Self-perceived mouthfeel and physico-chemical surface effects after chewing gums containing sorbitol and Magnolia bark extract


The European Food Safety Authority recognizes the contribution of sugar-free chewing gum to oral health through increased salivation, clearance of food debris, and neutralization of biofilm pH. Magnolia bark extract is a gum additive shown to reduce the prevalence of bad-breath bacteria but its effects on self-perceived mouthfeel are unknown. This paper aims to relate the effects of sorbitol-containing chewing gum, with and without Magnolia bark extract, on tooth-surface hydrophobicity and salivary-film composition with self-perceived mouthfeel. In a crossover clinical trial, volunteers chewed sorbitol-containing gum, with or without Magnolia bark extract added, three times daily during a 4-wk time period. A subset of volunteers also chewed Parafilm as a mastication control. Oral moistness and tooth smoothness were assessed using questionnaires, and intra-oral water-contact angles were measured before, immediately after, and 60 min after, chewing. Simultaneously, saliva samples were collected, placed on glass slides, and the compositions of the adsorbed film were measured using X-ray photoelectron spectroscopy. Chewing of gum, regardless of whether or not it contained Magnolia bark extract, improved self-perceived mouthfeel up to 60 min, concurrent with a more hydrophilic tooth surface and an increased amount of O_{1s} electrons bound at 532.6 eV in salivary films. Chewing of Parafilm affected neither tooth-surface hydrophobicity nor salivary-film composition. Accordingly, adsorption of sorbitol, rather than the presence of Magnolia bark extract or increased salivation, is responsible for improved self-perceived mouthfeel.

Saliva facilitates lubrication of oral surfaces to enable speech and mastication (1). The salivary lubricating properties are largely determined by the presence of adsorbed hydrophilic and often glycosylated proteins, such as high-molecular-weight mucins, which maintain moist and lubricious oral surfaces (2–4). Lubricity of oral surfaces is not only of physiological and functional importance but also provides a positive self-perceived clean mouth feel, which may constitute a driver of oral-hygiene behaviors. Accordingly, considerable research has been undertaken to evaluate mouthfeel in consumers after use of specific toothpastes, mouthrinses, or toothbrushes (5–8). Sugar-free chewing gum has also been developed as an oral health product to be used as an adjunct to regular oral hygiene (9). Sugar-free gum has been recognized by the European Food Safety Association (EFSA) to promote oral health by increasing salivary flow (10), removing food debris (11), and neutralizing the pH of oral biofilm (12). Moreover, easy incorporation and gradual release, combined with prolonged contact with oral surfaces (13), may make chewing gum a suitable carrier for active ingredients, although hitherto the effects of additives to chewing gum have not been acknowledged by the EFSA as they are mostly overshadowed by the impact of increased salivation and mastication following use of regular, sugar-free gum (9).

Natural products are gaining interest for the maintenance of health. Magnolia bark extract (MBE), harvested from the Magnolia officinalis tree, is a product that has been used in traditional medicine for centuries as an anti-inflammatory, anti-platelet, and even chemopreventive compound (14–18). In oral health, MBE has been shown to inhibit in vitro growth of various oral bacterial strains and species, notably including species associated with oral malodor (19, 20). Chewing gum into which MBE had been incorporated was shown to reduce the total number of bacteria in saliva and to be
beneficial for gingival health (20, 21). The potential beneficial effects of MBE on gingival health have been suggested to be brought about by adsorption of MBE to predominantly Gram-negative bacteria, including bacteria responsible for bad breath, making them more hydrophobic and amenable to removal from the oral cavity (22).

The aim of this study was to evaluate the effects of chewing gum, containing and not containing MBE, on tooth-surface hydrophobicity, composition of salivary films, and self-perceived mouthfeel perception in volunteers.

