ACTA SCIENTIARUM POLONORUM
Acta Sci. Pol. Technol. Aliment. 21(4) 2022, 359–368
www.food.actapol.net pISSN 1644-0730 eISSN 1898-9594 http://dx.doi.org/10.17306/J.AFS.2022.1056

ORIGINAL PAPER

Received: 15.05.2022
Accepted: 3.10.2022

INVESTIGATION OF FLAVOUR PERMANENCE IN LEMON-FLAVOURED CHEWING GUM BY RETRONASAL AROMA-TRAPPING AND SENSORY ANALYSIS TECHNIQUES

Pinar Uzun[1,2], Ahmet Emir Ozunal[1], Hakan Baskaya[1], Nuray Dogan[1], Sonay Merve Gulay[1], Busra Hantal[1], Melike Uner[3], Asli Barla Demirkoz[1,4]✉

[1]Department of Research and Development Center, Aromsa Flavours and Food Additives 41480 Kocaeli, **Turkey**
[2]Department of Food Technology Isparta, University of Applied Sciences, Gelendost VS 32200 Isparta, **Turkey**
[3]Department of Pharmaceutical Technology, Faculty of Pharmacy, Istanbul University, Beyazıt 34116 Istanbul, **Turkey**
[4]Department of Nutrition and Dietetics, Faculty of Health Sciences, Haliç University Güzeltepe Mahallesi 15, Temmuz Şehitler Caddesi 15, 34060 Eyüp, Istanbul, **Turkey**

ABSTRACT

The use of *in vivo* instrumental analysis and sensory evaluation techniques in the investigation of volatile compounds released from gum matrices is a convenient method for scientists to obtain more decisive results on the properties of chewing gums. In this study, four types of lemon-flavoured chewing gum were prepared, and their flavour permanence was monitored by *in vitro* and *in vivo* techniques. For this purpose, both sensory and analytical techniques were conducted using a retronasal aroma-trapping device and DHA-GC/MS. *Cis*-citral and *trans*-citral were chosen as the flavour compounds to be monitored. An accelerated shelf-life test was also performed. Although the presence of *cis*-citral and *trans*-citral in the breath was most intense 5 minutes after chewing the gum, it could still be detected even 45 minutes later. The lemon flavouring was also perceived by the assessors in the 45th minute. The lemon taste was found to persist in the formulations for 12 months.

Keywords: chewing gum, flavour permanence, flavourings, lemon flavouring, retronasal aroma-trapping device, sensory analysis

INTRODUCTION

Chewing gum is one of the most consumed confectionery products all over the world (Fenimore, 2008). The gum industry has also been growing in Turkey and new products which meet consumer expectations are constantly being put on the market. According to the Mintel market size report, 11,000 tons of chewing gum were put on the market for consumption in Turkey in 2021.

Chewing gum is also used for medicinal purposes (Ozcelik and Kunduk, 2021; Rassing, 1994). The first commercial medical gum, "Aspergum", with a formula containing acetylsalicylic acid, was put on the market in 1924 (Woodford and Lesco, 1981). A new line of sugar-free chewing gum for dental care was introduced by Mondelez and P&G-owned Oral B in

✉aslibarla@gmail.com, https://orcid.org/0000-0002-3261-0212, phone +902623261111, fax +902627511390

2020 in Europe. Fluoride is added to the product to strengthen teeth against acids, preserve tooth mineralization, and aid in the neutralization of dental plaque. Chewing gums are also used to manage nicotine addiction. They can provide an advantage in masking the bad taste of the drug. Chewing gums containing some drugs, such as nystatin (Andersen et al., 1990), methadone (Christrup et al., 1990a), verapamil (Christrup et al., 1990b), caffeine (Guest et al., 2021), ondansetron (Nagaich et al., 2010), and miconazole (Bastian et al., 2004), have been designed for their oral and topical application. Dental caries can be treated and oral health can be maintained with specifically formulated chewing gums which stimulate the flow of saliva, thus providing more rapid oral clearance, neutralizing plaque pH, and increasing remineralization of early lesions (Dodds, 2012).

Xylitol is a polyol widely used in both medical and non-medical chewing gums. It has a significant antiplaque effect on tooth surfaces and decreases gingival inflammation. As a result, it is commonly used in chewing gums as a preventive agent for dental caries as it reduces levels of pathogenic microorganisms (Gasmi Benahmed et al., 2020). Xylitol was reported to prevent acute otitis media in children up to 12 years of age (Azarpazhooh et al., 2016).

