [mg/L]	173±53	223±71	313±82	500±90	693±222	902±283	1187±272
	Release [%]	2.2±0.7	2.8±0.9	3.9±1.0	6.3±1.1	8.7±2.8	11.3±3.5	14.8±3.4
Aspartame	Release [mg/L]	2.5±0.2	2.72±0.2	2.97±0.4	4.67±0.7	7.02±1	23.11±1.3	26.61±1.9
	Release [%]	1.6±0.2	1.7±0.2	1.9±0.2	2.9±0.4	4.4±0.6	14.4±0.8	16.6±1.2

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Figure 1: Average temporal profiles (± SE) for (A) aroma intensity and (B) sweetness intensity across the chewing gum samples. Each curve represents the mean ratings obtained from the sensory panel, calculated from three replicated evaluations per participant. Variations among aroma concentrations are depicted using different shades of blue.

Figure 2: Average nose-space aroma release (± SE) over time for relevant mass peaks: (A) *m/z* 89.060 ($C_4H_8O_2H^+$) ethyl acetate, (B) *m/z* 117.091 ($C_6H_{13}O_2^+$), ethyl butyrate/isobutyl acetate (C) *m/z* 131.107 (C7H14O2H$^+$) isoamyl acetate, (D) *m/z* 153.057 ($C_8H_8O_3H^+$) vanillin. Each curve represents the mean ratings obtained from the sensory panel, calculated from three replicated evaluations per participant. Variations between the samples with different aroma concentrations are shown based on different shades of blue.

Figure 3: Sample TF3 (0.6% aroma concentration): A) Mean ± SE of the panel (n = 6, three replicates) for the perceived sweetness and aroma intensity during consumption; B) Mean ± SD of the panel (n = 6, three replicates) for the nose space aroma release during consumption of *m/z* 131.107 (C7H14O2H$^+$) – identified as isoamyl acetate and *m/z* 153.057 ($C_8H_8O_3H^+$) – identified as vanillin; C) Release of all the sweeteners (sorbitol, xylitol, mannitol, maltitol, aspartame) and of their sum normalized per time interval and per relative sweetness (Formula 1). The measurements were performed in triplicates.

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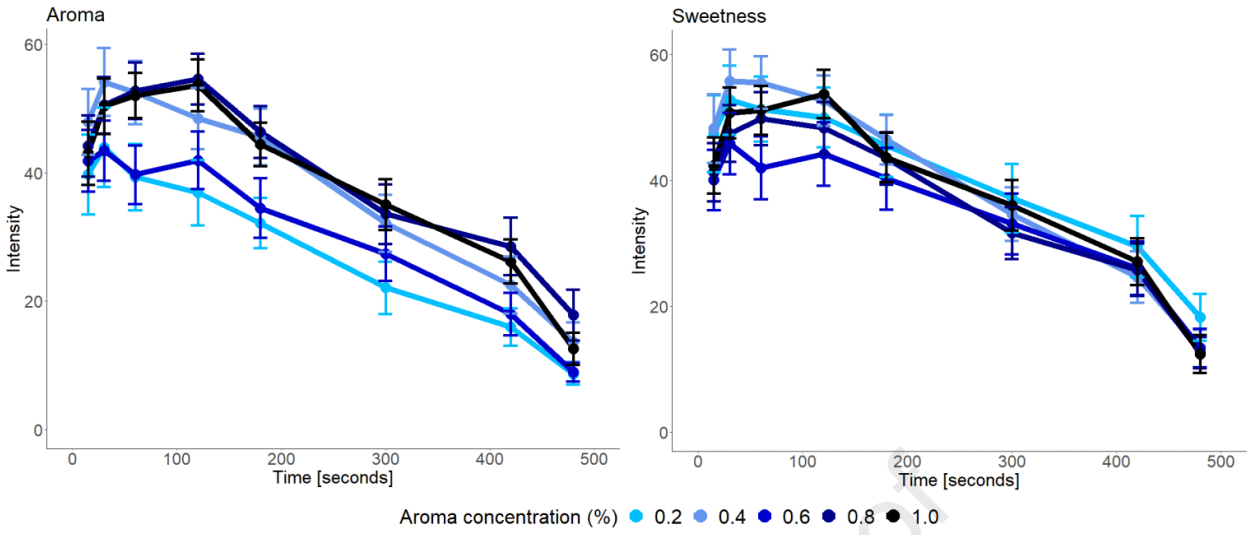