Material and methods

Chewing gums

Chewing gums were provided by Wm. Wrigley Jr. Company (Chicago, IL, USA). Magnolia bark extract (3 mg; Honsea Sunshine Biotech, Guangzhou, China) was added to the coating of 1.5 g pellet-shaped chewing gums containing gum base, sorbitol, flavoring agents, sweeteners, and coolants. A chewing gum without MBE, but otherwise identical in composition, was used as the control gum. Parafilm (Bemis NA, Neenah, WI, USA), a tasteless and odorless paraffin-coated polyethylene film, was cut into slices of 2 cm x 4 cm and, after removal of the backing paper, was chewed by volunteers to determine the impact of mastication on self-perceived mouthfeel control.

Participants, inclusion criteria, and experimental schedule

Ten healthy volunteers (five men and five women, 29–61 yr of age) participated in this study after giving their written informed consent. The study was approved by the Medical Ethical Testing Committee of the University Medical Center Groningen (METc 2011/330). Inclusion criteria described that volunteers should consider themselves in good health and have a dentition with at least 16 natural teeth, including the central incisors. Volunteers were excluded from the study if they had used antibiotics in the 3 months prior to the study or if they had used a mouthrinse in the month preceding the study. Also, the use of mouthrinses and consumption of mints or chewing gums other than the prescribed chewing gum was not permitted during the entire study, while prescribed usage of antibiotics during the study was a reason for exclusion (22).

Two weeks before the start of the study and continuing through the entire study, volunteers brushed their teeth with a fluoride toothpaste (Prodent Softmint; Sara Lee Household & Bodycare, The Hague, the Netherlands) according to their habitual routine. After 2 weeks of regular brushing, at the beginning of the third week, volunteers were asked to attend the laboratory after they had eaten breakfast but before brushing their teeth. This marked the start of 4 wk of chewing two pellets of gum three times per day. Each gum was chewed for 10 min, spread evenly throughout the day, preferably after breakfast, lunch, and dinner. Gums, with or without active ingredients, were randomly assigned to the volunteers in a double-blind, but balanced, manner (five MBE-containing and five non-MBE-containing gums per experimental period). Laboratory visits were repeated after 1, 2, and 4 wk of chewing. As previous studies on antimicrobial mouthrinses (23) and toothpastes (24) had demonstrated that a washout period of 7 d was sufficient to allow the oral biofilm to regain its prestudy viability, in the present study we applied a 4-wk washout period during which no gum was chewed, after which volunteers crossed over to the alternative treatment regimen and the cycle of laboratory visits was repeated (22). During each laboratory visit, volunteers filled out a mouthfeel questionnaire, and intra-oral water-contact angles were measured before and after chewing. In addition, in five randomly chosen volunteers (two men and three women, 29–61 yr of age) from the above group, saliva was collected and adsorbed onto glass slides for surface physico-chemical analyses (see below). Also, these five volunteers were asked to chew a piece of Parafilm, according to the above protocol. These volunteers were termed the ‘mastication control’. As these experiments were confined to surface physico-chemical analyses, the effects of the chewing of Parafilm on self-perceived mouthfeel were not evaluated.

Mouthfeel questionnaire

On each visit to the laboratory, and before chewing, directly after 10 min of chewing, as well as 30 and 60 min after chewing, volunteers were asked to fill out a questionnaire to evaluate their self-perceived mouthfeel. The following questions needed to be scored on a 5-point ‘mouth condition Likert-scale’, ranging from −2 (dislike extremely) to 2 (like extremely):

- How moist is your mouth?
- How smooth are your teeth?

Tooth-surface hydrophobicity assessed using intra-oral water-contact angles

Tooth-surface hydrophobicity was assessed at the same study time points as mouthfeel was assessed and was measured as follows. Volunteers were seated in a dental chair in a horizontal position wearing a mouth-opening device to expose the maxillary central incisors. These surfaces were dried to a plateau level by waving ambient air over the tooth surface for 30 s. Plateau level was inferred from stable water-contact angles over time, representing the removal of all free water but leaving the adsorbed film in a hydrated state (25, 26). Next, a droplet (approximately 1 µl) of ultrapure water (Sartorius Arium 611; Sartorius, Göttingen, Germany) was placed in the middle of the labial surface of a maxillary central incisor and photographed (Canon EOS D30; Canon, Tokyo, Japan) 1 s after placement. A minimum of three droplets for each time point were photographed after which water-contact angles (θ) were determined according to the formula:

$$\theta = 2 \tan^{-1} \frac{2h}{b},$$

in which h is the height of the droplet and b the width of the base of the droplet.