Whether it is used as confectionery or for therapeutic purposes, the flavour of chewing gum must be palatable and permanent. Due to the increasing demand for this indispensable confectionery, manufacturers try to diversify their products by considering consumer satisfaction all over the world. Flavourings that give taste and odour to foods are one of the most important factors determining the quality and market value of chewing gums (De Roos, 2008). 0.4–1 g/100 g flavour components are generally used in chewing gum formulations for this purpose (Wong et al., 2009). However, after 3–5 minutes of chewing, most gums usually lose their flavour and consumer perception fades (Fenimore, 2008). Flavour permanence (in other words, flavour release during chewing) is closely related to factors including chewing power, saliva flow rate, and the properties of chewing gums, such as rheological properties and the ingredients used in the production of the gum base (Boland et al., 2007; Haahr et al., 2003; Hodgson et al., 2003; Koliandris et al., 2008; Wong et al., 2009). Gum base is produced with or without sugar. Various additives such as texture modifiers and preservatives are used in combination in the production of gum base, and these ingredients specify the desirable characteristics of chewing gums. Distribution of the flavour components between the hydrophobic and hydrophilic phases of gums depends on their hydrophobicity and hence their affinity to these phases (Hinderink et al., 2019; Potineni and Peterson, 2008; Taylor et al., 2000). In addition, increasing the sweetness and sourness of the gum base affects the intensity of the perceived flavour (Bonnans and Noble, 1993; Lotfabadi et al., 2020; McBride and Johnson, 1987; Pfeiffer et al., 2006). Existing encapsulation techniques are used to protect the flavour compounds being processed and to protect the final product during storage (Castro and Johnson, 2006). Perception of the flavourings of the chewing gum during chewing begins and continues in the oronasal tract. Since the flavour permanence of the chewing gum determines consumer preference, reliable analytical techniques have been developed to monitor the release of the flavour volatiles from production to storage (Guichard, 2002). These techniques involve *in vivo* and *in vitro* instrumental analysis. Methods used to determine the perception of food products in the past were based on static perception in a given moment. However, current dynamic techniques have higher reliability and allow for better detection of consumer perceptions (Dijksterhuis and Piggott, 2000; Lotfabadi et al., 2020).

Sophisticated instrumental methods and specific instruments have been developed to analyse flavour compounds in chewing gums including headspace solid-phase microextraction (HS-SPME), atmospheric pressure-chemical-ionization mass spectrometry (APCI-MS, MS-noise), and proton transfer reaction mass spectrometry (PTR-MS; Boland et al., 2006; Buettner and Schieberle, 2000; Buettner et al., 2008; Dijksterhuis and Piggott, 2000; Kataoka et al., 2000; Taylor et al., 2000). The retronasal aroma-trapping device (RATD--GC/MS; retronasal olfactometer-GC/MS) has been designed to detect flavour release as part of the retronasal olfaction test. To use this device, assessors breathe into a nozzle placed in a glass mask and the air sample (breath) passing through the mask is trapped in the Tenax TA. Then, the trapped molecules are analysed through GC/MS to determine the flavour components

in the breath (Bonneau et al., 2018; Muñoz-González et al., 2014). Sensory evaluation techniques are used to determine consumers' reactions to the appearance, taste, texture, and other sensory characteristics of foods. Among sensory analysis tests, the affective (preference) test is mostly used to determine consumer preferences. In the literature, no *in vivo* studies involving simultaneous sensory and instrumental analysis of lemon-flavoured chewing gums were found, but it is very important to conduct *in vivo* instrumental analysis with sensory evaluation techniques to monitor the release of volatile components from the gum matrix. Flavourings include a lot of complex flavour compounds, which can react with each other and with other ingredients in foods. In the food optimization process, it is difficult to define the permanence of perception and to determine the release of flavours using instruments. The RATD-GC/MS can provide a reliable analysis by using flavour precursors for each flavouring component.

Flavour permanence studies on different food matrices using an RATD-GC/MS can support the creation of more permanent flavourings by assisting with process optimization in confectionery production. In this study, the permanence of lemon-flavoured gums during chewing was analysed *in vivo* by RATD-GC/MS combined with sensory evaluation techniques. An affective test and descriptive sensory analysis (DSA) were performed to assess the correspondence between analytical and sensory evaluation techniques.

MATERIALS AND METHODS

Material

The gum base was purchased from Remik Chemistry Trade Inc. Co., Pendik, Istanbul. The gum base contained *cis*-citral (CAS 106-26-3), *trans*-citral (CAS 141-27-5), and citric acid (CAS 77-92-9; Jungbunzlauer Suisse AG); malic acid (CAS 6915-15-7; Tate & Lyle, Turkey); encapsulated citric acid (50%; Tastetech, Bristol, UK); sorbitol, maltitol syrup, mannitol and xylitol (Roquette Agriculture and Food LLC, Turkey); cooling agent (WS3 Type; Henan Sunlake Enterprise Corporation, Henan, China); isomalt (Beneo, Turkey); sucralose (Splenda, Turkey); encapsulated sucralose (10%; Tastetech, Bristol, UK). Lemon flavour, encapsulated lemon flavour, and extruded lemon flavour were obtained internally (Aromsa Flavours and the Food Additives Inc. Co., Kocaeli, Turkey). All other chemicals were of analytical grade.

Sample preparation

Lemon-flavoured chewing gums were prepared in accordance with Council Regulations EU No: 1330/2008 (The European Parliament and of the Council of 16 December 2008 on Food Additives; Table 1). An artificial flavouring mixture of the same concentration was used in all chewing gum formulations (equal amounts of its liquid, encapsulated, and extruded forms). Since stick gums are the most preferred chewing gum type in the world, 2.2 ±0.2 g of stick gum were prepared in this study (Fritz, 2008). Immediately after preparation, they were put in a plastic bag and wrapped in aluminium foil. They were stored at 21°C and 35% relative humidity until being used for analysis.