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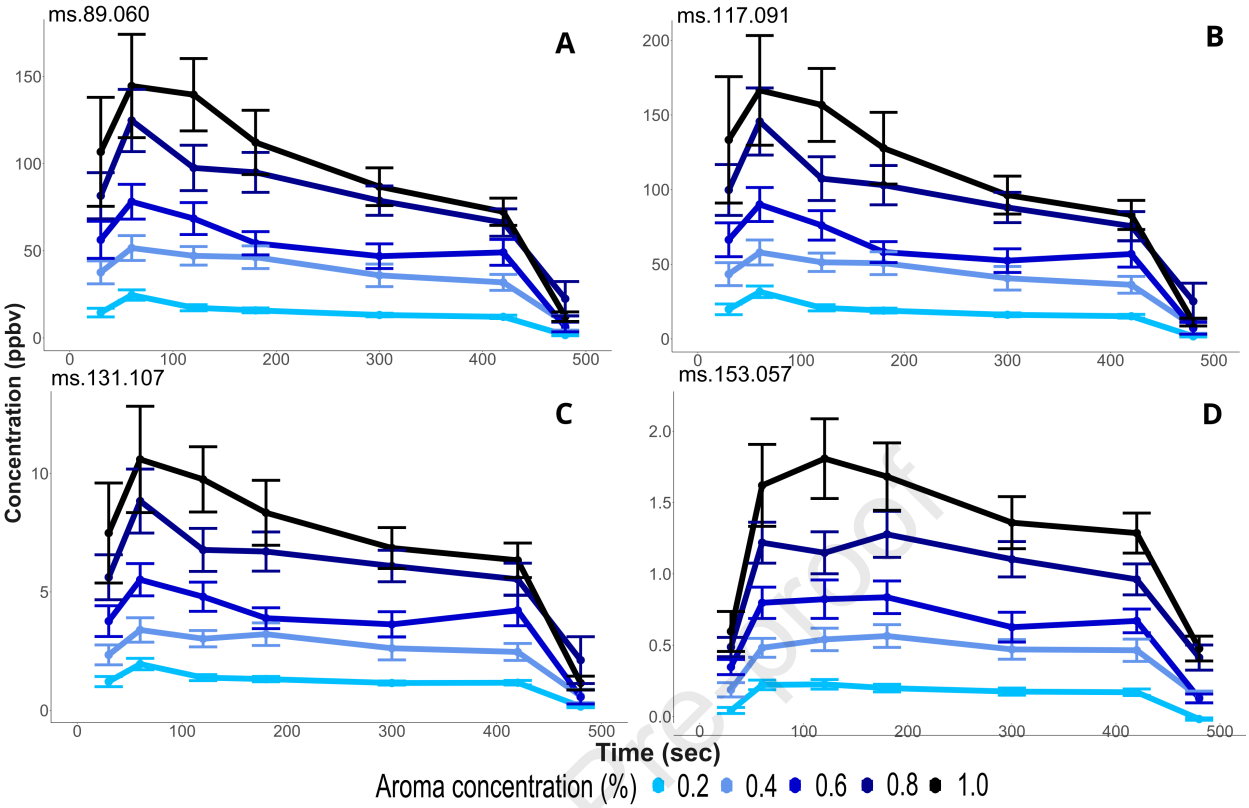