Composition of the salivary conditioning film as determined using X-ray photoelectron spectroscopy

The elemental composition of the adsorbed salivary films was determined using X-ray photoelectron spectroscopy...
(XPS) (S-Probe; Surface Science Instruments, Mountain View, CA, USA) in five randomly selected volunteers. Two millilitres of saliva was collected before chewing and immediately after chewing, as well as at 30 and 60 min after chewing, according to the above protocol. One-hundred microlitres of each saliva sample was placed on microscope glass slides (1 cm × 1 cm; Menzel, Braunschweig, Germany) [which were precleaned by sonication (Transonic TP 690; ELMA, Singen, Germany) for 5 min in 2% RBS35 (Omnilabo International, Breda, the Netherlands), rinsed with methanol (99.8%; Merck Millipore, Billerica, MA, USA), then blow-dried using nitrogen] for adsorption of a salivary film and left to dry overnight in a covered Petri dish. The samples were subsequently placed in the pre-vacuum chamber of the XPS system (10⁻⁷ Pa) for 4 h and then transferred to the XPS system measuring chamber. X-rays (10 kV, 22 mA) were generated using an aluminum anode at a spot size of 250 μm × 1,000 μm. Scans of the broad spectrum of binding energies were made in the range of 1–1,100 eV, recorded at a low resolution (pass energy 150 eV) on two different spots on each sample. The O1s peak was also scanned over a range of 20 eV at high resolution (pass energy 50 eV). The area under each peak was used to determine the elemental surface composition, in at% of the most abundant elements C1s, O1s, and N1s, accounting for minor salivary constituents such as C2p, P2p, S2p, Ca2p, C1s, and N1s originating from the glass surface. The O1s peak was decomposed in two peaks corresponding to amide groups (C=O-N: 532.6 eV) and alcohol or (hemi) acetal groups (C-OH, C-O-C-O-C, 532.6 eV) (27), as occurring in glycosylated proteins and hydrophilic sugar-alcohols, such as sorbitol. The total presence of these oxygen groups in salivary films was calculated as follows:

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\%O_{532.6} = \frac{\text{area } O_{532.6}}{\text{total area}} 
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in which % area O_{532.6} is the percentage area of the O1s electron-binding energy peak at 532.6 eV and %O_{total} is the total percentage of oxygen groups. Accordingly, % O_{532.6} denotes the percentage of oxygen involved in glycosylated proteins or sugar-alcohols. Previous research has shown that a glass surface coated with salivary proteins displays an interaction with oral bacteria similar to that of saliva-coated enamel and glass surfaces and accordingly may be considered a model for enamel surfaces with respect to adhesive and adsorptive properties (28).

Statistics

Mouthfeel questionnaire data are presented in the Figures as interval data. Although it can be debated whether Likert-scale data should be interpreted as ordered (ordinal) or equal distance (interval) data, it was decided to describe the results using means (interval data) because of the relatively small sample sizes. However, as criteria for parametric testing are not met with a 5-point Likert-scale, effects within individuals during the 4-wk use of chewing gum were analyzed using the Friedman test \((P < 0.05)\). An effect over the 4 wk of use was tested separately for MBE and control gum at the corresponding time points.

As no differences were found regarding self-perceived mouthfeel for the different weekly visits over the 4 wk of use (Table S1), data taken at corresponding time points after chewing were pooled according to type of gum for all four visits and were analyzed for short-term changes up to 60 min after chewing and for differences between chewing gums with and without active ingredients incorporated. Again, the Friedman test was used followed by a Wilcoxon signed-rank test to identify differences between groups.