Table 1. Composition of lemon-flavoured chewing gums, %

Ingredients	Formulations			
	A	B	C	D
Flavour mixture*	1.5	1.5	1.5	1.5
Gum Base (Talk Type)	30	30	30	30
Encapsulated citric acid	1.2	1.2	1.2	1.2
Citric acid	0.5	0.5	0.5	0.5
Malic acid	0.5	0.5	0.5	0.5
Sorbitol	47.3	47.3	47.75	47.75
Maltitol syrup	7.5	7.5	7.5	7.5
Mannitol	3	3	3	3
Encapsulated sucralose	1	1	0.5	0.5
Sucralose	–	–	0.05	0.05
Xylitol	7.5	–	7.5	–
Isomalt	–	7.5	–	7.5
Cooling agent	0.1	0.1	0.1	0.1

*Flavour mixture includes the same flavourings (liquid, encapsulated and extruded ingredients).

Table 2. Lemon flavouring attributes selected for DSA

Attribute	Characteristics
Lemon	typical acidity of citrus
Sour	lemon sourness
Astringent	lemon juice and the bitter taste in its peel
Sweet	a small amount of sweetness felt in lemon
Peely	the typical taste of lemon peel
Juicy	the typical taste of lemon juice
Sweet	the typical sweetness of strawberries

Flavouring attributes were selected by tasting the flavoured chewing gums.

Study design

The study was designed in two stages. The first was the preselection stage, including a sensory evaluation test (affective test), and the second was the instrumental analysis stage, including a sensory evaluation (descriptive sensory analysis − DSA) only for selected samples. The effectiveness of formulations was evaluated and accordingly, the optimum taste for chewing gums was selected by trained and experienced assessors.

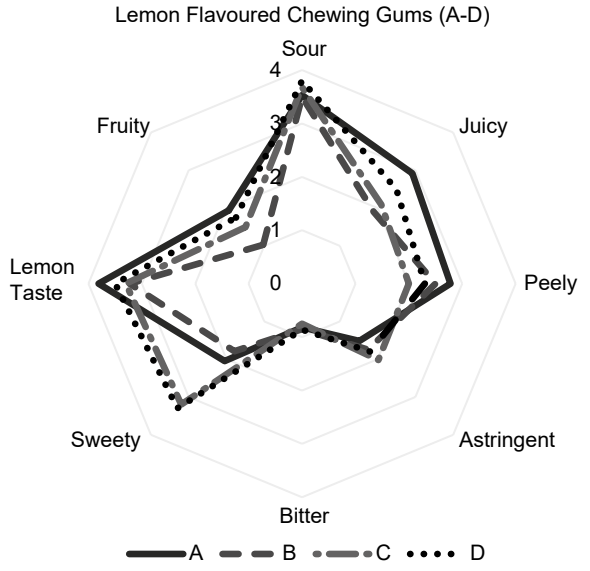

Fig. 1. Sensory analysis radars of lemon-flavoured chewing gums. Flavouring attributes of the assessors on formulations A, B, C and D

Formulation A included xylitol and encapsulated suclarose. Formulation B included isomalt and encapsulated suclarose. Formulation C included xylitol, encapsulated suclarose, and free suclarose. Formulation D included isomalt, encapsulated suclarose, and free suclarose. Hence, the use of free and encapsulated forms of sucralose in the presence of xylitol or isomalt was assessed by comparing A with C and B with D. Then, the most preferred formulation was determined by comparing the favourite among A and C with the favourite among B and D. Thereafter, the flavour profile of all formulations was characterized by DSA (Table 2 and Fig. 1). For the second stage of the study, according to the results of the sensory analysis, the flavour permanence of each selected chewing gum was monitored both by sensory evaluation analysis and using an *in vivo* aroma trapping device.

Assessors. All assessors were selected and trained according to guidelines from the International Organization for Standardization (ISO 8586:2014). The formulations were evaluated by the affective test, conducted with an experienced and trained assessor group (16 male and 24 female healthy assessors aged 25–50, 7 of whom participated in the *in vivo* RATD-GC/MS experiments) to determine the intensities of the tasting and flavouring attributes. The assessors, volunteers from AROMSA Inc. Co., were non-smoking, non-pregnant, had no piercings in the mouth, had no mastication or swallowing disorders, and had no known illnesses. They also had normal olfactory and gustatory function. All assessors provided informed consent for this study.

Sensory evaluation analyses

Affective (preference/acceptance) test. This test was performed in the personal cabins of the AROMSA Inc. Co. Sensory Analysis Laboratory. Positive pressure was applied to remove various odours from the cabins. The cabin temperature and relative humidity were kept at 21 ±1°C and 50–55% during the analysis. There were no objects that could have disturbed the concentration of the assessors in the cabins. A five-point linear scale was used to evaluate the intensity of the sample taste. Instructions about the evaluation and the scale were also provided on evaluation monitors. The samples were presented to the assessors with no distinguishing features and coded with randomly selected 3-digit numbers (Lawless and Heymann, 1998).