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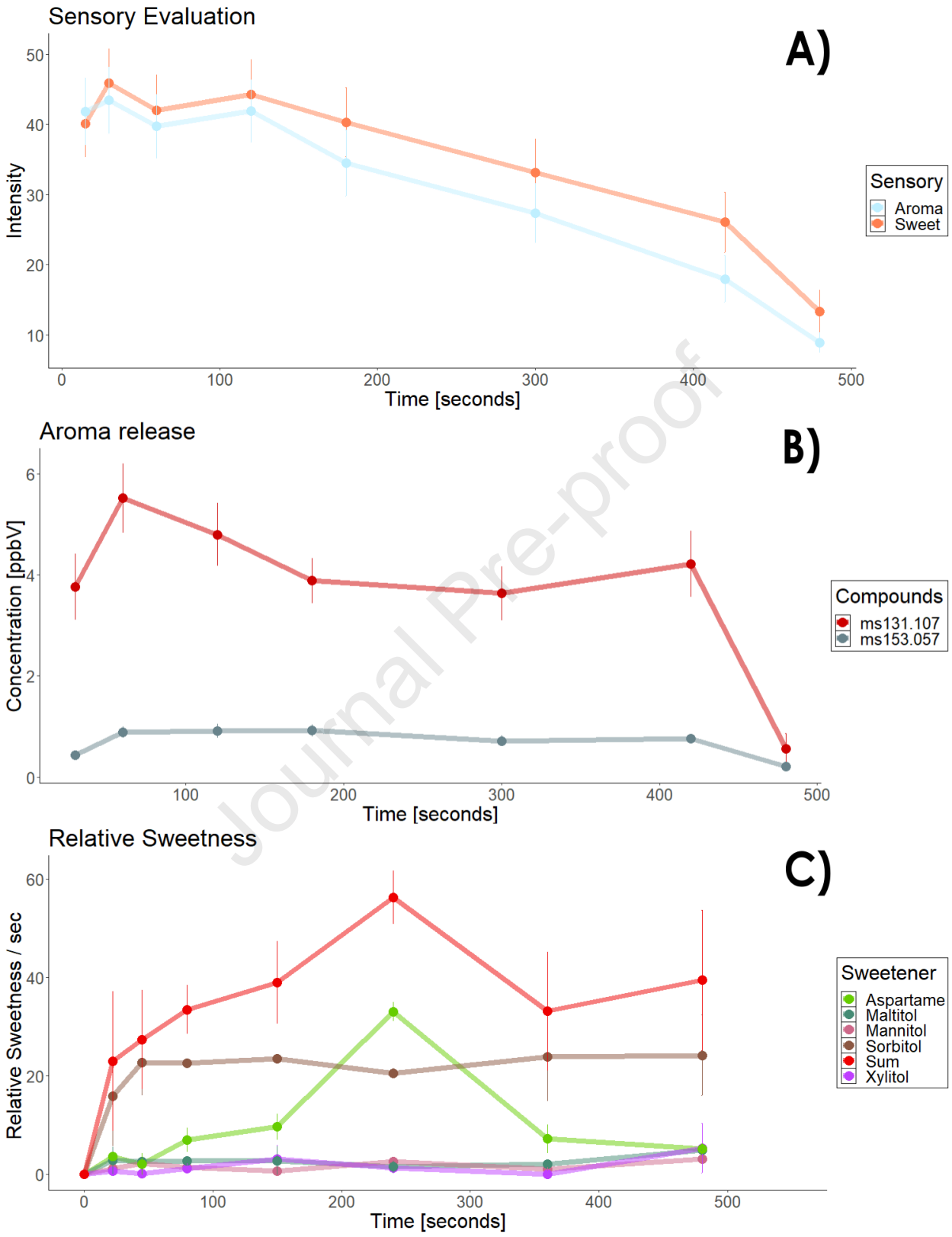

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Highlights

- Aroma concentration increased in-nose aroma release but not perceived sweetness.
- Aroma perception rose with concentration but was shaped by perceptual thresholds.
- Esters showed burst release, while vanillin displayed sustained release.
- Sweetener release followed biphasic kinetics with compound-specific patterns.
- Combined nose-space PTR-MS, sensory dTI, and UHPLC-MS revealed flavour dynamics.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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