Intra-oral water-contact angles and the percentage oxygen bound at 532.6 eV were both expressed on a continuous scale and were evaluated for normality using probability plots, and Kolmogorov–Smirnov and Shapiro–Wilk tests \((P < 0.05)\). Subsequently, data were assessed for an effect within individuals during 4 wk of chewing, employing a repeated-measures ANOVA. As no changes in intra-oral water contact angles were observed over the four, weekly, visits at corresponding time points (Table S2), data from these visits were pooled according to type of gum at each study time point (prior, 0, 30 and 60 min). These were then analyzed for short-term changes up to 60 min after chewing and for differences between chewing gums with and without active ingredients. Again, a repeated-measures ANOVA was performed, followed by a Bonferroni test for pairwise comparison.

To assess a possible relationship between intra-oral water-contact angles and mouthfeel perception, average intra-oral water-contact angles were plotted against the average mouthfeel scores at each time point after the chewing of gum. In addition, intra-oral water-contact angles were plotted against the percentage oxygen bound at 532.6 eV in the adsorbed salivary films. All statistical analyses were conducted with SPSS v23.0 (IBM, Armonk, NY, USA).

Results

Immediately after chewing gums, volunteers experienced a significantly better mouthfeel, with respect both to perceived moistness and to smoothness of their teeth, regardless of whether or not the gums contained MBE (Fig. 1). These effects lasted for up to 60 min, after which scores returned to their pre-chew values. There were no significant differences between gums with and without MBE (Fig. 1).

Concurrent with an improved, self-perceived mouthfeel, the results demonstrate that intra-oral water-contact angles significantly decreased directly after chewing, indicating reduced tooth-surface hydrophobicity (Fig. 2). Although this decrease in water-contact angle became smaller over time, it remained significant up to 60 min after chewing for the gums, whether or not MBE was added. However, the MBE-containing gum yielded a slightly more hydrophobic tooth surface than the gum without MBE, suggesting adsorption of the hydrophobic MBE components. Chewing of the mastication control (Parafilm) also led to a reduction in tooth-surface hydrophobicity directly after chewing, but less than observed after the chewing of a gum (Fig. 2). In the absence of any active ingredients in Parafilm this decrease in tooth-surface hydrophobicity must be attributed to increased salivary flow.

X-ray photoelectron spectroscopy was subsequently carried out in order to determine whether the chemical basis for the improved self-perceived mouthfeel and hydrophilicity observed after the chewing of gum was caused by adsorption of gum ingredients other than
The percentage oxygen bound at 532.6 eV in adsorbed salivary films increased significantly directly after chewing of either gum, but not after the chewing of Parafilm (Fig. 3). Thus, the changes in tooth-surface hydrophobicity after the chewing of gum in comparison with those observed when chewing Parafilm are not related to increased salivation but must be a result of adsorption of the sugar-alcohol sorbitol from the sugar-free gums.

In Fig. 4, the intra-oral water-contact angles on teeth are presented as a function of time after chewing a sorbitol-containing gum with (MBE gum) or without (Control gum) Magnolia bark extract (MBE). The time points –10 min and 0 min indicate evaluations made immediately before chewing and directly after chewing, respectively. Lines were drawn by eye and serve solely as a guide to connect the corresponding data points. Asterisks (*) indicate significant differences compared with prior to chewing. Values are given as mean ± standard error and were calculated from the data obtained from 10 volunteers who chewed gum over a 4-wk time period.

Discussion
In this study, we have shown that adsorption of MBE during the chewing of sugar-free gum may yield minor adsorption of its hydrophobic MBE components honokiol and magnolol (14, 29) but the effects on tooth-surface hydrophobicity are completely overshadowed by adsorption of the hydrophilic sugar-alcohol, sorbitol. Concurrent with a more hydrophilic tooth surface after the chewing of gum was an improved mouthfeel perception in volunteers. Previous studies also showed that the chewing of gum created a self-perceived mouthfeel

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**Fig. 1.** Mouthfeel scores. Results are given as a function of time after chewing a sorbitol-containing gum with (MBE gum) or without (Control gum) addition of Magnolia bark extract (MBE). The time points –10 min and 0 min indicate evaluations made immediately before chewing and directly after chewing, respectively. Lines were drawn by eye and serve solely as a guide to connect the corresponding data points. Asterisks (*) indicate significant differences compared with prior to chewing. Values are given as mean ± standard error and were calculated from the data obtained from 10 volunteers who chewed gum over a 4-wk time period.