In the affective test, assessors were first asked which gum formulation they preferred. In addition, they were asked to rate the chewing gums according to the hedonic scale of 0–5 with regard to Figure 1. The flavoured chewing gums were evaluated in separate sessions by each assessor. The assessors participated in two evaluation sessions, which occurred over the whole day, one in the morning and the other in the afternoon. Each assessor evaluated two samples in each session. A and C were evaluated in one session; B and D in the other session. Finally, the preferred samples were re-evaluated in each session. In the third session, the most preferred sample was chosen by the assessors. In every session, water and crackers were provided to clean the assessor's mouth (Hort and Hollowood, 2004).

Descriptive sensory analysis (DSA) was performed for the flavouring attributes of each selected lemon-flavoured chewing gum (Table 2). The formulations were evaluated by an experienced assessor group to determine the intensities of the tasting and flavouring attributes. Before DSA, flavoured chewing gums were given to the assessors, who were informed about the method, analysis steps, and product information (ISO/DIS 13299: 2016). Critical information like production and expiration dates was not given to the assessors. Evaluations were also performed in the personal cabins of the Aromsa Sensory Analysis Laboratory. A five-point linear scale was used to evaluate the sample taste intensities. Instructions for the evaluation and the scale were also provided on the evaluation monitors. The samples were presented to the assessors with no distinguishing features and coded with randomly selected 3-digit numbers.

Flavour lexicons are used in a sensory evaluation to determine the flavour profiles of food products. In the DSA method, we used a consumer panel composed of a small, highly trained group of people known as a descriptive panel. Descriptive panels use lexicons, a list of defined terms, to describe products. The assessors discussed and determined the characteristics of each attribute of the samples and reference products (Lawless and Heymann, 1998).

In vivo aroma trapping device (RATD-GC/MS)

A RATD-GC/MS was used for *in vivo* experiments as reported in our earlier study (Uzun et al., 2022). The method reported by several groups (Bonneau et al., 2018; Buettner and Schieberle, 2000; Buettner et al., 2008; Muñoz-González et al., 2014) was modified for this purpose. The device has 3 parts: an olfactory glass port, the Tenax TA part, and a vacuum pump. Assessors put their noses into the glass port and exhaled through the nasal cavity during the mastication of flavoured chewing gum. The olfactory port was combined with an adsorbent polymer conditioned in stainless steel tube (200 mg Tenax TA, 60–80 mesh, Gerstel, Germany). The Tenax TA tube was connected to a vacuum pump at the outlet of the device. The vacuum pump provided a stable 500 mL/min flow during *in vivo* aroma trapping. Here, a gas flowmeter (ADM 2000 Flowmeter, Agilent Technologies, Wilmington, USA) was used to measure the stable gas flow. The flavoured compounds from the assessors' breath were collected in the Tenax TA, and then the thermal desorption unit, which is a unit of DHA-GC/MS, desorbed these compounds from the Tenax TA. The desorbed compounds were injected automatically into the same column used for calibration.

Protocol. The protocol of this study was prepared based on earlier studies performed at Aromsa Research and Development Center. The assessors were not permitted to eat or drink anything except water in the 2 hours before the *in vivo* experiments. Lemon-flavoured chewing gum formulations were studied in triplicate over six sessions held on separate days, at the same time of day. The first gum sample was put into the mouth and chewed for 20 s by exhalation through the nose. At the end of 20 s, 3 breaths were blown into the olfactory glass port at approximately 5 s intervals and data T0 were collected. The same process was repeated in the 5th and 45th minutes of chewing (3 blows at 5 s intervals) and the permanence of the flavour components, which were collected in the Tenax TA, was measured with DHA-GC/MS (Uzun et al., 2022).

Volatile compounds analysis. *Cis*-citral and *trans*-citral, as flavouring precursors, were dissolved in ethanol for preparation of the calibration curves (0–2 mg/mL, 7 points). Solutions were put into vials and collected in the Tenax TA, and the thermal desorption unit then desorbed these compounds from the Tenax TA. The desorbed compounds were injected automatically

into a DP-WAX UI GC column (60 m × 25 μm film thickness × 0.25 mm innerdia; INNOWAX, Germany). A calibration curve was obtained for each compound.

The volatile compounds of the flavoured chewing gum were analysed by DHA-GC/MS. Their total ion chromatogram was obtained using a Gerstel DHS System (Germany) connected to an Agilent 7890A GC and 5975C MS equipped with an Inert MSD with triple axis detector (Germany).

The flow rate of the helium carrier gas was adjusted to 1.2 mL/min. Each sample in the Tenax TA was analysed in splitless mode. The GC oven temperature was programmed to increase from 40°C to 240°C at 5°C/min.

Data treatment and statistics. RATD-GC/MS data were evaluated by one-way analysis of variance (ANOVA), and significant differences found among the assessors were further evaluated by Tukey's test in the SPSS package program (Version 23.0, IBM Inc., New York, USA). The results were considered statistically significant at $p < 0.05$. All sensorial analyses were carried out using Fizz Software (the non-parametric Friedman test; Biosystems, Couternon, France) at a 95% confidence level.

Accelerated shelf-life analyses

The shelf-life of chewing gums is generally reported to be monitored for at least 12 months in the market. According to previous research at Aromsa Research and Development Center, storage at 35 ±1°C and 30–40% relative humidity for 3 months is accepted to be equal to 12 months. For this purpose, the samples were divided into experimental and control groups. Experimental samples were used for both sensory and instrumental analysis. The lemon-flavoured chewing gums were chewed by 7 assessors for 45 min. The experimental samples were stored under 35 ±1°C and 30–40% relative humidity in an incubator (Nüve ES252, Istanbul, Turkey) as the control group of chewing gums was freshly produced for each analysis. The samples were analysed with RATD-GC/MS.

RESULTS AND DISCUSSION

The first stage of the study

In the first session of the affective test, formulation A was preferred by 66.67% of the assessors, and in the second session, formulation D was preferred by 60% of the assessors. In the third and final session, groups D and A were compared, and formulation A was found to be preferred by 82.35% of the assessors. The assessors evaluated formulation A as a juicier and softer chewing gum with a balanced lemon peel taste which is more permanent and with more intensive lemon taste nuances than other formulations (Fig. 1).

In the first session of the affective test, formula A was found to be preferred by 82.35% of the assessors. Xylitol, which is used in formulation A, is known as a raw material with refreshing and positive dental effects. It makes the flavour more intense in the presence of acid and sucralose (Fritz, 2008). The use of encapsulated sucralose in formulation A influences taste assessments due to natural lemon sourness rather than sweetness. Because of lemon's sourness, taste was more important than sweetness for the assessors. Acidity regulators should be used in acidic fruit flavours due to their flavour-supporting properties in lemon-flavoured chewing gums. Especially in lemon flavourings, the acidity in gum is determined by higher citric acid concentrations, increased maximum sourness intensity, and the time of the end of a sour taste in the lemon flavour (Veldhuizen et al., 2018). Therefore, the formulations with xylitol are preferred in lemon-flavoured chewing gums due to its cooling effect and better enhancement of fruit flavourings, especially in acidic environments.

The second stage of the study

The evaluation results on the permanency of flavourings and fruity taste nuances over 45 minutes of chewing are presented in Figure 2.

The most observable compounds for lemon-flavoured chewing gum were chosen to monitor the permanency with *in vivo* RATD-GC/MS after specifically formulated lemon flavouring was analysed in GC. For this purpose, *cis*-citral and *trans*-citral were monitored and both were still observed in the 45th minute (Table 3). The sensory and DHA-GC/MS analyses were confirmed to be compatible. According to the sensory evaluation results, the lemon was still perceived as a lemon flavouring by the assessors after 45 minutes. It should be considered that working with devices with precisely selected compounds gives parallel results in sensory evaluation studies.

Table 3. Data obtained from RATD-GC/MS experiment on lemon-flavoured chewing gums

Assessors	T0		5 min		45 min	
	cis-citral	*trans*-citral	*cis*-citral	*trans*-citral	*cis*-citral	*trans*-citral
1	86.34 ±0.11[a]	100.78 ±0.12[a]	78.43 ±0.13[a]	91.65 ±0.12[a]	15.57 ±0.09[a]	20.28 ±0.07[a]
2	41.51 ±0.10[b]	44.92 ±0.12[b]	39.14 ±0.10[b]	42.19 ±0.09[b]	20.28 ±0.11[b]	20.95 ±0.09[b]
3	57.45 ±0.12[c]	62.62 ±0.11[c]	55.01 ±0.07[c]	59.12 ±0.11[c]	36.04 ±0.0012[c]	31.29 ±0.10[c]
4	63.08 ±0.11[d]	59.08 ±0.08[d]	59.28 ±0.11[d]	55.67 ±0.11[d]	30.05 ±0.10[d]	29.15 ±0.09[d]
5	77.22 ±0.09[e]	79.89 ±0.14[e]	71.25 ±0.09[e]	74.16 ±0.13[e]	23.48 ±0.08[e]	29.75 ±0.10[e]
6	47.07 ±0.13[f]	53.25 ±0.11[f]	44.81 ±0.11[f]	50.09 ±0.09[f]	27.26 ±0.09[f]	25.79 ±0.11[f]
7	32.45 ±0.10[g]	36.51 ±0.13[g]	30.79 ±0.10[g]	33.95 ±0.13[g]	18.01 ±0.09[g]	13.61 ±0.08[g]

Results are mean ±SD (10^{-2} mg/mL). Different letters in the same column indicate significant differences at $p < 0.05$.
While preparing the curve, weights were made on the flavour and the quantity analysis was started.
$R = 0.99716$, $y = 1.19 \times 107X + 3.39 \times 104$ for *cis*-citral and $R = 0.99733$, $y = 1.20 \times 107X + 7.29 \times 104$ for *trans*-citral.