**Fig. 2.** Intra-oral water-contact angles. Results are given as a function of time after chewing of sorbitol-containing gum with (MBE gum) or without (Control gum) addition of Magnolia bark extract (MBE) and with Parafilm as a mastication control. The time points –10 min and 0 min indicate evaluations immediately prior to chewing and directly after chewing, respectively. Lines were drawn by eye and serve solely as a guide to connect the corresponding data points. Asterisks (*) indicate significant differences compared with prior to chewing. Values are given as mean ± standard error and were calculated from the data obtained from 10 volunteers who chewed gum over a 4-wk time period and from five volunteers who chewed Parafilm.

**Fig. 3.** Percentage of oxygen bound at 532.6 eV in ex vivo adsorbed salivary films. Results are given as a function of time after chewing of a sorbitol-containing gum with (MBE gum) or without (Control gum) Magnolia bark extract (MBE) added and with Parafilm as a mastication control. The time points –10 min and 0 min indicate evaluations immediately prior to chewing and directly after chewing, respectively. Lines were drawn by eye and serve solely as a guide to connect correspond-
of a healthy oral cavity’ (30, 31) but these studies did not relate mouthfeel to the properties of the tooth surface or adsorbed salivary film composition.

Chewing of the mastication control (Parafilm) also led to a reduction in the hydrophobicity of the tooth surface directly after chewing (Fig. 2), but in the absence of any active ingredients in Parafilm this decrease in tooth-surface hydrophobicity must be attributed to increased salivary flow, although there are no major differences between stimulated and unstimulated saliva in terms of water content and the amount of (glycosylated) proteins (2, 32, 33).

Overall, intra-oral water-contact angles were lower with a higher percentage of oxygen bound at 532.6 eV in adsorbed salivary films after the chewing of either gum. A relationship has also been observed between intra-oral contact angles and composition of salivary film following the use of different toothpastes (34). Importantly, tooth-surface hydrophilicity was not altered after chewing the mastication control (Parafilm) (Fig. 5). Therefore, it was concluded that the chewing of sugar-free gum containing sorbitol yields improved self-perceived mouthfeel owing to adsorption of sorbitol onto the tooth surface. Adsorption of the sugar-alcohol sorbitol in adsorbed salivary films favors hydrogen bonding by which higher numbers of water molecules are retained at the surface, thus increasing tooth-surface hydrophilicity, in line with a previous report demonstrating decreased mucosal friction after chewing a sorbitol-containing gum (35). All changes recorded in the present study were short-lived, however, and returned to baseline values within 60 min after the chewing of gum. This is probably a result of the rapid washout of sorbitol from the oral cavity, which confirms that our choice for a washout period of 4 wk between the use of different gums was more than sufficient. The washout of sorbitol and the loss of tooth-surface hydrophilicity after chewing within 60 min (Fig. 2) followed a similar pattern as observed after toothbrushing (36), albeit tooth-surface hydrophilicity established by toothpaste components was lost within 12 h. Thus, by comparison, sorbitol adsorption to salivary films is not very substantive.

In summary, in the present study we have established that self-perceived mouthfeel in volunteers improves up to 60 min after the chewing of regular, sugar-free gum as a result of adsorption of sorbitol to the tooth surface, creating a more hydrophilic surface. Addition of MBE did not impact the self-perceived mouthfeel in terms of perceived moistness or smoothness of teeth.

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References


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Supporting Information

Additional Supporting Information may be found in the online version of this article:

Table S1. Average values ± SD over 10 volunteers of the differences between magnolia-bark-extract (MBE) containing gum and control gum at every time point over the four-wk experimental period for the mouthfeel questionnaire data.

Table S2. Average values ± SD over 10 volunteers of the differences between magnolia-bark-extract (MBE) containing gum and control gum at every time point over the four-wk experimental period for intra-oral water contact angles (degrees).