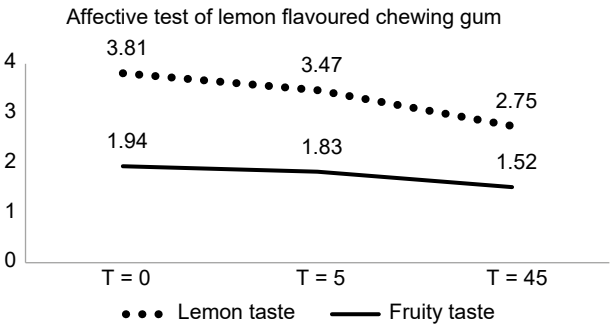

Fig. 2. Sensory analysis of lemon-flavoured chewing gum

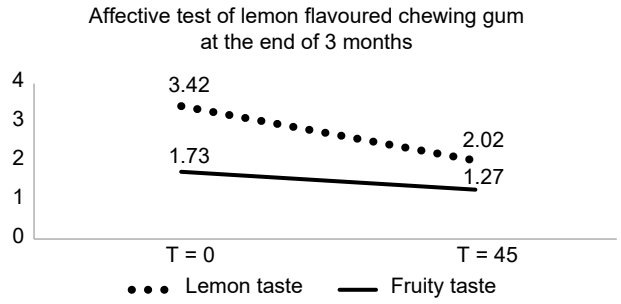

Fig. 3. End of shelf-life sensory analysis results for lemon-flavoured chewing gum

Because they are the most observable compounds for lemon-flavoured chewing gum, *cis*-citral and *trans*-citral were monitored with *in vivo* RATD-GC/MS. The sensory and DHA-GC/MS analyses were confirmed to be compatible. Headspace solid-phase microextraction (HS-SPME; Parliment, 1998; Pawliszyn, 1997), positive chemical ionization tandem mass spectrometry (PCI-MS), microstructure noise (MS-noise; Boland et al., 2006; Taylor et al., 2000), and proton-transfer-reaction mass spectrometry (PTR-MS; Buettner et al., 2008; Kataoka et al., 2000) are some of the instrumental methods used to analyse flavour compounds in chewing gums. Although other systems have been developed, they are expensive and hard to use. RATD-GC/MS is easier to use and cheaper than the others. Only a vacuum pump, special glass apparatus, and Tenax TA are required in this apparatus. It can be easily adapted to GC/MS. RATD-GC/MS could be an easy solution to monitor flavour quality parameters in the production of gum-type products as it gives similar and close results to human perception.

Accelerated shelf-life analysis

The permanency of flavourings and fruity taste nuances were evaluated during the product's shelf-life (Tables 3 and 4, and Figures 2 and 3). It was confirmed that the results obtained from the sensory analysis were consistent with the data from the RATD-GC/MS.

Table 4. Shelf-life of lemon-flavoured chewing gums

Assessors	T0		45 min	
	cis-citral*	*cis*-citral*	*trans*-citral*	*trans*-citral*
1	10.84 ±0.10[a]	2.95 ±0.07[b]	14.62 ±0.78[a]	33.83 ±0.11[e]
2	2.74 ±0.08[b]	0.88 ±0.01[a]	3.33 ±0.09[b]	1.08 ±0.09[a]
3	87.76 ±0.11[c]	5.63 ±0.08[c]	11.435 ±0.11[c]	8.72 ±0.88[cd]
4	23.75 ±0.09[d]	7.53 ±0.09[d]	25.86 ±0.10[d]	9.88 ±0.87[d]
5	84.33 ±0.76[e]	0.02 ±0.001[a]	11.79 ±0.10[e]	0.05 ±0.001[a]
6	12.46 ±0.95[f]	5.50 ±0.92[c]	19.82 ±0.10[f]	7.78 ±0.08[c]
7	8.52 ±0.08[g]	4.88 ±0.10[c]	13.20 ±0.78[g]	5.04 ±0.79[b]

Results are mean ±SD (10^{-2} mg/mL).
Different letters in the same column indicate significant differences at $p < 0.05$.

CONCLUSION

The flavour of foods is typically estimated exclusively by using sensory perception analysis to evaluate flavour release and permanence. However, because flavour and taste perception is a human sensory function, there may be circumstances in which flavour release cannot be properly predicted and the perception of taste and flavour might not be apparent due to various characteristics of food products. Due to the intricacy of mixing effects and the possibility of perceptual interaction, changes in objective measurements of flavour release should always be compared to human perception.

The data gained in this study are useful for the formulation and manufacture of confectionary products that meet consumer preferences. The perception of flavourings and the permanence of the flavours cannot be determined only by sensory techniques. Taste perception may not be clear due to properties of food products such as sugar or acidity in the food matrix. In this study, the taste persistence of chewing gums was examined using both sensory methods and the RATD--GC/MS methodology in the presence of specifically chosen chemicals from the flavourings of chewing gum and the results were found to be compatible. It was noted that the measurements and the assessors' ratings agreed on certain key points.

It was concluded that the use of RATD-GC/MS along with in vivo sensory analysis to create more permanent flavours in food could be used to carry out more meticulous and detailed research. In addition, this methodology would provide great support to manufacturers in optimizing the production process. It could also be used to determine how to mask the undesirable flavours that drugs may leave in the mouth by adding active ingredients to chewing gum formulations. Since chewing gums with added xylitol are expected to be effective in protecting the health of teeth, gingiva, and oral mucosa, it is important to develop effective methodologies to assess its impact on taste perception.

ACKNOWLEDGMENTS

This work was supported by the TUBITAK (The Scientific and Technological Research Council of Turkey) [Grant Number TEYDEB 3180337]. The authors would also like to thank Aromsa Flavours and Food Additives Inc. Co., Kocaeli, Turkey and are grateful to Murat YASA and Melis YASA for their support in this study.

REFERENCES

Andersen, T., Gram-Hansen, M., Pedersen, M., Rassing, M. R. (1990). Chewing gum as a drug delivery system for nystatin, influence of solubilizing agents upon the release of water insoluble drugs. Drug Dev. Ind. Pharm., 16, 13, 1985–94. https://doi.org/10.3109/03639049009023636

Azarpazhooh, A., Lawrence, H. P., Shah, P. S. (2016). Xylitol for preventing acute otitis media in children up to 12 years of age. Cochr. Syst. Rev., 8, CD007095. https://doi.org/10.1002/14651858.CD007095.pub3

Bastian, H. L., Rindum, J., Lindeberg, H. (2004). A double-dummy, double-blind, placebo-controlled phase III study comparing the efficacy and efficiency of miconazole chewing gum with a known drug (Brentan gel) and a placebo in patients with oral candidosis. Oral Surg. Oral Med. Oral Path. Oral Rad. Endodont., 98, 4, 423–428. https://doi.org/10.1016/j.tripleo.2003.12.028

Boland, A. B., Delahunty, C. M., van Ruth, S. M. (2006). Influence of the texture of gelatin gels and pectin gels on strawberry flavour release and perception. Food Chem., 96, 3, 452–460. https://doi.org/10.1016/j.foodchem.2005.02.027

Bonnans, S., Noble, A. C. (1993). Effect of sweetener type and of sweetener and acid levels on temporal perception of sweetness, sourness and fruitiness. Chem. Sens., 18, 3, 273–283. https://doi.org/10.1093/chemse/18.3.273

Bonneau, A., Boulanger, R., Lebrun, M., Maraval, I., Valette, J., Guichard, É. (2018). Impact of fruit texture on the release and perception of aroma compounds during in vivo consumption using fresh and processed mango fruits. Food Chem., 239, 806–815. https://doi.org/10.1016/j.foodchem.2017.07.017

Buettner, A., Otto, S., Beer, A., Mestres, M., Schieberle, P., Hummel, T. (2008). Dynamics of retronasal aroma perception during consumption: Cross-linking on-line breath analysis with medico-analytical tools to elucidate a complex process. Food Chem., 108, 4, 1234–46. https://doi.org/10.1016/j.foodchem.2007.03.042

Buettner, A., Schieberle, P. (2000). Exhaled odorant measurement (EXOM) − a new approach to quantify the degree of in-mouth release of food aroma compounds. LWT – Food Sci. Technol., 33, 8, 553–559. https://doi.org/10.1006/fstl.2000.0708

Castro, A. J., Johnson, S. S. (2006). Encapsulated flavors and chewing gum using same. United States patent US 7,022,352. Apr 4.

Christrup, L. L., Angelo, H. R., Bonde, J., Kristensen, F., Rasmussen, S. N. (1990a). Relative bioavailability of methadone hydrochloride administered in chewing gum and tablets. Acta Pharm. Nord., 2, 2, 83–88.

Christrup, L. L., Bonde, J., Rasmussen, S. N., Rassing, M. R. (1990b). Relative bioavailability of (±)-verapamil hydrochloride administered in tablets and chewing gum. Acta Pharm. Nord., 2, 6, 371–6.

De Roos, K. B. (2008). Flavourings for chewing gum. In D. Fritz (Ed.), Formulation and production of chewing and bubble gum (pp. 205–237). London, UK: Loughton, Kennedys Publ.

Dijksterhuis, G. B., Piggott, J. R. (2000). Dynamic methods of sensory analysis. Trends Food Sci. Technol., 11, 8, 284–290. https://doi.org/10.1016/S0924-2244(01)00020-6

Dodds, M. W. J. (2012). The oral health benefits of chewing gum. J. Irish Dental Assoc., 58(5), 253–261.

Fenimore, E. L. (2008). Flavourings for chewing gum. In D. Fritz (Ed.), Formulation and production of chewing and bubble gum (pp. 1–46). London, UK: Loughton, Kennedys Publ.

Fritz, D. (Ed., 2008). Formulation and production of chewing and bubble gum. London, UK: Loughton, Kennedys Publ.

Gasmi Benahmed, A., Gasmi, A., Arshad, M., Shanaida, M., Lysiuk, R., Peana, M., ..., Bjørklund, G. (2020). Health benefits of xylitol. Appl. Microbiol. Biotechnol., 104, 7225–37. https://doi.org/10.1007/s00253-020-10708-7

Guest, N. S., VanDusseldorp, T. A., Nelson, M. T., Grgic, J., Schoenfeld, B. J., Jenkins, N. D. M., ..., Campbell, B. I. (2021). International society of sports nutrition position stand: caffeine and exercise performance. J. Int. Soc. Sports Nutr., 18, 1, 1–37. https://doi.org/10.1186/s12970-020-00383-4

Guichard, E. (2002). Interactions between flavor compounds and food ingredients and their influence on flavor perception. Food Rev. Int., 18, 1, 49–70. https://doi.org/10.1081/FRI-120003417

Haahr, A. M., Madsen, H., Smedsgaard, J., Bredie, W. L., Stahnke, L. H., Refsgaard, H. H. (2003). Flavor release measurement by atmospheric pressure chemical ionization ion trap mass spectrometry, construction of interface and mathematical modeling of release profiles. Anal. Chem., 75, 3, 655–662. https://doi.org/10.1021/ac026035u

Hinderink, E. B., Avison, S., Boom, R., Bodnár, I. (2019). Dynamic flavor release from chewing gum: Mechanisms of release. Food Res. Int., 116, 717–723. https://doi.org/10.1016/j.foodres.2018.09.002

Hodgson, M., Linforth, R. S. T., Taylor, A. J. (2003). Simultaneous real-time measurements of mastication, swallowing, nasal airflow, and aroma release. J. Agric. Food Chem., 51, 17, 5052–57. https://doi.org/10.1021/jf030118+

Hort, J., Hollowood, T. A. (2004). Controlled continuous flow delivery system for investigating taste − aroma interactions. J. Agric. Food Chem., 52, 15, 4834–43. https://doi.org/10.1021/jf049681y

Kataoka, H., Lord, H. L., Pawliszyn, J. (2000). Applications of solid-phase microextraction in food analysis. J.

Chromat. A, 880, 1–2, 35–62. https://doi.org/10.1016/S0021-9673(00)00309-5

Koliandris, A., Lee, A., Ferry, A. L, Hill, S., Mitchell, J. (2008). Relationship between structure of hydrocolloid gels and solutions and flavour release. Food Hydrocoll., 22, 4, 623–630. https://doi.org/10.1016/j.foodhyd.2007.02.009

Lawless, H. T., Heymann, H. (1998). Sensory evaluation of food: Principles and practices. New York, USA: Aspen Publ.

Lotfabadi, S. V., Mortazavi, S. A., Yeganehzad, S. (2020). Study on the release and sensory perception of encapsulated d-limonene flavor in crystal rock candy using the time-intensity analysis and HS-GC/MS spectrometry. Food Sci. Nutr., 8, 2, 933–41. https://doi.org/10.1002/fsn3.1372

McBride, R. L., Johnson, R. L. (1987). Perception of sugar--acid mixtures in lemon juice drink. Int. J. Food Sci. Technol., 22, 4, 399–408. https://doi.org/10.1111/j.1365-2621.1987.tb00503.x

Muñoz-González, C., Rodríguez-Bencomo, J. J., Moreno--Arribas, M. V., Pozo-Bayón, M. Á. (2014). Feasibility and application of a retronasal aroma-trapping device to study in vivo aroma release during the consumption of model wine-derived beverages. Food Sci. Nutr., 2, 4, 361–370. https://doi.org/10.1002/fsn3.111

Nagaich, U., Chaudhary, V., Karki, R., Yadav, A., Sharma, P. (2010). Formulation of medicated chewing gum of ondansetron hydrochloride and its pharmacokinetic evaluation. Int. J. Pharm. Sci. Res., 1, 2, 32–40.

Ozcelik, H., Kunduk, S. (2021). Preliminary trials on medical gum production. Süleyman Demirel Univ. J. Nat. Appl. Sci., 25, 2, 185–194.

Parliment, T. H. (1998). Applications of a microextraction class separation technique to the analysis of complex flavor mixtures. ACS Symposium Series 705. American Chemical Society.

Pawliszyn, J. (1997). Solid phase microextraction: Theory and practice. John Wiley.

Pfeiffer, J. C., Hort, J., Hollowood, T. A., Taylor, A. J. (2006). Taste-aroma interactions in a ternary system: A model of fruitiness perception in sucrose/acid solutions. Percept. Psychophys., 68, 2, 216–227. https://doi.org/10.3758/BF03193671

Potineni, R. V., Peterson, D. G. (2008). Mechanisms of flavor release in chewing gum: cinnamaldehyde. J. Agric. Food Chem., 56, 9, 3260–67. https://doi.org/10.1021/jf0727847

Rassing, M. R. (1994). Chewing gum as a drug delivery system. Adv. Drug Deliv. Rev., 13, 1–2, 89–121. https://doi.org/10.1016/0169-409X(94)90028-0

Taylor, A. J., Linforth, R. S. T., Harvey, B. A., Blake, A. (2000). Atmospheric pressure chemical ionisation mass spectrometry for in vivo analysis of volatile flavour release. Food Chem., 71, 3, 327–338. https://doi.org/10.1016/S0308-8146(00)00182-5

Uzun, P., Özünal, A. E., Başkaya, H., Doğan, N., Gülay, S. M., Hantal, B., …, Barla Demirkoz, A. (2022). Investigation of flavour permanence of strawberry and waterlemon flavoured chewing gums by using retronasal aroma trapping device and sensory analysis techniques. Gıda, 47, 3, 408–419.

Veldhuizen, M. G., Siddique, A., Rosenthal, S., Marks, L. E. (2018). Interactions of lemon, sucrose and citric acid in enhancing citrus, sweet and sour flavors. Chem. Senses, 43, 1, 17–26. https://doi.org/10.1093/chemse/bjx063

Wong, S. W., Yu, B., Curran, P., Zhou, W. (2009). Characterising the release of flavour compounds from chewing gum through HS-SPME analysis and mathematical modelling. Food Chem., 114, 3, 852–858. https://doi.org/10.1016/j.foodchem.2008.10.030

Woodford, W., Lesco, L. J. (1981). Relative bioavailability of aspirin gum. J. Pharm. Sci., 70, 12, 1341–3. https://doi.org/10.1002/jps.2600701213